millenium

Journal of Education, Technologies, and Health

8

Série | Serie 2 • Ano | Year 4

novembro•november | dezembro•december
janeiro•january | fevereiro•february 2019

en | pt

Diretor • Director
Madalena Cunha

Período temporal de publicação | Time period of publication

Série • Serie 1 - 1996/2016 | ano • year 1-21

Série • Serie 2 - 2016/2019 | ano • year 1-4

Acesso livre e gratuito • Free access

ISSNe (versão electrónica•electronic version)1647-662X

millenium
Journal of Education, Technologies, and Health

Ficha Técnica | Technical Sheet | Ficha Técnica

Propriedade | Property | Propiedad

Instituto Politécnico de Viseu (IPV)
NIPC – 680033548
Centro de Estudos em Educação, Tecnologias e Saúde
Unidade de I&D do Instituto Politécnico de Viseu

Sede do Proprietário/Editor/Redator/Impressor | Owner´s Headquarters/Publisher/Writer/Printer | Sede del Proprietario/Editor/Redactor/Impresor

Av. Cor. José Maria Vale de Andrade
Campus Politécnico
3504 - 510 VISEU

232 480 700 (ext.2100)
millenium@sc.ipv.pt (Revista Millenium)
http://www.ipv.pt/millenium/ (Revista Millenium)
http://www.ipv.pt/ci (Centro de Estudos em Educação, Tecnologias e Saúde (CI&DETS) - Unidade de I&D do Instituto Politécnico de Viseu)

Diretor | Director | Director

Madalena Cunha

Ficha Catalográfica | Catalogue File | Ficha Catalográfica

Revista Millenium. / prop. Instituto Politécnico de Viseu, 1996 - 2016

Título da Revista | Journal title |Título de la Revista: Millenium- Revista do Instituto Politécnico de Viseu (IPV)
Título da Revista abreviado | Abbreviated title of the Journal | Título de la Revista abreviado: Rev. Mill
Sigla da Revista | Acronym of the Journal |Sigla de la Revista: Mill
Depósito Legal Nº | Legal Deposit |Depósito Legal: 973 71/96
Número de Registo ERC | ERC Registration Number | Número de Registo ERC: "Anotada"
Estatuto Editorial | Editorial Status | Estatuto Editorial: Estatuto Editorial da Revista Millenium *(http://revistas.rcaap.pt/millenium/pages/view/estatuto)*

ISSNe (versão eletrónica) 1647-662X
Prefixo DOI CrossRef: *https://doi.org/10.29352/mill0208*

Acesso livre e gratuito para autores, revisores e leitores | Free access to authors, rewiers and readers | Acceso libre el autor, revisores e lectores

Periodicidade | Publication Frequency | Periodicidad
Quadrimestral, sendo editada em fevereiro, junho e outubro | Quarterly released in February, June and October | Cuatrimestral, siendo editada em febrero, junio y octubre

Período temporal da publicação | Temporal period of publication | Período de tiempo de publicación
Série 1 - 1996 - 2016 | ano 1 - 21
Série 2 - 2016 - 2019 | ano 0 - 4

Indexação | Indexation | Indexación

- Repositório Científico do Instituto Politécnico de Viseu - http://repositorio.ipv.pt/
- DIALNET – http://dialnet.unirioja.es/
- Latindex – Sistema Regional de Información en Línea para Revistas Científicas de América Latina, el Caribe, España y Portugal http://www.latindex.unam.mx/index.html
- DOAJ - Directory of Open Access Journals – http://www.doaj.org/

Avaliada por Qualis/CAPES | Qualis/CAPES Assessment | Evaluado por Qualis/CAPES

ÁREAS DE AVALIAÇÃO EVALUATION AREAS ÁREAS DE EVALUACIÓN	2012 CLASSIFICAÇÃO CLASSIFICATION CLASIFICACIÓN	2013 CLASSIFICAÇÃO CLASSIFICATION CLASIFICACIÓN	2014 CLASSIFICAÇÃO CLASSIFICATION CLASIFICACIÓN	2013-2016 CLASSIFICAÇÃO CLASSIFICATION CLASIFICACIÓN ISSN 0873-3015	 ISSN 1647-662X
Educação **Education** **Educación**	B2			C	C
Filosofia/Tecnologia: Subcomissão de Filosofia **Philosophy/Theology: Philosophy Subcommittee** **Filosofía/Teología: Filosofía subcomité**	B5				
Interdisciplinar **Interdisciplinary** **Interdisciplinaria**	B2	B3		B3	B3
Literatura / Lingüística **Literature/Linguistics** **Literatura / Lingüística**	B4		B1		
Ciências Agrícolas **Agricultural Sciences** **Ciências Agrícolas**		B5			
Medicina III **Medicine III** **Medicina III**			C	B5	
Enfermagem **Nursing** **Enfermería**					B4
Engenharias I **Engineering I** **Ingenierías I**				B5	B5
Letras/Linguística **Literature/Linguistics** **Letras/Linguística**				B5	B5
Psicologia **Psychology** **Psicología**					B3
Ciências Agrárias I **Agricultural Sciences I** **Ciencias Agrarias I**				B5	
Comunicação e Informação **Communication and Information** **Comunicación e Información**				B5	
História **History** **Historia**				B5	
Odontologia **Dentistry** **Odontología**				B4	
Saúde Coletiva **Collective Health** **Salud Pública**				B4	

Nota | Note | Nota

• Os artigos assinados são da responsabilidade dos seus autores, não refletindo necessariamente os pontos de vista da Direção da Revista

millenium
Journal of Education, Technologies, and Health

Corpo Editorial | Editorial | Consejo Editorial

millenium
Journal of Education, Technologies, and Health

millenium
Journal of Education, Technologies, and Health

Editorial | Editorial | Editorial

A revista Millenium – Journal of Education, Technologies and Health, inaugura o seu terceiro ano da série 2, com a edição regular do número 8, privilegiando a produção científica de manuscritos originais, atestados por blind peer review. A multidisciplinariedade e transdisciplinaridade dos artigos científicos presentes neste número evidenciam o próprio escopo e génese da revista.

A secção Engenharias, Tecnologia, Gestão e Turismo, é composta pelos artigos "Pesquisa sobre as propriedades de resistência do geopolímero feito de resíduos de minas de tungsténio e vidro reciclado", onde se pretende definir o impacto da adição de vidro moído reciclado nas propriedades mecânicas do aglomerante geopolimérico produzido a partir de resíduos de minério de tungsténio; "Tratamento de água residual proveniente da produção de biodiesel com recurso ao oxidante químico ião persulfato, onde encontramos os resultados da aplicação deste oxidante para limpeza da água residual; "Indicadores de qualidade do serviço da ERSAR – caso prático de um município", que apresenta um estudo, realizado entre 2011 e 2016, para avaliar, em termos técnicos, ambientais e económicos, um sistema municipal de gestão de resíduos sólidos, num município de média dimensão; e ainda o artigo "Aplicação das metodologias lean na redução dos tempos de setup na indústria farmacêutica", no qual se demonstra a importância de aplicar a metodologia lean nesta indústria, com vista à competitividade e produtividade.

Na secção Educação e Desenvolvimento Social, apresenta-se o artigo "A dinâmica da criação do conhecimento numa rede intra-organizacional" com evidências que o contexto de uma rede intra-organizacional pode proporcionar um ambiente de aprendizagem coletivo, representado sobretudo pela interação que ocorre entre os colaboradores das unidades e por meio de vários "espaços de interação" e criação de conhecimento.

A secção Ciências Agrárias, Alimentares e Veterinárias contempla os artigos: "Efeito dos hidrocolóides no revestimento e nas propriedades organoléticas do rissol regenerado em forno e comparação com o processo de fritura", no qual se procuram apresentar opções para uma alimentação mais saudável, reduzindo os malefícios associados à fritura; "Caraterização química, propriedades antioxidantes e perfil fenólico de casca de pinheiro", que visa estudar a composição química da casca de Pinus pinaster Aiton subsp. atlantica e o perfil fenólico dos seus extratos aquosos, etanólicos e hidroetanólicos; e o artigo "Influência dos solventes de extração no conteúdo em compostos bioactivos e actividade antioxidante de flores de amores-perfeitos", onde se evidencia que a extração de compostos bioativos e a atividade antioxidante de flores de amores-perfeitos são influenciadas pelo solvente usado suprarreferido.

Na secção Ciências da Vida e da Saúde encontramos o artigo "Promoção da independência Funcional em Idosos Institucionalizados" apresenta, por sua vez, um estudo onde se pretendeu comparar a independência funcional de dois grupos de idosos com características inicialmente semelhantes, residentes numa Estrutura Residencial para Idosos, sedentários, sendo um dos grupos submetido a um programa de exercício físico.

A Equipa Editorial
Madalena Cunha, José Luís Abrantes, Maria João Amante, Paula Correia, Paula Santos

Millenium - Journal of Education, Technologies and Health, inaugurates its third year of the series 2, with the regular edition number 8, privileging the scientific production of original manuscripts, attested by blind peer review. The multidisciplinarity and transdisciplinarity of the scientific papers present in this issue show the very scope and genesis of the journal.

In the section of Engineering, Technology, Management and Tourism, we can find the following papers: "Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass", that intends to define the impact of recycled ground glass addition on mechanical properties of geopolymeric binder made from tungsten mine waste; "Wastewater treatment of biodiesel production using persulphate ion as an oxidant", where we can analyze the results of the application of this oxidant for cleaning the residual water; "ERSAR service quality indicators - practical case of a municipality", which presents a study between 2011 and 2016 to evaluate, in technical, environmental and economic terms, a municipal solid waste management system in a medium-sized municipality; as well as "Application of lean methodologies in the reduction of setup times in the pharmaceutical industry", which seeks to demonstrate the importance of applying lean methodology in this industry regarding competitiveness and productivity.

In section Education and Social Development Sciences, we can find "Dynamics of knowledge creation in intra-organizational networks", that presents evidence that the context of an intra-organizational network may help a collective learning environment, mainly represented by the interaction that occurs between the collaborators of the different units and with the help of several "interaction", creating knowledge spaces.

The Agriculture, Food and Veterinary Sciences section publishes the following papers: "Hydrocolloids effect on coating batter and on organoleptic properties of rissol regenerated in oven and comparison with deep-frying process", in which they try to present options for a healthier diet, reducing the damages associated with frying; "Chemical characterization of pine bark, antioxidant properties and phenolic profile of its extracts", that aims to study the chemical composition of the bark from Pinus pinaster Aiton subsp. atlantica and the phenolic profile of its aqueous, ethanolic and hydroethanolic extracts.; "Extraction solvents' influence on the content of bioactive compounds and antioxidant activity of pansies", that shows that the bioactive compounds and antioxidant activity of pansies' extracts are affected by the solvent used.

The Life and Healthcare Sciences section has the following paper O artigo "Promotion of functional independence institutionalized elderly" in which the authors try to compare functional independence of two elderly groups with initially similar characteristics, living in a nursing home care, after one of the groups being submitted to a physical exercise program.

The Editorial Board
Madalena Cunha, José Luís Abrantes, Maria João Amante, Paula Correia, Paula Santos

La revista Millenium - Journal of Education, Technologies and Health, inaugura su tercer año de la serie 2, con la edición regular del número 8, privilegiando la producción científica de manuscritos originales, atestados por revisión de pares ciego. La multidisciplinariedad y transdisciplinariedad de los artículos científicos presentes en este número evidencian el propio alcance y génesis de la revista.

La sección Ingeniería, Tecnología, Gestión y Turismo, se compone de los artículos "Investigación sobre las propiedades de resistencia del geopolímero hecho de residuos de minas de tungsteno y vidrio reciclado", donde se pretende definir el impacto de la adición de vidrio molido reciclado en las propiedades mecánicas del aglomerante geopolimérico producido a partir de residuos de mineral de tungsteno; "Tratamiento del agua residual proveniente de la producción de biodiesel con recurso al oxidante químico y persulfato, donde encontramos los resultados de la aplicación de este oxidante para la limpieza del agua residual; "Indicadores de calidad del servicio de ERSAR - caso práctico de un municipio", que presenta un estudio, realizado entre 2011 y 2016, para evaluar, en términos técnicos, medioambientales y económicos, un sistema municipal de gestión de residuos sólidos, en un municipio de tamaño medio; y el artículo "Aplicación de las metodologías lean en la reducción de los tiempos de setup en la industria farmacéutica", en el cual se demuestra la importancia de aplicar la metodología lean en esta industria, con el fin de competitividad y productividad.

En la sección Educación y Desarrollo Social, se presenta el artículo "La dinámica de la creación del conocimiento en una red intra-organizacional" con evidencias que el contexto de una red intra-organizacional puede proporcionar un ambiente de aprendizaje colectivo, representado sobre todo por la interacción que ocurre entre los colaboradores de las unidades y por medio de varios "espacios de interacción" y creación de conocimiento.

La sección Ciencias Agrarias, Alimentares y Veterinarias contempla los artículos: "Efecto de los hidrocoloides en el revestimiento y en las propiedades organolépticas del rissole regenerado en el horno y comparación con el proceso de fritura", en el que se buscan presentar opciones para una alimentación más sana, reduciendo los maleficios asociados a la fritura; "Caracterización química, propiedades antioxidantes y perfil fenólico de cáscara de pino", que pretende estudiar la composición química de la cáscara de Pinus pinaster Aiton subsp. atlantica y el perfil fenólico de sus extractos acuosos, etanólicos y hidroetanólicos; y el artículo "Influencia de los disolventes de extracción en el contenido en compuestos bioactivos y actividad antioxidante de flores de amores perfectos", donde se evidencia que la extracción de compuestos bioactivos y la actividad antioxidante de flores de amores-perfectos son influenciadas por el solvente utilizado suprarreferido.

En la sección Ciencias de la Vida y de la Salud encontramos el artículo "Promoción de la independencia funcional en ancianos institucionalizados" presenta, a su vez, un estudio donde se pretendió comparar la independencia funcional de dos grupos de ancianos con características inicialmente similares, residentes en una estructura residencial para ancianos, sedentarios, siendo uno de los grupos sometidos a un programa de ejercicio físico.

El Equipo Editorial
Madalena Cunha, José Luís Abrantes, Maria João Amante, Paula Correia, Paula Santos

millenium
Journal of Education, Technologies, and Health

Sumário | Summary | Resumen

ENGINEERING, TECHNOLOGY, MANAGEMENT AND TOURISM

EDUCATION AND SOCIAL DEVELOPMENT

AGRICULTURAL SCIENCES, FOOD AND VETERINARY

LIFE AND HEALTH SCIENCES

Autores | Authors | Autores

ENGENHARIAS, TECNOLOGIA, GESTÃO E TURISMO

ENGINEERING, TECHNOLOGY, MANAGEMENT AND TOURISM

INGENIERÍA, TECNOLOGÍA, ADMINISTRACIÓN Y TURISMO

millenium

ENGENHARIAS, TECNOLOGIA, GESTÃO E TURISMO
ENGINEERING, TECHNOLOGY, MANAGEMENT AND TOURISM
INGENIERÍA, TECNOLOGÍA, ADMINISTRACIÓN Y TURISMO

millenium 13

Millenium, *2*(8), 13-20.

en

PESQUISA SOBRE AS PROPRIEDADES DE RESISTÊNCIA DO GEOPOLÍMERO FEITO DE RESÍDUOS DE MINAS DE TUNGSTÉNIO E VIDRO RECICLADO

RESEARCH ON STRENGTH PROPERTIES OF GEOPOLYMER BASED ON TUNGSTEN MINE WASTE AND RECYCLED GROUND GLASS

INVESTIGACIÓN SOBRE LAS PROPIEDADES DE RESISTENCIA DEL GEOPOLÍMERO HECHO DE RESIDUOS DE MINERÍA DE TUNGSTENO Y VIDRIO RECICLADO

Szymon Dawczyński[1]
Paulina Cieśla[1]
Marcin Górski[1]
Rafał Krzywoń[1]

[1] *Silesian University of Technology, Gliwice, Polónia*

Szymon Dawczyński - szymon.dawczynski@polsl.pl | Paulina Cieśla - paulinaciesla6@gmail.com | Marcin Górski - marcin.gorski@polsl.pl | Rafał Krzywoń - rafal.krzywon@polsl.pl

Corresponding Author
Szymon Dawczyński
Silesian University of Technology
Akademicka 2A, 44-100 Gliwice, Polónia
szymon.dawczynski@polsl.pl

RECEIVED: 12th April, 2018
ACCEPTED: 18th September, 2018

Dawczyński, S., Cieśla, P., Górski, M. & Krzywoń, R. (2019).
Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass. *Millenium, 2*(8), 13-20.
DOI: https://doi.org/10.29352/mill0208.01.00185

RESUMO

Introdução: Os geopolímeros são os materiais inorgânicos modernos obtidos como resultado da reação química sob condições fortemente alcalinas. As principais vantagens destes materiais são: parâmetros de alta resistência; resistência ao fogo; resistência ao gelo e resistência a ambientes agressivos. Por causa da menor emissão de CO_2 durante o processo de produção, eles também são considerados materiais ecológicos em comparação com materiais de cimento Portland comuns.
Objetivos: O objetivo da investigação foi definir o impacto da adição de vidro moído reciclado nas propriedades mecânicas do aglomerante geopolimérico produzido a partir de resíduos de minério de tungsténio.
Métodos: Os parâmetros de resistência de amostras geopoliméricas foram determinados com base no teste de flexão e teste de compressão.
Resultados: As amostras geopoliméricas que contêm 20%, 40% e 80% de vidro moído reciclado apresentam resistência à flexão similar. A resistência à compressão de geopolímeros à base de vidro aumenta com o crescente conteúdo de vidro moído reciclado.
Conclusões: Em termos de resistência à flexão, a composição mais favorável foi a mistura composta por 60% de vidro moído reciclado e 40% de resíduos de minério de tungsténio. Em termos de resistência à compressão, a composição mais favorável foi a mistura composta de 80% de vidro moído reciclado e 20% de resíduos de minério de tungsténio.

Palavras-chave: geopolímero; ligante geopolimérico; resíduos de minério de tungsténio; vidro moído reciclado.

ABSTRACT

Introduction: Geopolymers are the modern inorganic materials obtained as a result of chemical reaction under strongly alkaline conditions. The main advantages of these materials are: high strength parameters, fire resistance, frost resistance and resistance to aggressive environment. Because of lower CO_2 emission during production process, they are also considered as environmentally friendly materials in comparison to ordinary Portland cement (OPC) systems.
Objectives: The aim of the investigation was to define the impact of recycled ground glass addition on mechanical properties of geopolymeric binder made from tungsten mine waste.
Methods: The strength parameters of geopolymeric samples were determined on the basis of three-point flexural test and uniaxial compression test.
Results: The geopolymer samples that contain 20%, 40% and 80% of recycled ground glass present similar flexural strength. The compressive strength of glass-based geopolymers increases with growing content of recycled ground glass.
Conclusions: In terms of flexural strength, the most favourable composition was the mixture composed of 60% recycled ground glass and 40% tungsten mine waste. In terms of compressive strength, the most favourable composition was the mixture composed of 80% recycled ground glass and 20% tungsten mine waste.

Keywords: geopolymer; geopolymeric binder; tungsten mine waste; recycled ground glass.

RESUMEN

Introducción: Los geopolímeros son los materiales inorgánicos modernos obtenidos como resultado de la reacción química bajo condiciones fuertemente alcalinas. Las principales ventajas de estos materiales son: parámetros de alta resistencia, resistencia al fuego, resistencia a las heladas y resistencia al entorno agresivo. Debido a la menor emisión de CO_2 durante el proceso de producción, también se consideran materiales ecológicos en comparación con los materiales ordinarios de cemento Portland.
Objetivos: El objetivo de la investigación fue definir el impacto de la adición de vidrio triturado reciclado en las propiedades mecánicas del aglutinante geopolimérico a partir de desechos de la mina de tungsteno.
Métodos: Los parámetros de fuerza de las muestras geopoliméricas se determinaron sobre la base de la prueba de flexión de tres puntos y la prueba de compresión.
Resultados: Las muestras de geopolímeros que contienen 20%, 40% y 80% de vidrio triturado reciclado presentan una resistencia a la flexión similar. La resistencia a la compresión de los geopolímeros basados en vidrio triturado aumenta con el creciente contenido de vidrio triturado reciclado.
Conclusiones: En términos de resistencia a la flexión, la composición más favorable fue la mezcla compuesta por 60% de vidrio triturado reciclado y 40% de desechos de la mina de tungsteno. En términos de resistencia a la compresión, la composición más favorable fue la mezcla compuesta de un 80% de vidrio reciclado y un 20% de desechos de la mina de tungsteno.

Palabras Clave: geopolímero; aglutinante geopolimérico; residuos de la mina de tungsteno; vidrio triturado reciclado.

Dawczyński, S., Cieśla, P., Górski, M. & Krzywoń, R. (2019).
Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass. *Millenium, 2*(8), 13-20.
DOI: https://doi.org/10.29352/mill0208.01.00185

INTRODUCTION

From a chemical point of view, the term "geopolymer" refers to a wide group of modern inorganic binders obtained as a result of chemical reaction under strongly alkaline conditions. Geopolymer synthesis, also known as geopolymerization, involves combining powdered solid precursor rich in silicon (Si) and aluminium (Al) with an alkaline solution. The product of chemical reaction is 3-dimensional amorphous aluminosilicate network built up of cross linked SiO_4 and AlO_4 tetrahedral units (Davidovits, 2008). Connection between tetrahedral is provided by bridging oxygen atoms. The negative charge of AlO_4 tetrahedron is balanced by positive metal ions such as sodium, potassium, barium, lithium or calcium.
Generally, alkali-activation technology provides numerous advantages over ordinary Portland cement (OPC) systems. Apart from being environmentally friendly, geopolymers are also characterized by extraordinary properties such as high flexural and compressive strength, excellent resistance to aggressive environment (acids, sulphates, chlorides) and ability to withstand extreme weather conditions and corrosion attack (Provis and van Deventer, 2009).

1. THEORETICAL FRAMEWORK

In practice, geopolymer synthesis starts with mixing finely ground aluminosilicate material with an alkaline solution. Currently, geopolymeric binders may be produced of a wide range of solid materials including industrial by-products (e.g. coal fly ashes, blast furnace slag) and primary raw materials (calcined clays). Alkaline activator is usually a combination of sodium and potassium hydroxide and silicate. Distilled water is often added to the mixture in order to provide smooth consistency, which in turn allows for easier mixing and moulding of the samples. The selection of raw materials becomes a very significant stage in formation of geopolymeric binders because it strongly influences resulting properties such as workability, setting behaviour, physical and chemical features of the final product. Moreover, various types of additives may be introduced into the mixture in order to enhance specific material properties. The most commonly added materials include: metakaolin; ground glass; fly ash; pozzolan; kaolinite and silica sand. Addition of heat resistance fillers such as chamotte, corundum, wollastonite, fly ash and carbon, basalt or ceramic fibres may help to control shrinkage (Vickers, van Riessen, Rickard, 2015). Moreover, the natural aggregates (e.g. limestone, granite or schist) used for the production of Ordinary Portland Cement (OPC) concrete are also suitable for the production of geopolymeric concrete. According to Provis and van Deventer (2009), the formation of geopolymers consists of two basic steps: mixing of ingredients and heat-curing. In most cases, geopolymeric binders are allowed to be cured at room temperature, however heat-curing is more favourable. It was also proved that temperature and the time of curing strongly influence compressive strength of geopolymers. Moreover, the fastest strength increase is observed during first 24 hours of heat treatment (Adam and Horianto, 2014).
In order to obtain satisfactory mechanical properties of geopolymers, source materials should contain an optimal amount of reactive silica and alumina. Recycled glass shows great potential for use in geopolymer technology because it contains a lot of reactive silica and is chemically and physically homogeneous (Christiansen, 2013). Moreover, such a high content of silica may eliminate the need for sodium silicate in the starting mixture. However, glass cullet contains relatively small amount of alumina, what may negatively affect the stability and mechanical properties of the final product. Geopolymer composition in this case should be carefully designed and a proper amount of materials rich in reactive alumina should be introduced into the mixture (Vafaei and Allahverdi, 2017).

2. METHODS

A comprehensive research program has been established to investigate the physical and mechanical properties of geopolymer made from tungsten mine waste. In this paper, only the influence of recycled ground glass addition on strength parameters is discussed.

2.1 Materials used

Tungsten mine waste was obtained from Panasqueira mine during international cooperation under the European Union's Horizon 2020 Research and Innovation Staff Exchange programme (REMINE, 2018). It is worth to mention that Panasqueira mine is one of the oldest tungsten mines in Europe and it is located in the Municipality of Covilhã, District of Castelo Branco. Currently, Panasqueira mine produces between 100-200 tonnes of waste material per day (Sangiorgi et al., 2016). During the tungsten excavation two main groups of waste are produced. First are coarse aggregates which are by-products of rock blasting. Such aggregates are used in small quantities for the construction of the road surfaces. Second by-product of tungsten mining is waste mud accumulated in large lagoons accounting for several million tonnes (Pacheco-Torgal et al., 2008). Over the years of mining activity, the heaps of waste mud have reached enormous proportions. In the area of former plant called Rio, about 730000 m^3 of tungsten mine waste are stored, while the Barroca Grande plant accumulated over 1.1 million m^3 of waste mud. The results of the diffractometer study (X-ray power diffraction system) indicated that the waste mud consists mainly of quartz and muscovite (Pacheco-Torgal et al., 2007). While analysing the chemical composition, tungsten mud contains a high

Dawczyński, S., Cieśla, P., Górski, M. & Krzywoń, R. (2019).
Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass. *Millenium, 2*(8), 13-20.
DOI: https://doi.org/10.29352/mill0208.01.00185

percentage of silica and alumina. Small quantities of sulphur, titanium and magnesium oxide are also present in the sample. Moreover, tungsten waste mud consists of relatively large amount of iron and potassium oxide which according to Pacheco-Torgal et al. (2005) may have a significant impact on the compressive strength of geopolymer binder. Considering the composition of material used as a precursor, it can be concluded that waste mud shows potential for use in geopolymer technology due to its relatively high percentage of silica and alumina which may guarantee a successful result of alkali-activation processes.

In this research, collected in Panasqueira mine waste mud was first dried in the oven and then manually sieved to obtain fine powder, with the granulation not bigger than 500 micrometres (0.5 mm).

Soda-lime glass is the least expensive and the most widely used type of glass constituting over 90% of all glass produced. Such glass is primarily used for the production of bottles, everyday drinking glasses, jars and also for window glass, where chemical and high temperature resistance are not required. Recycled glass powder (cullet) is normally made from soda-lime glass of type used for glass containers. Standard composition of soda-lime glass contains mainly sodium oxide, calcium oxide and silica which is favourable in terms of geopolymerization process.

To obtain the ground glass for the research, first some clear bottles were collected and then with the use of Los Angeles machine a fine cullet was made. Similarly, as it was in the case of tungsten mud waste, also the ground glass was manually sieved to obtain grain fraction from 0 to 0.5 mm.

A combination of both sodium hydroxide and sodium silicate solution was applied as an alkaline activator in geopolymer formation. Sodium-containing solutions were chosen because they are less expensive and more widely available than potassium-based ones. The solution of sodium hydroxide (NaOH) was made from pellets (with 97.5% purity and molar mass of 40 g/mol) which were dissolved in demineralised water. The final NaOH solution of 10 M concentration was prepared 24 hours before forming the samples to allow time for liquid equilibrating.

The second liquid ingredient used in the experiment included sodium silicate solution of R-145 type. While taking into consideration its chemical composition, the molar ratio (SiO_2/Na_2O) was equal to 2.6 and the solid content ($Na_2O + SiO_2$) ranged between 39-43%. According to specifications, the relative density of waterglass solution was equal to about 1.45-1.48 g/cm^3.

The liquid ingredients such as sodium hydroxide and sodium silicate were combined together in ratio 4.0.

2.2 Samples preparation

The geopolymer samples were synthesized from various proportions of waste mud and recycled ground glass (Table 1).

Table 1 - Proportion of solid ingredients in individual samples

no.	sample	ground glass (w/w)	waste mud (w/w)
1	GG 20	20	80
2	GG 40	40	60
3	GG 60	60	40
4	GG 80	80	20

Preparation of geopolymer samples is an important stage which strongly influences the final characteristics of the finished products, their microstructure and resulting mechanical properties. The formation of geopolymer samples consists of three main steps: mixture proportioning and mixing, casting and heat curing.

Weighing of raw materials was performed with use of the laboratory scale accurate to 1.0 mg. The dry ingredients such as tungsten waste mud and ground glass were dry mixed before being added to the alkaline solution. Solid components were blended using putty knife until the material became homogenous. Then, the sodium silicate and sodium hydroxide (NaOH) solutions were combined together and mixed for 5 minutes. The next step involved adding the liquid to the solid constituents and precise mixing until the geopolymer paste became smooth and homogenous. The time and the way of mixing are very significant factors that strongly affect the quality of the finished sample. The distilled water was also added to the mixture in order to provide more fluid consistency and enable easier mixing. The excessive amounts of water contained in the samples may lead to drying shrinkage and occurrence of cracks. This in turn may cause material weakening and reduction of strength. Such phenomenon was also considered and the water dosing was carefully controlled. Finally, the following dependence was observed – the higher the ground glass content, the lower the water demand of geopolymer sample. The second stage involved pouring the geopolymer mixture into the special mould made out of Plexiglas. The choice of Plexiglas instead of steel was made in order to avoid any interaction between mixture components and internal walls of the mould during casting and curing process. The mould provided a space for six specimens with dimensions 40 x 40 x 160 mm. The mixture was carefully placed into the mould using a putty knife. After casting, the geopolymer samples were wrapped in protective

Dawczyński, S., Cieśla, P., Górski, M. & Krzywoń, R. (2019).
Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass. *Millenium, 2*(8), 13-20.
DOI: https://doi.org/10.29352/mill0208.01.00185

film to prevent water evaporation and to protect them against the impact of humidity during the curing process. Generally, alkali-activated binders are allowed to be cured at ambient temperature, however heat-curing is more favourable and results in higher compressive and flexural strength of the final product. For this reason, the geopolymer samples were cured in a climatic chamber for 24 hours at temperature 60°C. After transferring from the mould, the geopolymer specimens were stored at room temperature (about 20°C) for the next 27 days.

2.3 Mechanical behaviour analysis

Mechanical parameters of geopolymeric samples were determined on the basis of three-point flexural test and uniaxial compression test. The strength tests were carried out using PILOT automatic compression-flexural tester (Controls S.p.A., Italy) in accordance with the Polish Standard PN-EN 1015-11:2001: Methods of test for mortar for masonry – Part 11: Determination of flexural and compressive strength of hardened mortar. The following standard provides the detailed requirements connected with testing of masonry samples.
The three-point bending test was performed using geopolymer prisms with dimensions 40 x 40 x 160 mm. During bend-testing, the samples were subjected to the concentrated force F acting in the middle of the beam span (Figure 1, left). The distance between supporting pins was equal to 100 mm. The loading pin was gradually lowered at a constant rate (50 N/s) until failure occurred. The flexure test was carried out using four geopolymer prisms, each of them contained different amount of ground glass (Table 1). Bending strength was determined on the basis of the following relationship:

$$f_b = 1.5 \times \left(\frac{Fl}{bd^2}\right) \quad (1)$$

where:
F – destructive force [N],
l – span length [mm],
b – width of the sample [mm],
d – height of the sample [mm].

Uniaxial compression test was carried out on the portions of prisms previously broken in flexure – two parts of each sample were examined. The geopolymer specimens were placed in between two steel plates (Figure 1, right) and axially loaded by two opposing forces directed towards each other. The moveable head of the testing machine was gradually lowered until the sample broke. The uniaxial compressive strength is calculated with the use of the following formula:

$$R = \frac{P}{A} \quad (2)$$

where:
P – critical compressive force [N],
A – cross-sectional area subjected to compression [mm^2].

Figure 1 - Left: geopolymer beam subjected to bending. Right: half of the beam exposed to uniaxial compression.

Dawczyński, S., Cieśla, P., Górski, M. & Krzywoń, R. (2019).
Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass. *Millenium, 2*(8), 13-20.
DOI: https://doi.org/10.29352/mill0208.01.00185

3. RESULTS

Flexural strength of geopolymer samples is shown in Table 2. The values of bending strength were determined on the basis of destructive force according to the equation (1). In most cases, as a result of the test an irregular crack formed in the middle of the sample (perpendicularly to the longitudinal axis of the beam), where the loading pin was applied. In case of specimen with the highest ground glass content (GG80), bending crack also formed in the middle of the beam, however its shape was completely different – except one main crack there were also some smeared cracks.

Table 2 - Results of three-point bending tests

Bending		
Sample	**Destructive force F [kN]**	**Strength f_b [MPa]**
GG20	0.778	1.902
GG40	0.893	2.093
GG60	1.309	3.093
GG80	0.778	1.951

The destructive force magnitudes in compression tests are presented in Table 3. On the basis of these values and according to equation (2) the compressive strength of geopolymer samples were calculated (Table 4).

Table 3 - Results of uniaxial compression test – values of destructive forces.

Compression			
Sample	**Force P_1 [kN]**	**Force P_2 [kN]**	**Mean value P [kN]**
GG20	9.16	8.49	8.83
GG40	13.90	13.59	13.75
GG60	21.96	19.23	20.60
GG80	73.53	82.50	78.02

Table 4 - Results of uniaxial compression test – compressive strength values.

Compression			
Sample	**Strength R_1 [MPa]**	**Strength R_2 [MPa]**	**Mean value R [MPa]**
GG20	5.73	5.31	5.52
GG40	8.69	8.49	8.59
GG60	13.73	12.02	12.88
GG80	45.96	51.56	48.76

4. DISCUSSION

While considering the results of bending test, it can be noticed that flexural strength of geopolymer samples varies with growing amount of ground glass addition. Analysing the results in Table 2, bending strength initially increases with increasing amount of ground glass. The geopolymer sample that contains 60% w/w glass present the highest flexural strength which is equal to 3.1 MPa. Addition of glass powder up to 80% w/w leads to the reduction of strength which then equals only 1.95 MPa. Moreover, geopolymer samples that contain 20%, 40% and 80% w/w ground glass show very similar flexural strength which is respectively 1.90; 2.09; 1.95 MPa. The obtained results are rather poor in comparison with bending strength values presented in other researches (Dawczyński et al., 2016). However, these differences may be caused by many factors such as manufacturing parameters (method and time of mixing, amount of water added to the mixture) or time and temperature of curing (Adam and

Horianto, 2014). Therefore, further series of strength test are necessary to examine the behaviour of geopolymers subjected to flexure better and to draw more detailed conclusions. Moreover, the investigation concerning material microstructure and particle morphology need to be performed in order to determine the exact causes of strength loss.
While taking into consideration the results of uniaxial compression test, it can be noticed that compressive strength of glass-based geopolymers increases with growing content of ground glass. Analysing the values in Table 4, the compressive strength rises almost linearly with an increase of ground glass content up to 60% w/w. The differences in strength between samples GG20, GG40 and GG60 are not greater than 5 MPa. With an increase of glass content up to 80% w/w the measured compressive strength reaches about 50 MPa, which is almost four times higher than in case of sample that contains 60% w/w ground glass. Such a good compression behaviour may be connected with smaller amount of water introduced into the mixture during preparatory works. According to the literature, an evaporation of water from highly saturated samples may lead to drying shrinkage and surface cracks occurrence. This in turn may cause material weakening and compressive strength reduction (Provis and van Deventer, 2009). Moreover, the results obtained by (Zaharaki and Komnitsas, 2009), partially confirm the behaviour observed in the following test. It was indicated that an addition of glass powder up to 50% w/w does not strongly influence the compressive strength of geopolymers which remains almost the same. They also reported that an increase of glass content above 90% w/w results in rapid compressive strength growth. Therefore, further series of strength test are required in order to examine such behaviour better and to identify the causes of the rapid strength increase.

CONCLUSIONS

On the basis of the results of three-point bending test and uniaxial compression test the following conclusions can be drawn:

- Flexural strength of glass-based geopolymer samples increases with an increase of ground glass addition up to 60% w/w. Addition of glass powder up to 80% w/w leads to bending strength reduction;
- The most favourable composition in terms of flexural strength was obtained for geopolymer sample composed of 60% w/w glass. In this case, the measured strength was equal to 3.1 MPa;
- Compressive strength of geopolymer samples composed of 20%, 40%, 60% and 80% w/w glass increases with increasing ground glass content;
- The most favourable composition in terms of compressive strength was obtained for geopolymer specimen composed of 80% w/w glass. In this case, the measured strength was equal to 48.8 MPa.

The mechanical properties of the tungsten mine waste geopolymeric binder depends not only on the proportion of the materials used (precursor as well as activator) but also on temperature and time of curing. In this case, to reduce the number of laboratory tests, numerical analysis may be helpful, but it requires a proven material model. An example of such an analysis is presented in the paper (Górski et al., 2016).

ACKNOWLEDGEMENTS

This paper has been prepared with the financial support of European Union's Horizon 2020 Research and Innovation Staff Exchange (RISE) programme (Marie Skłodowska-Curie Actions) under grant agreement no. 645696, project REMINE (Reuse of Mining Waste into Innovative Geopolymeric-based Structural Panels, Precast, Ready Mixes and Insitu Applications) and Polish Ministry of Science and Higher Education project no. 341284/PnH/2016.

REFERENCES

Adam, A. A., Horianto (2014). The effect of temperature and duration of curing on the strength of fly ash based geopolymer mortar. *Procedia Engineering, 95,* 410-414.

Christiansen, M.U. (2013). An Investigation of Waste Glass-Based Geopolymers Supplemented with Alumina. PhD Thesis, Michigan Technological University.

Davidovits, J. (2008): *Geopolymer Chemistry & Applications*. Saint-Quentin: Institut Géopolymere.

Dawczyński, S., Górski, M., Krzywoń, R. (2016). Geopolymer as an alternative ecological material for buildings. *Proceedings of 14th International Conference on New Trends in Statics and Dynamics of Buildings*. Bratislava: Slovak University of Technology.

Górski, M., Krzywoń, R., Safuta, M., Paszek, N., Dawczyński, S., Pizoń, J. (2016). Laboratory test of material properties of geopolymers made out of postmining waste and their numerical modelling'. *Materiały Budowlane, 8,* 89-91.

Pacheco-Torgal, F., Castro-Gomes, J. P., Jalali, S. (2005). Geopolimeric Binder Using Tungsten Mine Waste: Primarily Investigation. Geopolymer, Green Chemistry and Sustainable Development Solutions, Geopolymer Institute.

Dawczyński, S., Cieśla, P., Górski, M. & Krzywoń, R. (2019).
Research on strength properties of geopolymer based on tungsten mine waste and recycled ground glass. *Millenium, 2*(8), 13-20.
DOI: https://doi.org/10.29352/mill0208.01.00185

Pacheco-Torgal, F., Castro-Gomes, J. P., Jalali, S. (2007). Using Mine Waste Mud to Produce Environmentally Friendly New Binders. In *Portugal SB07, Sustainable Construction, Materials and Practices, Challenge of the industry for the New Millennium* (pp. 912-917). Amsterdam: IOS Press BV.

Pacheco-Torgal, F., Castro-Gomes, J. P., Jalali, S. (2008). Investigations of tungsten mine waste geopolimeric binder: Strength and microstructure. *Construction and Building Materials, Vol. 22 (11),* 2212-2210.

Provis, J. L., van Deventer, J. S. J. (2009): *Geopolymers - Structure, processing, properties and industrial applications.* Oxford, Cambridge, New Delhi: Woodhead Publishing Limited.

REMINE (2018). Retrieved from https://reminemsca.wordpress.com

Sangiorgi, C., Lantieri, C., Tatarani, P., Castro-Gomes, J., Gabriel, M. (2016). Reuse of mining waste into innovative alkali-activated-based materials for road pavement applications. *Functional Pavement Design*. Erkens et al. (Eds). London: Taylor & Francis Group.

Vafaei, M., Allahverdi, A. (2017). High strength geopolymer binder based on waste-glass powder. *Advanced Powder Technology*, *Vol. 28 (1),* 215-222.

Vickers, L., van Riessen, A., Rickard, W.D.A. (2015): *Fire-resistant Geopolymers. Role of Fibres and Fillers to enhance Thermal Properties*. Singapore: Springer.

Zaharaki, D., Komnitsas, K. (2009). Effect of additives on the compressive strength of slag-based inorganic polymers. *Global NEST Journal, Vol. 11(2),* 137-146.

ENGENHARIAS, TECNOLOGIA, GESTÃO E TURISMO
ENGINEERING, TECHNOLOGY, MANAGEMENT AND TOURISM
INGENIERÍA, TECNOLOGÍA, ADMINISTRACIÓN Y TURISMO

millenium 21

Millenium, *2*(8), 21-27.

en

TRATAMENTO DE ÁGUA RESIDUAL PROVENIENTE DA PRODUÇÃO DE BIODIESEL COM RECURSO AO OXIDANTE QUÍMICO IÃO PERSULFATO

WASTEWATER TREATMENT OF BIODIESEL PRODUCTION USING PERSULPHATE ION AS AN OXIDANT

TRATAMIENTO DE AGUAS RESIDUALES DE LA PRODUCCIÓN DE BIODIESEL UTILIZANDO ION PERSULFATO COMO OXIDANTE

Maria Teresa Borralho[1]
Ana Pardal[2]
Solange Coelho[2]

[1] *Polytechnic Institute of Beja, School of Agriculture, Department of Technology and Applied Sciences, Beja, Portugal*
[2] *Polytechnic Institute of Beja, School of Agriculture, Beja, Portugal*

Maria Teresa Borralho - mtcarvalhos@ipbeja.pt | Ana Pardal - apardal@ipbeja.pt| Solange Coelho - solangecoelhopias@hotmail.com

Corresponding Author
Maria Teresa Borralho
Instituto Politécnico de Beja
Rua Pedro Soares, Campus do Instituto Politécnico de Beja
Apartado 6155
7800-295 Beja
mtcarvalhos@ipbeja.pt

RECEIVED: 17th June, 2018
ACCEPTED: 19th September, 2018

Borralho, M. T., Pardal, A. & Coelho, S. (2019).
Wastewater treatment of biodiesel production using persulphate ion as an oxidant. Millenium, 2(8), 21 -27.
DOI: https://doi.org/10.29352/mill0208.02.00199

RESUMO

Introdução: A produção de biodiesel produz um efluente altamente poluente, apresentando altos valores de Carência Química de Oxigênio (CQO), óleos e gorduras, entre outros contaminantes.
O persulfato de sódio é um poderoso oxidante (Eº = 2.1V). Além da oxidação direta que é favorecida em meios alcalinos, ela pode ser induzida por processos fotolíticos, fotos catalíticos e / ou catalíticos formando radicais sulfato (Eº = 2.6 V), proporcionando mecanismos de reação com radicais livres como radicais hidróxilo.
Objetivos: Neste trabalho foi testado o efeito do composto monopersulfato de potássio (2KHSO5KHSO4K2SO4), (Oxone) na remoção da CQO em água residual da etapa de purificação do biodiesel.
Resultados: Os ensaios, oxidação direta a pH alcalino e oxidação catalítica com iões cobalto, Co (II), mostraram que os primeiros, para concentrações de Oxone de 1.00x10-2 M e 4.00x10-3 M, não foram efetivos, pois não houve decomposição do mesmo, para tempos de reação de três horas, não tendo ocorrido, portanto, remoções da CQO. Nos ensaios catalíticos foram testadas combinações variando entre 5.00x10-3 M e 7.50x10-2 M para o oxidante e 0.10 µM e 1.00 µM para o catalisador.
A decomposição completa do oxidante foi sempre verificada entre 15 min. e 2 h.
Os testes com menor concentração de cobalto mostraram taxas de remoção de CQO em torno de 20% enquanto as maiores atingiram os 60%.
Conclusões: O ensaio mais favorável consegue remoções significativas da CQO, mas não o suficiente para que o efluente seja descarregado em meio hídrico, de acordo com a legislação portuguesa.

Palavras-chave: “Monopersulfato de potássio”, “Oxidação química”, “Carência Química de Oxigênio”, “Águas residuais da purificação do biodiesel”.

ABSTRACT

Introduction: The production of biodiesel produces a highly polluting effluent, because presents high values of Chemical Oxygen Demand (COD), oils and fats, among other contaminants.
Sodium persulphate is a powerful oxidant (Eº = 2.1V). In addition to the direct oxidation that is favored in alkaline media, it can be induced by photolytic processes, catalytic and / or catalytic photos forming sulphate radicals (Eº = 2.6 V) thus providing reaction mechanisms with free radicals like hydroxyl radicals.
Methods: In this work the effect of the potassium monopersulphate compound ($2KHSO_5.KHSO_4K_2SO_4$), (Oxone) on the removal of COD in residual water from the biodiesel purification step was tested.
Results: The tests, direct oxidation at alkaline pH and catalytic oxidation with cobalt ions, Co(II), showed that the former, for Oxone concentrations of $1.00x10^{-2}$ M and $4.00x10^{-3}$ M, were not effective because no decomposition occurred, for reaction times over three hours, thus not occurring removal of COD. In the catalytic assays combinations were tested, varying between $5.00x10^{-3}$ M and $7.50x10^{-2}$ M for the oxidant and 0.10 µM and 1.00 µM for the catalyst.
The complete decomposition of the oxidant was always verified between 15 min. and 2 h.
The tests with the lowest concentration of cobalt showed COD removal rates around 20% and the highest one reached 60%.
Conclusions: The most favorable assay achieves significant COD removals, but not enough for the effluent to be discharged in the hydric medium according the portuguese legislation.

Keywords: “Potassium monopersulphate”, “Chemical oxidation”, “Chemical Oxygen Demand”, “Wastewater from biodiesel purification”.

RESUMEN

Introducción: La producción de biodiesel produce un efluente altamente contaminante, presentando altos valores de Demanda Química de Oxígeno (DQO), aceites y grasas, entre otros contaminantes.
El persulfato de sodio es un poderoso oxidante (Eº = 2.1V). Además de la oxidación directa que es favorecida en medios alcalinos, puede ser inducida por procesos foto líticos, fotoscatalíticos y/o catalíticas formando radicales sulfato (Eº = 2.6 V), proporcionando mecanismos de reacción con radicales libres como los radicales hidroxilo.
Objetivos: En este trabajo se probó el efecto del compuesto monopersulfato de potasio (2KHSO5KHSO4K2SO4), (Oxone) en la remoción de la CQO en agua residual de la etapa de purificación del biodiesel.
Resultados: En los ensayos catalíticos se probaron combinaciones que oscilan entre 5.00x10-3 M y 7.50x10-2 M para el oxidante y 0.10 µM y 1.00 µM para el catalizador.La descomposición completa del oxidante ha sido siempre comprobada entre 15 min. y 2 h.
Los ensayos, oxidación directa a pH alcalino y oxidación catalítica con iones cobalto, Co (II), mostraron que los primeros, para concentraciones de Oxone de 1.00x10-2 M y 4.00x10-3 M, no fueron efectivos, pues no hubo descomposición de este, para

tiempos de reacción de tres horas, no habiendo ocurrido, por lo tanto, remociones de la CQO. En los ensayos catalíticos se probaron combinaciones que oscilan entre 5.00x10-3 M y 7.50x10-2 M para el oxidante y 0.10 µM y 1.00 µM para el catalizador. La descomposición completa del oxidante ha sido siempre comprobada entre 15 min. y 2 h.
Las pruebas con menor concentración de cobalto mostraron tasas de remoción de DQO en torno al 20% mientras que las mayores alcanzaron el 60%.
Conclusiones: La prueba más favorable logró una importante eliminación de DQO, pero no lo suficiente para que el efluente se descarga en el medio acuático, de acuerdo con la ley portuguesa.

Palabras clave: "Monopersulfato de potasio", "Oxidación química", "Demanda Química de Oxígeno",
“Aguas residuales de depuración de biodiesel”.

1. STATE OF ART

The untreated biodiesel contains several impurities, which will impact on the performance and durability of the diesel engine. Therefore, the purification stage is essential. The more traditional purification method is wet washing. However, the inclusion of additional water to the process offers many disadvantages, including the generation of a highly polluting wastewater that needs to be treated prior to environmental discharge and the significant loss of biodiesel into the wastewater phase. This type of effluent has quality parameters much higher than allowed by the portuguese legislation, Decree Law 236/98 of 1st August.
Several strategies based on physical, chemical and biological treatments have been purposed to treat this type of effluent (Rattanapan, Sawain, Suksaroj & Suksaroj, 2011; Pardal *et al*, 2014; Borralho, Pardal, Carvalho, Almeida & Baltazar, 2015). However, none of the processes reduced the Chemical Oxygen Demand (COD) levels sufficiently to allow direct discharge. The use of persulphate salts (Na_2S2O_8, K_2S2O_8 and $KHSO_5$), is an emerging technology in situ of chemical oxidation of organic compounds (Wei et al., 2015). This compound can directly react with organic molecules with a redox potencial comparable to hydrogen peroxid (Hussain, Green & Ahmed, 2013; Solís, 2017). In addition to direct oxidation, the persulphate can be induced to form sulphate radicals, thereby providing mechanisms for reaction with free radicals like to the hydroxyl radicals. Activation of persulphate to form sulphate radicals is a potent remediation tool for a wide variety of contaminants (Rivas, Gimeno & Borralho, 2012).
Monopersulphate (HSO_5^-) is the active ingredient of a triple potassium salt, $2KHSO_5KHSO_4K_2SO_4$ (Oxone). At pH above the 8-9, monopersulphate, (pKa=9.13), dissociates and the predominant specie is SO_5^{2-}, which, apparently, reacts with ionic forms of organic matter (Solís, 2017). It also can be induced by photolytic, catalytic and /or photocatalytic processes to form sulphate radicals (Chen et al., 2012; Wang & Chu, 2012; Chi et al., 2016) thereby providing mechanisms for reaction with some advantages comparing to hydroxyl radicals (Hu & Long, 2016; Oh, Dong & Lim, 2016). Oxone can also be decomposed into radicals by means of transition metals such Co(II), Ru(II) and Mn(II) (Rodriguez- Chueca et al., 2017), according Eq.1:

$$HSO_5^- + M^{n+} \rightarrow SO_4^{\cdot -} + M^{(n+1)+} + OH^- \quad \text{(Eq.1)}$$

M= Co(II); Co(II), Ru(II) and Mn (II)
Moreover, Oxone is relatively stable at room temperature and easy to handle since is available in powder form (Rodriguez-Chueca et al., 2017).
So, this work was intended to study the effect of the potassium monopersulphate compound in the removal of chemical oxygen demand (COD) of wastewater from the biodiesel purification stage. Two types of tests were performed; direct oxidation and oxidation using the metal catalyst Co (II).

2. METHODS

In this work the effect of the potassium monopersulphate compound ($2KHSO_5.KHSO_4K_2SO_4$), (Oxone) on the removal of COD in residual water from the biodiesel purification step was tested.

2.1. Sample

Table 1 summarises the physicochemical characteristics of the biodiesel wastewater (BWP) used, that it was supplied by PRIO (Prio Energy Co).

Borralho, M. T., Pardal, A. & Coelho, S. (2019).
Wastewater treatment of biodiesel production using persulphate ion as an oxidant. Millenium, 2(8), 21 -27.
DOI: https://doi.org/10.29352/mill0208.02.00199

Table 1. Biodiesel wastewater physicochemical characteristics.

Parameter	Biodiesel wastewater
pH	3.0
Redox Potential (mV)	360
Conductivity (µS/cm)	190
Turbidity (NTU)	0.36
COD (g O_2/L)	667
Fats (mg/L)	17

2.2 Data collection instruments

The pH and Redox Potential were determined by the potentiometer pH – Metron 654 pH meter and WTW- Inolab potentiometer respectively, while Conductivity was measured by the conductivity meter Methohm and the Turbidity using a WTW Turb.550 turbidimeter. The absorbance at 254nm was measured using a UV/Visible espectrofotometer Pharmacia Biotech Ultraspec 2000. In the determination of Sulphates it was used the furnace, Thermo – Heraeus, model K 114.

The reagents used in this work were:

Sulfuric acid, H_2SO_4, 95-98%, (Panreac); Ultrapure water, Mili-Q. Resistivity 10 MΩ cm; Sodium hydroxide, NaOH, 98%, (Panreac); Potassium iodate, $KI0_3$, (Panreac); Potassium iodide, KI, 99.0-100.5%, (Panreac); Ammonium molybdate, $(NH_4)_2MoO_4.4H_2O$, 93.1-101.0% (Panreac); Oxone, $2KHSO_5.KHSO_4.K_2SO_4$, 99.9%, (Sigma Aldrich) ; Cobalt sulphate, $CoSO_4$ $7H_2O$, (Sigma Aldrich); Sodium thiosulphate, $Na_2S_2O_3$, 98-102%, (Panreac); Cloridric acid, HCl, 36% (Panreac); Barium chloride, $BaCl_2.H_2O$ (Panreac).

All reagents used were analytical grade.

2.3 Analytical determinations

The experiments were carried out in alkaline medium, so the pH was maintained (9≤pH≤10) with addition of NaOH. For both treatments (direct oxidation and catalytic oxidation) several concentrations of Oxone were tested and its evolution over time of reaction was monitored using the iodometric method. This method consists of adding to the sample an excess of iodide which is titrated with sodium thiosulphate solution.

Thus, 5 mL of sample was taken, 10 mL of H_2 SO_4, 2.0 N, 25 mL of KI (20 g/L) and a few drops of saturated ammonium molybdate solution were added, which acts as a catalyst. The reaction that occurs is:

$HSO_5^- + 2I^- + 2H^+ \rightarrow I_2 + H_2O + HSO_4^+$ (Eq.2)

Then it is titrated with sodium thiosulfate, previously measured with KIO_3 and the desired concentration is obtained by:

$C_{HSO5^-} = (V_{S_2O_3^{2-}} . C_{S_2O_3^{2-}}) / 2V_{sample}$ (Eq.3)

Where $V_{S_2O_3^{2-}}$ is the volume of thiosulphate spent in the titration, $C_{S_2O_3^{2-}}$ is the concentration of the thiosulphate solution used and V_{sample} is the volume of the sample.

The evolution of the organic matter degradation was carried out by COD determination (APHA 2012).

The sulphates were determined using the gravimetric method (APHA 2012).

2.4 Procedures

All the experiments were carried out in triplicate and values presented are the average of the results.

The observed standard deviation was always less than 5% of the reported value.

Different physic-chemical parameters such as pH, Conductivity, Redox potential, Turbidity and COD were analyzed for the sample characterization.

Both experiments were carried out in a discontinuous way using 300 mL of BWP with stirring. The assays started after the addition of different dosages of (i) Oxone and (ii) Oxone and Co (II) depending of the type of test (direct oxidation or catalytic oxidation, respectively). Sodium hydroxide had been added to maintain the pH (9≤pH≤10) along the assays.

Samples were withdrawn over time of the assay to analyze the evolution of the concentration of Oxone and the pH.

COD was analyzed at the end of each treatment, to assess the respective removal efficiency.

Finally, for the assay that reached the best removal of COD were measured the absorvance at 254 nm and the sulphates.

Borralho, M. T., Pardal, A. & Coelho, S. (2019).
Wastewater treatment of biodiesel production using persulphate ion as an oxidant. *Millenium, 2*(8), 21-27.
DOI: https://doi.org/10.29352/mill0208.02.00199

3. RESULTS

Direct oxidation results are shown in figure 1:

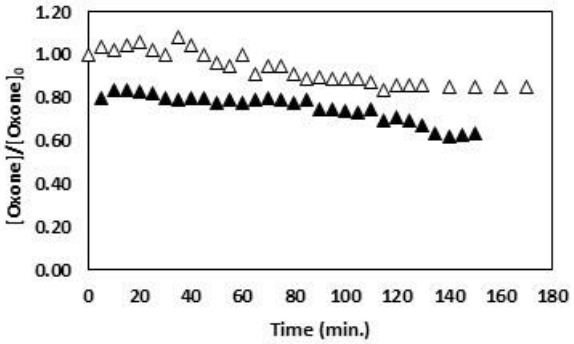


Figure 1 – Evolution of normalised concentration of Oxone in direct oxidation.
Experimental conditions: Empty symbols-$[Oxone]_0$= $4.00x10^{-3}$ M; Full symbols- $[Oxone]_0$= $1.00x10^{-2}$ M; 9<pH<10; T= 25ºC.

Next, it was studied the effect of the catalyst Co (II) into the process of decomposition of Oxone and were made different combinations of oxidant and catalyst.
Figure 2 shows the results obtained:

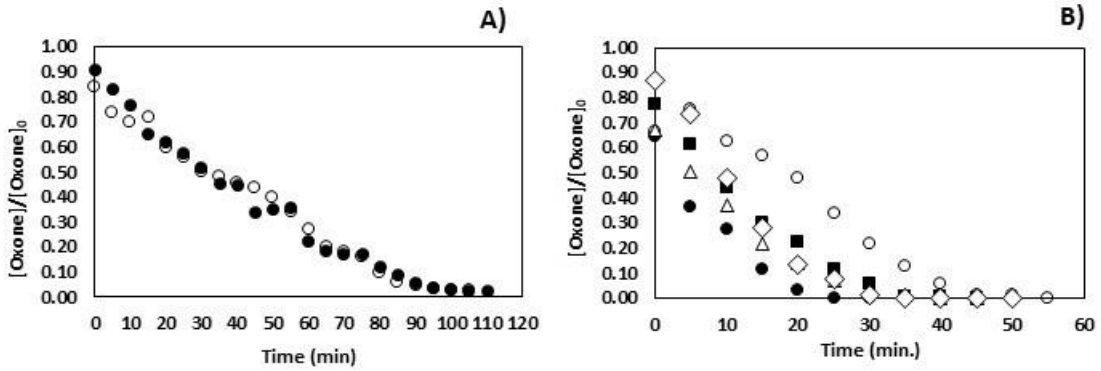


Figure 2 – Evolution of normalized concentration of Oxone for catalytic assays. Experimental conditions at A: ●-$[Oxone]_0$=$1.00x10^{-2}$M:[Co(II)]=0.1µM; ○-$[Oxone]_0$=$5.00x10^{-3}$M:[Co(II])=0.1 µM. Experimental conditions at B: ○-$[Oxone]_0$=$1.00x10^{-2}$M: [Co(II])=1.00µM:●[-$Oxone]_0$=$2.00x10^{-2}$M: [Co(II)]=1.00µM;▪-$[Oxone]_0$=$3.00x10^{-2}$M:[Co(II)]=1.00µM; Δ-$[Oxone]_0$=$5.00x10^{-2}$ M:[Co(II)]=1.00 µM; ◊-[$Oxone]_0$=$7.50x10^{-2}$ M[:Co(II)]=1.00 µM; 9<pH<10; T= 25ºC.

The results of COD removal for all experiments are in figure 3.

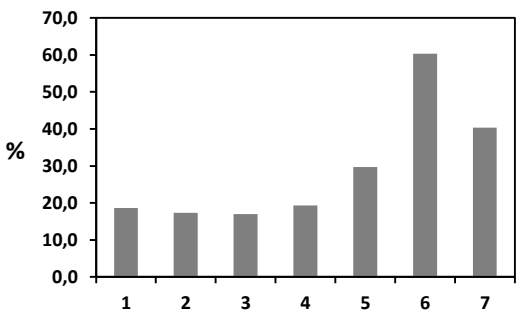


Figure 3 – COD removal rates for the different catalytic assays. Experimental conditions;1-$[Oxone]_0$=$5.00x10^{-3}$ M: [Co(II)]=0.1µM; 2-$[Oxone]_0$=$1.00x10^{-2}$M: [Co (II)]=0.1µM; 3-$[Oxone]_0$=$1.00x10^{-2}$ M: [Co(II)]=1.00µM; 4-$[Oxone]_0$=$2.00x10^{-2}$ M: [Co(II)]=1.00µM; 5- $[Oxone]_0$=$3.00x10^{-2}$ M: [Co(II)]=1.00 µM; 6- $[Oxone]_0$=$5.00x10^{-2}$ M: [Co(II)]=1.00 µM; 7-$[Oxone]_0$=$7.50x10^{-2}$ M:[Co(II)]=1.00 µM; 9<pH<10; T= 25ºC.

Borralho, M. T., Pardal, A. & Coelho, S. (2019).
Wastewater treatment of biodiesel production using persulphate ion as an oxidant. Millenium, 2(8), 21 -27.
DOI: https://doi.org/10.29352/mill0208.02.00199

At 254 nm the aromatic and, in general, the unsaturated compounds have a high molar extinction coefficient and consequently high absorbance. Thus, given the proportionality between this and the concentration, it can be said that the measurement obtained at this wavelength is indicative of the total amount of aromatic and unsaturated compounds present.
So, for the best assay, ($[Oxone]_0$= 5.00x10^{-2}M and [Co (II)]= 1.00 µM), the absorbance at ʎ= 254 nm was measured. The results are in figure 4.

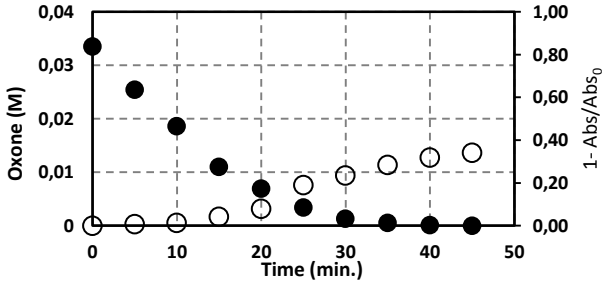


Figure 4 – Evolution of Oxone and absorbance (ʎ= 254 nm) along de time.
Experimental conditions: Full Symbol- $[Oxone]_0$=5.00x10^{-2} M: [Co(II)]=1.00x10^{-6} M; Empty Symbol-Absorbance (ʎ= 254 nm).

4. DISCUSSION

The results show that the direct oxidation tests for the potassium monopersulphate concentrations of 1.00x10^{-2} M and 4.00x10^{-3} M were not effective because there was no significant decomposition over more than three hours (Figure 1.), but after 2 days of reaction the monopersulphate suffered an abatement of 70% for the lower concentration tested.
The removal of COD was almost nonexistent, for these the assays (results not shown). The direct oxidation reported by some authors (Yu, Cui & Zhang, 2010; Rivas et al., 2012) and /or the initiation of the sulphate radical generation process by alkaline conditions (Guan, Ma, Li, Fang & Chen, 2011), seems to be a very slow process.
Co(II) has been reported as one of the most effective metal catalyst for the activation of HSO_5^-, promoting a radical sulphate complex mechanism (Sun, Li, Feng & Tian, 2009; Rivas et al., 2012).
The reduction of Co(III) to Co(II) mediated by the oxidation of monopersulphate is thermodynamically feasible (0.82 V) and fast, and the process proceeds cyclically many times until monopersulphate is totally consumed (Rodriguez- Chueca et al., 2017). Actually, all catalytic assays showed total decomposition of the oxidant for contact times ranging from 15min. to 2 hours depending of the initial concentration (Figure2). The figure 3 shows the COD removal rates that were variable and assumed values between 17% and 60%.
The treatments were started with cobalt concentrations of 0.10 µM, figure 2A, but the difficulty in dosing the compound led to its increase to 1.00 µM which significantly increased the rate of Oxone decomposition, Figure 2B. Obviously, for identical concentrations of Oxone tested (1.00x10^{-2} M) COD removal was identical too (Figure 3).
An increase in the initial Oxone concentration had exert a positive effect on COD removal until reach an optimum value (60 %) after what it slightly started to be negative. This result was also mentioned by (Solís, 2017).
The most effective test in terms of removal of COD was the combination of $[Oxone]_0$ = 5.00 x 10^{-2} M; [Co(II)]=1.00 µM, that reached a COD removal near of 60%, assuming the value of 260 g /L. This result shows the efficacy of the monopersulphate into oxidate organic matter, but due to the large amount in the raw wastewater the final COD concentration is still above to the desired one. The Portuguese legislation, Decree-Law 236/98 of 1st August, indicates the parametric value of 150 mg O_2/L as the discharge limit in a hydric medium. Anyway, the decrease of absorbance, (Figure 4), may indicate that the organic matter may be being decomposed into simpler molecules.
As monopersulphate is a source of sulphates they were measured (2000 mg/L SO_4^{2-}) for the most favorable test. Although elevated, still are within the values allowed by the Portuguese legislation (2000 mg /LSO_4).
In summary, the direct oxidation $HSO5^-$/Co(II) treatment could be an interesting alternative for the treatment of this wastewater as a pre- or post-treatment process, alone or in combination with a biological system. In the latter case a biodegradation study should be recommended.

CONCLUSIONS

The BWP has COD values much higher than allowed by law. It is therefore imperative to treat them before being discharged into the hydric medium.
The direct oxidation with monopersulphate showed that decomposition was very slow, so there wasn't COD removal for the times of reaction monitorized. On the other hand, the use of Oxone with de catalyst cobalt seems to be a suitable alternative

to oxidize organic matter. Oxone improves catalytic oxidation of COD. The most effective test reached a COD removal rate near 60%, assuming the value of 260 g /L. This value is still far from the indicate for portuguese legislation as the discharge limit value in a hydric medium.
This treatment may to be a previous or post step to a treatment of this type of wastewater. The sulphates and cobalt must, also, be monitorizated.

REFERENCES

APHA (2012). Standard Methods for the Examination of Water and Wastewater, American Public Health Association, Washington DC, USA.

Borralho, T., Pardal, A., Carvalho; F., Almeida; A., Baltazar; A. (2015). Caracterização e Tratamento de Água Residual Proveniente da Produção de Biodiesel - Estudo Preliminar. 1st National Congress of Higher Schools of Agriculture. Bragança, 2-3 d December.

Chen, X., Wang, W., Xiao, H., Hong, C., Zhu, F., Yao, Y., Xue, Z. (2012). Accelerated TiO_2 photocatalytic degradation of acid orange 7 under visible light mediated by peroxymonopersulphate. Chemical Engineering Journal, 193-194, 290-295.

Chi, F.L., Zhou, G.O., Song, B., Yang, B., Lv, Y.H., Ran, S.L., Li, C.G. (2016). $CoTiO_3$ nanoparticules as a highly active heterogeneous catalytic of monoperoxymonpersulphate for the degradation of organic pollutants under visible- light illumination. Journal of Nano Research, 42, 73-79.

Decree- Law 236/98 of 1st August.

Guan, Y.H, Ma, J., Li, X.C, Fang, J.Y., Chen, L.W. (2011). Influence of pH on the Formation of Sulfate and Hydroxyl Radicals in the UV/Peroxymonosulfate System. Environmental Science & Technology, 45:9308–9314.

Hu, P., Long, M. (2016). Cobalt-calatysed sulphate radical-based advanced oxidation a review on heterogeneous catalysis and applications. Applied Catalysis B: Environmental, 181, 103.

Hussain, H., Green I.R., Ahmed, I. (2013). Journey describing applications of oxone in synthetic chemistry. Chemical Reviews, 113, 3329-3371.

Oh, W.D., Z. Dong, Lim, T.T. (2016). Generation of sulphate radical through heterogeneous catalysis of organic contaminants removal: Current development, challenges and prospects. Applied Catalysis B: Environmental, 194, 169.

Pardal, A., Borralho, T., Carvalho, F., Chaves H., Baltazar, A., Catarro, J. (2014). Study of Integrated Chemical Physical Processes for Biodiesel Wastewater Treatment. 9º IWA World Water Congress – 20th-26th Setember, Lisboa.

Rattanapan, C., Sawain, A., Suksaroj, T., Suksaroj, C. (2011). Enhanced efficiency of dissolved air flotation for biodiesel wastewater treatment by acidification and coagulation processes. Desalination, 280 (1–3) 370-377.

Rivas, F.J., Gimeno, O., Borralho, T. (2012). Aqueous pharmaceutical compounds removal by potassium monopersulphate. Uncatalyzed and catalyzed semicontinuous experiments". Chemical Engeneering Journal, 192, 326-333.

Rodriguez- Chueca, J., Amor, C., Silva, T., Dionysiou, D.D., Li Puma, G., Lucas, M.S., Peres, J.A. (2017). Treament of winery wastewater by sulphate radicals: HSO5-/ transition metal/UV-A- LEDS. Chemical Engineering Journal, 310, 473-483.

Solís, R. R. (2017). Photolytic Oxidation of aqueosus herbicides with radiation of low energy. Application of catalyts, ozone and peroxid promoters. Tesis doctoral. Departamento de Ingeniería química y Química Física. Universidad de Extremadura (Espãna).

Sun, J., Li, X., Feng, J., Tian, X. (2009). Oxone/Co^{2+} oxidation as an advanced oxidation process: comparison with traditional Fenton oxidation for treatment of landfill leachate, Water Research, 43 4363–4369.

Wang, H. R., Chu, W. (2012). Photo-assisted degradation of 2,4,5-trichlorophenoxyacetic acid by Fe(II)-catalyzed activation of Oxone process: The role of UV irradiation, reaction mechanism and mineralization . Applied Catalysis B: Environmental, 123-124(23), 151-161.

Wei, G., Liang, X., He, Z., Liao, Y., Xie, Z., Liu, P., Ji, S., H. He, H., Li, D., Zhang, J. (2015). Heterogeneous activation of Oxone by substituted magnetites $Fe_3M_xO_4$ (Cr, Mn Co, Ni) for degradation of Acid Orange II at neutral pH. Journal of Molecular Catalysis A: Chemical, 398, 86–94.

Yu, J., Cui,J., Zhang, C. (2010). A simple and effective method for α-hydroxylation of β-dicarbonyl compounds using oxone as an oxidant without a catalyst. European Journal of Organic Chemistry 7020–7026.

ENGENHARIAS, TECNOLOGIA, GESTÃO E TURISMO
ENGINEERING, TECHNOLOGY, MANAGEMENT AND TOURISM
INGENIERÍA, TECNOLOGÍA, ADMINISTRACIÓN Y TURISMO

millenium 29

Millenium, *2*(8), 29-35.

en

INDICADORES DE QUALIDADE DO SERVIÇO DA ERSAR – CASO PRÁTICO DE UM MUNICÍPIO

ERSAR SERVICE QUALITY INDICATORS – PRACTICAL CASE OF A MUNICIPALITY

INDICADORES DE CALIDADE SEVICIO ERSAR – CASO PRÁTICO DE UN MUNICÍPIO

Ana Pardal[1]
Catarina Miguel[1]

[1] *Polytechnic Institute of Beja, Department of Technology and Applied Sciences, Beja, Portugal*

Ana Pardal - anap@ipbeja.pt | Catarina Miguel - catarina.1994@hotmail.com

Corresponding Author
Ana Pardal
Instituto Politécnico de Beja
Rua Pedro Soares, Campus do Instituto Politécnico de Beja
Apartado 6155
7800-295 Beja
anap@ipbeja.pt

RECEIVED: 31th July, 2018
ACCEPTED: 27th September, 2018

Pardal, A. & Miguel, C. (2019).
ERSAR Service Quality Indicators - practical case of a Municipality. Millenium, 2(8), 29-35.
DOI: https://doi.org/10.29352/mill0208.03.00207

RESUMO

Introdução: Os serviços de resíduos são fundamentais nas sociedades modernas, pois contribuem significativamente para o desenvolvimento social e económico do país. Portanto, devem obedecer a um conjunto de princípios, de entre os quais a universalidade do acesso, continuidade e qualidade de serviço e eficiência e equidade de preços, constituindo-se em importante fator de equilíbrio social.
Do ponto de vista da regulação e avaliação da qualidade dos serviços públicos prestados aos consumidores, a Autoridade de Regulação de Serviços de Água e Resíduos (ERSAR) desempenha um papel de liderança.
Objetivos: Analisar os indicadores de qualidade de serviço (IQS) de um município de médio porte para o período de 2011 a 2016, que permitam avaliar, em termos técnicos, ambientais e económicos, um sistema municipal de gestão de resíduos sólidos.
Métodos: A avaliação dos serviços de gestão de resíduos sólidos urbanos (RSU) é realizada por meio de três grupos de indicadores de qualidade de serviço (indicadores de desempenho): indicadores que refletem a proteção dos interesses dos consumidores; indicadores que refletem a sustentabilidade da gestão de serviços e, por fim, indicadores que traduzem a sustentabilidade ambiental.
Resultados: Os resultados obtidos nos indicadores de qualidade de serviço para a avaliação dos objetivos do Plano Estratégico Português para Gestão de RSU (PERSU 2020) mostram que a cidade ainda tem que fazer um esforço para atingir os objetivos definidos para o setor até 2020.
Conclusões: No geral, a análise mostra que o sistema apresenta principalmente níveis médios e insatisfatórios de qualidade de serviço.

Palavras-chave: Gestão Municipal de Resíduos Sólidos, ERSAR, Indicadores de Qualidade de Serviço.

ABSTRACT

Introduction: Waste services are fundamental in modern societies as they contribute significantly to the social and economic development of the country. Therefore, they must comply with a set of principles, among which are the universality of access, continuity and quality of service and price efficiency and equity, constituting an important factor of social balance.
From the point of view of regulation and evaluation of the quality of public services provided to users, The Water and Waste Services Regulation Authority (ERSAR) plays a leading role.
Objectives: To analyze the service quality indicators (SQI) of a medium-sized city council for the period 2011 to 2016, which allow to evaluate in a technical, environmental and economic terms an municipal solid waste management system.
Methods: The evaluation of municipal solid waste (MSW) management services is carried out through three groups of quality of service quality indicators (performance indicators): indicators that reflect the protection of user interests; indicators that reflect the sustainability of service management and, finally, indicators that translate environmental sustainability.
Results: The results obtained in the service quality indicators for the evaluation of the goals of the Portuguese Strategic Plan for MSW Management (PERSU 2020) show that the city has yet to make an effort to meet the goals defined for the sector by 2020. **Conclusions:** Overall, the analysis shows that the system presents mainly levels medium and unsatisfactory quality of service.

Keywords: Municipal Solid Waste Management, ERSAR, Service Quality Indicators.

RESUMEN

Introducción: Los servicios de residuos son fundamentales en las sociedades modernas, pues contribuyen significativamente al desarrollo social y económico del país. Por lo tanto, deben obedecer a un conjunto de principios, entre los cuales la universalidad del acceso, continuidad y calidad de servicio y eficiencia y equidad de precios, constituyéndose en un importante factor de equilibrio social.
Desde el punto de vista de la regulación y evaluación de la calidad de los servicios públicos prestados a los consumidores, la Autoridad de regulación de los servicios de agua y de desechos (ERSAR) desempeña un papel de liderazgo.
Objetivos: Analizar los indicadores de calidad de servicio (IQS) de un municipio de mediano porte para el período de 2011 a 2016, que permitan evaluar, en términos técnicos, ambientales y económicos, un sistema municipal de gestión de residuos sólidos.
Métodos: La evaluación de los servicios de gestión de residuos sólidos urbanos (RSU) se realiza a través de tres grupos de indicadores de calidad de servicio (indicadores de desempeño): indicadores que reflejan la protección de los intereses de los consumidores; indicadores que reflejan la sostenibilidad de la gestión de servicios y, por último, indicadores que reflejan la sostenibilidad ambiental.

Resultados: Los resultados obtenidos en la calidad de los indicadores de servicio para la evaluación de los objetivos del Plan Estratégico para la Gestión de RSU portuguesa (PERSU 2020) muestran que la ciudad todavía tiene que hacer un esfuerzo para alcanzar los objetivos fijados para el sector en 2020.

Conclusiones: En general, el análisis muestra que el sistema presenta principalmente niveles medios e insatisfactorios de calidad de servicio.

Palabras clave: Gestión Municipal de Residuos Sólidos, ERSAR, Indicadores de Calidad de Servicio.

INTRODUCTION

In the past, "household wastes" practically were not a problem. Almost all of the materials used contained components of animal or plant origin that, once returned to earth, were naturally decomposed into their elemental constituents, re-integrating a new life cycle (Levis & Cabeças, 2006).
Throughout the twentieth century, all this scenario has changed, with the appearance and production of new materials resulting from technological development, and with society orientated by consumerist values, where the choice of products has become increasingly varied (Levis & Cabeças, 2006).
The management of municipal waste, from its production to its final destination, presupposes the systematic and in-depth knowledge of its characteristics, both quantitative and qualitative (Levis & Cabeças, 2006).
Solid waste management can be a challenge, taking into account:

- Population expansion and economic development;
- Risk to human health and the environment;
- Contaminating water, attracting insects and rodents, and increasing flooding due to blocked drainage canals or gullies;
- Safety hazards from fires or explosions;
- Increases GHG emissions, which contribute to climate changes.

Adequate waste management contributes to the preservation of natural resources, both at the level of prevention and through recycling and valorization, as well as other specific legal instruments, reflecting the importance of this sector, considered in its aspects, as environmental and as sector of economic activity and the challenges facing policy makers and all actors in the chain of management, from the Public Administration, through economic operators to citizens, in general, as producers of waste and indispensable agents of the continuation of these policies (APA, 2017).
In 2015, 4,765 million tonnes of MW were produced in Portugal, 1% more than in 2014, reversing the trend of decrease observed in previous years (close to those of 2012). For 2015, in Continental Portugal, a capitation of 459 kg/person.year was below the European average (474 kg/person.year). Including the amounts of the Autonomous Regions this value would increase to 460 kg/person.year (APA, Resíduos Urbanos Relatório Anual 2015, 2016).
This increase will be related to an improvement of the economic situation in Portugal, which seems to indicate that the objective of decoupling waste production from economic growth is not being achieved. On the other hand, it is also noted that measures to prevent waste production are not having the expected effectiveness (APA, 2016).

Regulation of the waste sector in Portugal

Regulation of the waste sector is key to management, environment or quality of service, in order to ensure acceptable conditions for users, the State and the environment. In addition, regulation becomes the sole verification by the State of investment in the construction of landfills, incineration plants and other equipment remunerated with state money and the approved by Decree-Law nº 379/93, of 5 November (Silva, Ferreira, & Brás, 2015).
In recent years, the sector of urban waste services has undergone major developments, setting ambitious targets for the treatment of waste produced. This development has resulted in increasingly demanding collection and treatment of waste, economically and in terms of the demand for an efficient service. Therefore, it is necessary to evaluate the performance of the Management Entities (EG), a task that has been assumed by The Water and Waste Services Regulation Authority (ERSAR), being the service quality indicators (SQI) a good quality assessment instrument, either for the Regulation Authority or for the management entities (ERSAR, 2017).
ERSAR is responsible for developing an assessment of the quality of service levels of all water and waste management entities, but also collecting, validating, processing and disseminating this information and preparing and publicizing comparative summaries in an annual report (ERSAR, 2017).

The experience acquired during the application of the 1st generation of the evaluation system, the need to prepare the 2nd generation for application to all management entities and the publication of the ISO 24500 standards related to performance evaluation of water services, important contributions to the improvement and consolidation of the system. The system of indicators is organized according

Pardal, A. & Miguel, C. (2019).
ERSAR Service Quality Indicators - practical case of a Municipality. Millenium, 2(8), 29-35.
DOI: https://doi.org/10.29352/mill0208.03.00207

to these standards which, although they cover only water supply and wastewater services, can also be applied to municipal waste management services (Cardoso, 2014).
Following the publication of the new strategic plans approved for the water sector (PENSAAR 2020) and for the waste sector (PERSU 2020), ERSAR carried out a deep reflection on the review of the quality of service evaluation system, to adapt the same to these plans.

1. METHODS

In order to have instruments to assess the service quality vis-a-vis the previously mentioned regulation goals three groups of service quality indicators were defined:

- Indicators that reflect the adequacy of the interface to users: this group of indicators aims to assess whether the service provided in the year under assessment was appropriate, particularly in terms of greater or lesser physical access and affordability to the service and the quality with which it is provided; this group is further subdivided into the two aspects mentioned: accessibility of the service and quality of the service provided.
- Indicators that reflect the sustainability of service management: this group of indicators aims to assess whether basic measures are being taken to ensure that the service provided is sustainable; this group is further subdivided into the aspects of economic sustainability of the service, infrastructural sustainability of the service and physical productivity of human resources.
- Indicators that reflect the environmental sustainability: this group of indicators aims to assess the level of protection of environmental aspects associated with the operator's activities; this group is further subdivided into efficiency aspects in the use of environmental resources and the prevention of pollution (ERSAR, 2017).

Sixteen indicators were defined to assess the quality of the municipal waste management service (listed in Table 1), indicating their scope of application depending on the type of system. The indicators are calculated according to the ERSAR technical guides (ERSAR, 2017b; ERSAR, 2017).
Table 2 shows the Reference values, for each indicator, for retail systems.

Table 1. Municipal waste management service quality indicators

	MUNICIPAL WASTE MANAGEMENT SERVICE QUALITY INDICATORS				Bulk	Retail
Protection of user interests	**Accessibility of the service to users**					
	RU01 – Service coverage (%)				•	•
	RU02 – Selective collection coverage (%)				•	•
	RU03 – Affordability of the service (%)				•	•
	Quality of the service provided					
	RU04 – Washing of containers (-)				•	•
	RU05 – Response to complaints and suggestions (%)				•	•
Operator sustainability	**Economic sustainability**					
	RU06 – Cost recovery ratio (%)				•	•
	Infrastructural sustainability					
	2nd generation			**3th generation**		
		Bulk	**Retail**		**Bulk**	**Retail**
	RU07 – Packaging waste recycling (%)	•	•	RU07- Recycling of selective waste collection (%)	•	•
	RU08 – Organic waste recovery (%)	•	n.a.	RU08 - recycling of undifferentiated waste (%)	•	•
	RU09 – Incineration (%)	•	n.a.	RU09 - Waste recovery by TMB (%)	•	n.a.
	RU10 – Landfill use (%)				•	n.a.
	RU11 – Renewal of waste collection vehicles (km/vehicle)				•	•
	RU12 – Efficient use of waste collection vehicles [kg/(m^3 · year]				•	•

Pardal, A. & Miguel, C. (2019).
ERSAR Service Quality Indicators - practical case of a Municipality. *Millenium, 2*(8), 29-35.
DOI: https://doi.org/10.29352/mill0208.03.00207

	MUNICIPAL WASTE MANAGEMENT SERVICE QUALITY INDICATORS		Bulk	Retail
	Physical productivity of human resources			
	RU13 – Adequacy of human resources (No./1000 t)		•	•
Environmental sustainability	**Efficiency in the use of environmental resources**			
	RU14 – Use of energy resources	(kWh/t)	•	n.a.
		(tep/1000t)	n.a.	•
	Efficiency in the prevention of pollution			
	RU15 – Quality of the leachate after treatment (%)		•	n.a.
	RU16 – Greenhouse gas emissions (kg CO_2/t)		•	•
	n.a. – not applicable			

Table 2 -Reference values, for each indicator, for retail systems

	MUNICIPAL WASTE MANAGEMENT SERVICE QUALITY INDICATORS		Reference values for retail system (for predominantly rural intervention areas)	
Protection of user interests	**Accessibility of the service to users**			
	RU01 – Service coverage (%)		Good service quality [80; 100] Average service quality [70; 80[Unsatisfactory service quality [0; 70[	
	RU02 – Selective collection coverage (%)		Good service quality [70; 100] Average service quality [50; 70[Unsatisfactory service quality [0; 50[	
	RU03 – Affordability of the service (%)		Good service quality [0; 0.50] Average service quality [0.50; 1.00[Unsatisfactory service quality [1.00; +∞[	
	Quality of the service provided			
	RU04 – Washing of containers (-)		Good service quality [6.0; 24.0] Average service quality [4.0; 6.0[or [24.0; 28.0[Unsatisfactory service quality [0.0; 4.0[or [30.0; +∞ [	
	RU05 – Response to complaints and suggestions (%)		Good service quality 100 Average service quality [85; 100[Unsatisfactory service quality [0; 85[	
Operator sustainability	**Economic sustainability**			
	RU06 – Cost recovery ratio (%)		Good service quality [100; 110] Average service quality [90; 100[or]110.0; 120.0[Unsatisfactory service quality [0; 90[or]120.0; +∞ [	
	Infrastructural sustainability			
	2nd generation		**3th generation**	
	RU07 – Packaging waste recycling (%)	Good service quality [95; +∞] Average service quality]90; 95] Unsatisfactory service quality [0; 90]	RU07- Recycling of selective waste collection (%)	Good service quality [100; +∞] Average service quality]90;100] Unsatisfactory service quality [0; 90[
	RU08 – Organic waste recovery (%)	n.a.	RU08 - recycling of undifferentiated waste (%)	Good service quality [7; +∞] Average service quality]5;7[Unsatisfactory service quality [0; 5[
	RU09 – Incineration (%)	n.a.	RU09 - Waste recovery by TMB (%)	n.a.
	RU10 – Landfill use (%)		Good service quality [24; +∞] Average service quality [18;7[Unsatisfactory service quality [0; 18[	
	RU11 – Renewal of waste collection vehicles (km/vehicle)		Good service quality [0; 250 000] Average service quality]250 000; 350 000 [Unsatisfactory service quality]350 000; +∞ [	
	RU12 – Efficient use of waste collection vehicles [kg/(m^3 · year]		Good service quality [400; 500[Average service quality]300; 400[and]500; 550[	

Pardal, A. & Miguel, C. (2019).
ERSAR Service Quality Indicators - practical case of a Municipality. Millenium, 2(8), 29-35.
DOI: https://doi.org/10.29352/mill0208.03.00207

	MUNICIPAL WASTE MANAGEMENT SERVICE QUALITY INDICATORS	Reference values for retail system (for predominantly rural intervention areas)
		Unsatisfactory service quality [0; 350[and [550; +∞[
	Physical productivity of human resources	
	RU13 – Adequacy of human resources (No./1000 t)	Good service quality [1.0; 3.0[Average service quality]0.5; 1.0[and]3.0; 3.5[Unsatisfactory service quality [0.0; 0.5[and [3.5; +∞[
	Efficiency in the use of environmental resources	
	RU14 – Use of energy resources (tep/1000t)	Good service quality [0; 6,5] Average service quality]6,5; 7,5] Unsatisfactory service quality]7,5; +∞ [
	Efficiency in the prevention of pollution	
	RU15 – Quality of the leachate after treatment (%)	n.a.
	RU16 – Greenhouse gas emissions (kg CO_2/t)	Good quality service [0; 15] Average quality service]15; 18] Unsatisfactory quality service]18; +∞ [
	n.a. – not applicable	

2. RESULTS

Table 3 presents the performance indicators for the years 2011 to 2015 and the performance indicators available for 2016, for the medium-sized city council studied. The Municipality has about thirty-six thousand inhabitants and an area about one thousand km^2.

Table 3 - Values for evaluating service quality indicators in the period 2011 to 2016

		2011	2012	2013	2014	2015	2016
PROTECTION OF USER INTERESTS	**Accessibility of the service to users**						
	RU01 – Service coverage (%)	93 ●	94●	94●	90●	90●	90,4●
	RU02 –Selective collection coverage (%)	8●	88●	97●	81●	82●	b)
	RU03 – Affordability of the service (%)	0.10●	0.21●	0.17●	0.22●	0.25●	b)
	Quality of the service provided						
	RU04 – Washing of containers (-)	6●	4.3●	7.2●	3.7●	4.8●	5,6●
	RU05 – Reply to written complaints and suggestions (%)	94●	96●	100●	95●	92●	78,6●
OPERATOR SUSTAINABILITY	**Economic sustainability**						
	RU06 – Cost recovery ratio (%)	w.i.	0.5●	0,4●	NA	54●	w.i.
	Infrastructural sustainability						
	RU07 – Packaging waste recycling (%)	102●	94●	91●	88●	115●	w.i.
	RU08 – Organic waste recovery (%)	w.i.	w.i.	w.i.	w.i.	w.i.	w.i.
	RU09 – Incineration (%)	w.i.	w.i.	w.i.	w.i.	w.i.	w.i.
	RU10 – Landfill use (%)	w.i.	w.i.	w.i.	w.i.	w.i.	w.i.
	RU11 – Renewal of waste collection vehicles (km/vehicle)	174,406●	194,064●	218,356●	260,870●	277,570●	w.i.
	RU12 – Efficient use of waste collection vehicles [kg/(m^3 · year]	322●	313●	292●	308●	320●	316●
	Physical productivity of human resources						
	RU13 – Adequacy of human resources (No./1000 t)	NR	3.2●	3.1●	3.6●	3.8●	4●
ENVIRONMENTAL SUSTAINABILITY	**Efficiency in the use of environmental resources**						
	RU14 – Use of energy resources (kWh/t)	5●	5●	5●	5●	5,2●	5,2●
	Efficiency in the prevention of pollution						
	RU15 – Quality of the leachate after treatment (%)	w.i.	w.i.	w.i.	w.i.	w.i.	w.i.
	RU16 – Emissions of greenhouse gases (kg CO_2/t)	16●	15●	15●	16●	16●	16●
	● Good quality service ● Average quality service ● Unsatisfactory quality service n.a - not applicable w.i. - without information						

Based on the analysis of the previous table, which corresponds to the evaluation of the management entity and the Alentejo City Hall, there are indicators for which the evaluation is almost always unsatisfactory during the period considered, such as the profitability of the car park and the laundering containers.

This assessment may be due to the lack of staff to carry out these tasks, which is reflected in several indicators since without sufficient staff in the service can not achieve positive results.

On the other hand, it was verified that the indicators physical accessibility of the service, selective collection, economic accessibility of the service, use of energy resources presented satisfactory evaluation.

In the year 2015 there was improvement in the parameter of packaging waste recycling, which went from an unsatisfactory quality of service to a good quality of service.

CONCLUSIONS

The Urban Waste service sector has been undergoing major developments in recent years. In addition to the individual policies of each Member State, the European Union itself has defined policies that have been translated into ambitious goals in terms of UW treatment targets produced.

The situation of lack of competition and the very requirements to comply with the goals and objectives established in the PERSU2020 create a need to evaluate performance and ERSAR has assumed this task through regulation, first in the sector in "Bulk" and then in the sector in "Retail".

The regulation, through the evaluation of service quality indicators of the waste management systems, allows an up-to-date and accurate knowledge of the state of the services, providing an awareness of what is working satisfactorily and what can still be improved.

In general, it can be concluded that ERSAR has played a key role in promoting the continuous improvement of the quality levels of the waste services provided, however, the Management Entities should continue to make an effort that translates into better transparency, efficiency and effectiveness of management and support the implementation and enforcement of public policies at national and community level.

Regarding the actions of the Municipality, it can be mentioned that there is a concern for good management of the UW, namely in the acquisition of new containers for the disposal of waste.

However, there is still little participation of the population in the separation of waste.

In this work, the service quality indicators were analyzed from 2011 to 2016. It was verified that there are indicators with a satisfactory result, others with unsatisfactory result and indicators with a median result. The indicators of performance with unsatisfactory result are those that require more attention, as is the case of the washing of the containers of the Municipality, as mentioned this evaluation is due to the lack of personnel, that to go about collecting the waste, they cannot do the washes.

REFERENCES

APA. (2016). Obtido de apambiente: https://rea.apambiente.pt/node/66

APA. (2016). *Resíduos Urbanos Relatório Anual 2015.* Amadora: Agência Portuguesa do Ambiente, I.P.

APA. (2017). *apambiente*. Obtido em 19 de junho de 2017, de https://www.apambiente.pt/index.php?ref=16&subref=84

Cardoso, J. F. (2014). *Indicadores para a caracterização do setor dos resíduos e para a avaliação da qualidade do serviço prestado em Portugal.* Lisboa: Universidade Nova de Lisaboa .

ERSAR. (2017). *Water and waste services assessment guide - 2nd generation of the assessment system.* Lisbon.

ERSAR. (2017b). *Guia de Avaliação da Qualidade dos Serviços de Águas e Resíduos Prestados aos Utilizadores (3.ª geração do sistema de avaliação).* ERSAR.

Levis, J. d., & Cabeças, A. J. (2006). *Resíduos Sólidos Urbanos - Princípios e Processos* (1ªEdição ed.). Lisboa: AEPSA.

Silva, M. E., Ferreira, B. S., & Brás, I. P. (2015). Indicadores de Qualidade na Prestação de Serviços de Gestão de Resíduos Urbanos – Caso de Estudo. *Millenium, n.º 48 (jun/dez)*, 91-109.

ENGENHARIAS, TECNOLOGIA, GESTÃO E TURISMO
ENGINEERING, TECHNOLOGY, MANAGEMENT AND TOURISM
INGENIERÍA, TECNOLOGÍA, ADMINISTRACIÓN Y TURISMO

millenium 37

Millenium, 2(8), 37-52.

en

APLICAÇÃO DAS METODOLOGIAS LEAN NA REDUÇÃO DOS TEMPOS DE SETUP NA INDÚSTRIA FARMACÊUTICA

APPLICATION OF LEAN METHODOLOGIES IN THE REDUCTION OF SETUP TIMES IN THE PHARMACEUTICAL INDUSTRY

APLICACIÓN DE LAS METODOLOGÍAS LEAN EN LA REDUCCIÓN DE LOS TIEMPOS DE SETUP EN LA INDUSTRIA FARMACÉUTICA

André Gil Curado[1]

[1] *Instituto Superior de Engenharia de Lisboa, Lisboa, Portugal*

André Gil Curado - andregcurado@gmail.com

Corresponding Author
André Gil Curado
Rua Ilha da Graciosa nº 117 3ºB Carcavelos
2775-803 Cascais
andregcurado@gmail.com

RECEIVED: 17th October, 2018
ACCEPTED: 05th December, 2018

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

RESUMO

Introdução: A competitividade internacional acentuada motivada pela globalização com o objetivo de alcançar um público que cada vez se nota mais exigente, faz da diminuição de custo e aumento da qualidade uma necessidade primária transversal a qualquer setor. A indústria de medicamentos genéricos onde o preço é a arma principal no alcance do consumidor não é exceção e cria uma forte necessidade na aplicação das metodologias Lean com o intuito de otimizar a produção.
Objetivos: Reduzir os tempos de setup mediante a aplicação de metodologias Lean estruturado por um modelo proposto.
Métodos: No decorrer deste estudo foi revista a bibliografia referente à origem e várias metodologias Lean, sendo proposto um modelo de aplicação. A implementação teve por base um estudo detalhado das mudanças resultando num conjunto de propostas de solução dedicados aos principais problemas aplicando diversas ferramentas Lean.
Resultados: Redução dos tempos de mudança na ordem dos 20% e número de espera na ordem dos 24,5% no espaço de 3 meses.
Conclusões: A aplicação das metodologias Lean revelou uma grande utilidade na identificação das causas e na criação de soluções. A implementação de este modelo, ainda que numa fase inicial, permitiu não só redução dos tempos de mudanças, mas como outras melhorias como a diminuição do número de esperas. Contudo, esta aplicação, por estar inserido numa indústria farmacêutica contempla alguns procedimentos que não podem ser alterados limitando os tempos de mudanças.

Palavras-chave: Lean; Indústria Farmacêutica; Ferramentas Lean; SMED

ABSTRACT

Introduction: The strong international competitiveness motivated by globalization with the purpose of reaching an increasingly demanding public, makes cost reduction and quality increase a primary need across all sectors. The generic drug industry where price is the primary weapon in consumer reach is no exception and creates a strong need in the application of Lean methodologies to optimize the production.
Objectives: Reduce of changeover times by the application of lean methodologies structured by a proposed model.
Methods: This study started with a review of the bibliography of the Lean's origin and the several methodologies ending with the application of a proposed application model. The implementation was based on a detailed study of the setups resulting in a set of solution proposals dedicated to the main problems applying several lean tools.
Results: Reduction of change times by 20% and waiting times by 24.5% within 3 months.
Conclusions: The application of Lean methodologies revealed a great utility in the identification of causes and in the creation of solutions. The implementation of this model, although in an initial phase, allowed not only reduction of the times of changes, but also other improvements such as the reduction of the number of waits. However, this application, due to being inserted in the pharmaceutical industry, is limited by some procedures that cannot be changed.

Keywords: Lean; Pharmaceutical Industry; Lean Tools; SMED

RESUMEN

Introducción: La competitividad internacional acentuada motivada por la globalización con el objetivo de alcanzar un público que cada vez se nota más exigente, hace de la disminución de costo y aumento de la calidad una necesidad primaria transversal a cualquier sector. La industria de medicamentos genéricos donde el precio es el arma principal en el alcance del consumidor no es una excepción y crea una fuerte necesidad en la aplicación de las metodologías Lean con el fin de optimizar la producción.
Objetivos: Reducir los tiempos de setup mediante la aplicación de metodologías lean estructurado por un modelo propuesto.
Métodos: En el transcurso de este estudio se revisó la bibliografía referente al origen y a las varias metodologías Lean, siendo propuesto un modelo de aplicación. La implementación se basó en un estudio detallado de los cambios resultando en un conjunto de propuestas de solución dedicadas a los principales problemas aplicando varias herramientas lean.
Resultados: Reducción de los tiempos de cambio en 20% y tiempos de espera en 24,5% en el plazo de 3 meses
Conclusiones: La aplicación de las metodologías Lean reveló una gran utilidad en la identificación de las causas y en la creación de soluciones. La implementación de este modelo, aunque en una fase inicial, permitió no sólo reducir los tiempos de cambio, sino como otras mejoras como la disminución del número de esperas. Sin embargo, esta aplicación, debido por estar inserta en una industria farmacéutica, tiene algunos procedimientos que no pueden ser alterados limitando los tiempos.

Palabras Clave: Lean; Industria Farmacéutica; Herramientas Lean; SMED

INTRODUTION

Today we are in presence of a global market created by the evolution of transports and communications ways. Nowadays, customers demand a wide range of products delivered with high quality, quicker response times and sold at reasonable prices. The importance of short changeover times has become critical to rapidly respond to changes in customer requirements (Sullivan, McDonald, & Van Aken, 2002) and to be able to switch from one product to another, thus putting the enterprise in front of other competitors (Wallace & Choi, 2011). The benefits of the setup reduction are the increase of flexibility by conducting more changeovers and reducing lot size, increase bottleneck capacities in order to maximize the line availability and minimization of costs (Van Goubergen & Van Landeghem, 2002).

Lean Manufacturing offers the proper tools in order to reduce the waste inside and between the different processes while being able to increase the value of the product (Karam, Liviu, Cristina, & Radu, 2018). This idea was initiated in the post-war period by Taiichi Ohno and his peers and initially applied in Toyota, an automotive industry company. Nowadays it is seen with great interest by any field due to the excellent results demonstrated in Toyota (Jeffrey K. Liker, 2006). Lean thinking aims, within a continuous improvement cycle, to optimize resources and processes by reducing all activities that do not add value or are not fundamental to achieve this goal. This concept uses several methodologies that have been developed over the years and make this goal tangible, examples of these methodologies are Total Productive Maintenance, SMED, Value Stream Mapping or 6-sigma (Rauch, Damian, Holzner, & Matt, 2016).

The need of reducing wastes is increasingly important in production lines with a wide variety of product (B. S. Kumar & Abuthakeer, 2013) and generic-product industries are no exception. In this work it is presented an example carried out in the pharmaceutical industry in a company nationally leader in the production of generic drugs. This application of lean methodologies has the purpose of reducing the change-over time. In this analysis, is proposal for an implementation model which, structured in a PDCA cycle, applies several Lean methodologies to identify causes and define the best practices to reduce change-over times.

A packaging line setup belonging to the pharmaceutical plant, has unique characteristics compared with the setup of other traditional assembly lines (Bevilacqua, Ciarapica, Mazzuto, & Paciarotti, 2013). This article, using Shingo and McIntosh methods, seeks to answer the questions, what precautions needs to be taken in this industry? how can waste be identified? What lean methods can we use? How to fight the wastes found in the plant? Showing step by step an example of a study case. This article is initiated with the historical background of Lean thinking presenting then the Shingo and McIntosh methods on reducing setup time and the proposed method that is applied on the case study demonstrated.

1. HISTORICAL BACKGROUND OF LEAN THINKING

The designation of Lean Thinking was given by John Krafcik in 1988 in the research "Triumph of the Lean Production System" in a MIT program called International Motor Vehicle Program where James Womack was the researcher director. James Womack and Daniel Jones after several years studying the success of Japanese companies, described the emergence and evolution of this model of management in two publications “The machine That Changed the World” and “Lean Thinking” (Pinto, 2014). Since then, this term is used worldwide and progressing from the automotive industry to several areas such as tourism, medicine or construction industry.

The Lean Thinking concept was born in Japan in the 1940s by Taiichi Ohno, an industrial engineer at Toyota. In a post-war time with the country in a severe crisis, where there were great needs and scarce resources, Ohno finds himself obligated to take action by putting his own ideas into practice (Womack, 1990).

This new approach has revolutionized the way the organization thinks and behaves, Lean thinking is not just a set of practices but a profound cultural change, believing in a change that sustains the dynamics and the process of continuous improvement. Taiichi Ohno has revolutionized the industry in three fundamental points, people, processes and solutions (Pinto, 2014).

It took more than 20 years of effort to fully implement this whole set of ideas into Toyota's entire supply chain. By the 1990s, Toyota already offered such a vast product range as General Motors and as a Lean producer it only took half the time and effort to design a new car compared to other brands (Womack, 1990).

The validity of Lean principles and solutions is corroborated by the success of companies such as Toyota Motors Corporations, which reached the top of the automotive industry in 2007 by dethroning General Motors from the top position who was since 1930 the largest company in this industry (Schmitt, 2017).

Lean thinking is a management philosophy for maximizing value through the consistent reduction of waste. To do so, it uses a set of methodologies, techniques and tools oriented to the simplification and optimization of processes, removal of activities and resources that do not add value and the involvement of all members, in the constant improvement of the organizations performance (Rauch et al., 2016). These methods, techniques and tools have several applications having been developed over the decades in several countries by different companies, examples of these methodologies are 5’s, SMED, 6-sigma or Total Productive Maintenance.

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

2. REDUCING SETUP TIME

Changeover time is the period between the last good product produced and the first of the next production (Karam et al., 2018). Rapid tool change techniques were introduced and developed for the first time by Shingeo Shingo in the 1950s which, after 19 years, examining this methodology reported his work in the book "A revolution in manufacturing: the SMED system" (Dillon & Shingo, 1985).

During the following decades, the reduction of setup times continued to be addressed by several Japanese companies such as the case of Mitsubishi Heavy Industries Ltd. and M Electric Japan appearing numerous publications. It was only in the 1980s that this methodology was extended to European industries in part because of the success of the Toyota Production System (Sohani, 2011).

2.1 MODEL OF THE CASE STUDY

In his work, Shingo points out the need of a structure or a methodology that guides the improvement techniques. The proposed model aims to detail a cycle based on the Deming's PDCA cycle that structures an improvement application in the scope of reduction of the change-over times. This proposal is based on the reports and observations of two major publications, "Shingo's Revolution in Manufacturing: The SMED System" and S. Culley et al. "Improving Changeover Performance" in which the authors review Shingo's work and propose organization-led and design-led solutions.

This methodology is structured in 4 phases: "Analyze", "Interpret and Plan", "Create" and "Check" (Figure 1).

Analyze - The first phase of the cycle is to analyze in detail all the elements from the characteristics of the industry in which it is inserted up to the details of each activity. This cycle is based on 5 points, "Knowing", "Observe", "Measure", "Distinguish" and "Compare". "Knowing" refers to know all the steps of a changeover and to understand the logic behind it. The first step would be to define the concept of "value" and understand it is chain, defining the "value" may present the priorities explaining the precautions that need to exist in a setup. The second point "Observe" is to know the steps in detail, to develop this point, it is necessary to monitor and observe the changes, in the observe phase, filming may be appealing. One methodologies that can be used is 5w2h by questioning and understanding all aspects of a change. It is important to observe the sequence of operations pointing out which tasks are handled, when and by whom, the sequence of operations can be quite significant in the performance of a change (Culley, Owen, Mileham, & McIntosh, 2001). Sometimes the setups between the various products are different having different times. This point, "Distinguish", differentiates the several characteristics. "Measure" is one of the important points, measuring and recording all the components of a setup. The "Compare" phase is the comparison between measurements. It conveys the current state, tells the progress that is being made and allows to analyze the impact of the different factors involved in a setup.

Interpret and Plan - The interpretation of collected data allows to identify the gaps in the processes, exposing the causes and setting a way to follow in the reduction of waste. A tool widely used in this analysis is the Ishikawa diagram, a tool which helps identify the causes of the problems. This phase aims to create a plan by the improvement's opportunities identified with the analysis. The interpretation of the difficulties allows at in this point, the creation of solutions and indicates the plan to follow. SMED and TPM are two major tools that can be used in planning the change-over times reduction strategy.

Create - This point consists in putting into practice the plan created earlier to fight the problems identified. The creation of solutions involves two elements:

> Do - put into practice all physical solutions defined to fight the gaps identified. Methodologies as the application of 5's or Error Proofing can be used in this phase to support the streamlining of the external and internal activities.
> Form - putting into practice non-physical solutions such as training, which have a great impact on performance (Culley et al., 2001).

Check - Like the last point of a PDCA cycle, this point verifies quantitatively, the consequences of the applications as the case of times recorded, or qualitatively as in the case of the satisfaction of the employees which is significantly increased with the successful implementation of Lean applications like 5s or SMED (Boztinaztepe & Canan, 2008).

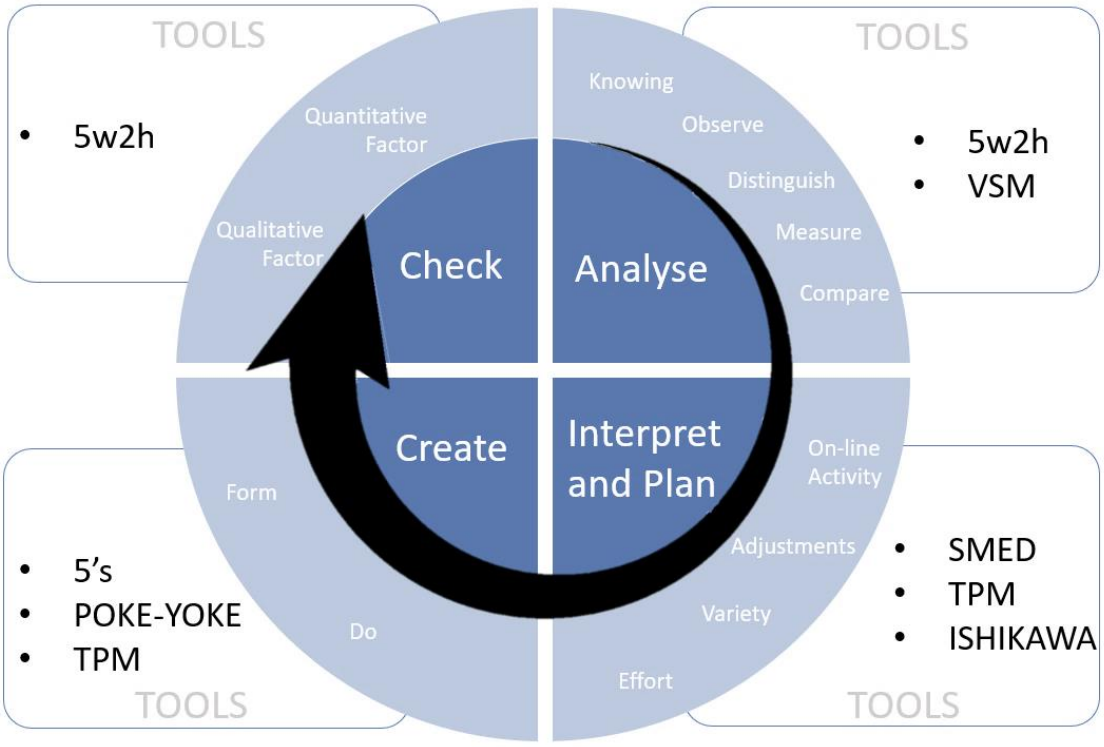


Figure 1 - Stages of the Case Study

3. LEAN TOOLS

According to some authors, SMED should be accompanied by other methods such as the application of 5s (Sohani, 2011) or TPM such as the use of OEE indicator (Karam et al., 2018). In this study case 6 different tools are used to facilitate the analysis of each step (Figure 1).

3.1 5w2h

5w2h is an introductory method of clarifying problems for the purpose of determining not only the root of the cause, but also facilitating the implementation of corrective and preventive actions (Nagyova, Palko, & Pacaiova, 2015). During the "Analyze" stage, this tool is used to study the setups.

3.2 Ishikawa Diagram

This method was developed by Ishikawa in the 1950s. Through this tool it is possible to analyze the possible causes of an effect such as a problem, a defect or a waste (Gwiazda, 2006). This method is used to identify the causes of the problems and points out solutions in the "Interpret and Plan" phase.

3.3 Value Stream Mapping

The Value Stream Mapping consists of recording the set of activities that create and deliver value to the customer and other stakeholders (Seth & Gupta, 2005). This methodology is used in the "Analyze" step to study the setup process.

3.4 SMED

The main tool used in this work to reduce setup time is SMED. The SMED approach is used in the phase "Interpret and Plan" to identify the activities that can be done while the machine is running. Single Minute Exchange of Die method was introduced by Shingo and consists of actions that aim at the systematic reduction of setup times to maximize the use of the components (Karam et al., 2018). The core of SMED is to reduce the time by performing as many activities as possible while the equipment is running and to simplify the remaining steps. The SMED method's objective is to systematically reduce setup times to maximize the use of the components. Shingo presented the SMED methodology with four stages that support the reduction of setup time: the preliminary stage where internal and external are not distinguished; separate internal and external setup activities, convert

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

internal setup activities to external ones; and streamline external and internal activities (Dillon & Shingo, 1985). The direct consequences of reducing changeover are reducing batch size, increasing flexibility, and reducing costs (Karam et al., 2018).
Although SMED is known as a successful method for more than twenty five years, (Ferradás & Salonitis, 2013) in his work, McIntosh et al. argue that the Shingo methodology, despite being supported by many examples, might not be the most efficient way. The authors refer that is given emphasis to low cost organization improvements and little attention to design changes. This practice can lessen the use of other important improvement options. McIntosh et al. affirm that improvement which frequently involves design changes that alter the nature of changeover tasks, (or eliminating some tasks) can have an equal or a greater result (McIntosh, Culley, Mileham, & Owen, 2000). In "Improving Changeover Performance", McIntosh at al. present organization-led and design-led solutions to overcome the changeover difficulties (Table 1) (Culley et al., 2001).

Table 1 - Improvement Techniques Source:(Culley et al., 2001) (adapted)

		Improvements	
		Organization-led Improvements *(Improvments by change the organazation)*	Design-led Improvements *(Improvments by changing the nature of the components)*
Dificulties	On-line Activity *(Time required in change-over activities)*	Move tasks into external time Conduct taks in parallel Break interdependencies beteween tasks and personnel *(Minimize the contribution of existing tasks by reallocating tasks)*	Mouting guards to complete manual tasks in parallel with automated tasks *(Minimize the contribution of existing tasks by changing the components permitting taks to be reallocated)*
	Adjustments *(Time spent in adjustments)*	Know of and use of predefined settings Define adjustments parameters Understand adjustments interrelationship Address precision requirements Damage Avoidance *(Minimize Adjustment tasks)*	Automate Adjustment Adress precision requirements Consider using position/condition monitoring equipment Make equipment more robust (less prone to damage) Upgrade item quality *(Change components to minimize time spent in adustment tasks)*
	Variety *(Obstacles imposed by variety)*	Implement standard changeover procedures Precheck that changeover items are present *(Standardize the way changeover is conducted)*	"Foolproof" Location Standardize machine system features *(Standardize physical conditions)*
	Effort *(Trouble created by the effort in performing some tasks)*	Eliminate superfluous tasks Employ the best tools/handling/storage aids Ensure cleanliness *(Eliminate or simplify existing tasks by working in a more efficient way)*	Add devices to aid existing tasks Modify hardware/product to aid existing tasks Improve access Avoid using hand tools *(Eliminate or simplify existing tasks by equipment or product modification)*

Shingo and McIntosh approaches lead to the minimization of change-over activities but with different perspectives, both methods are represented in the Figure 2.

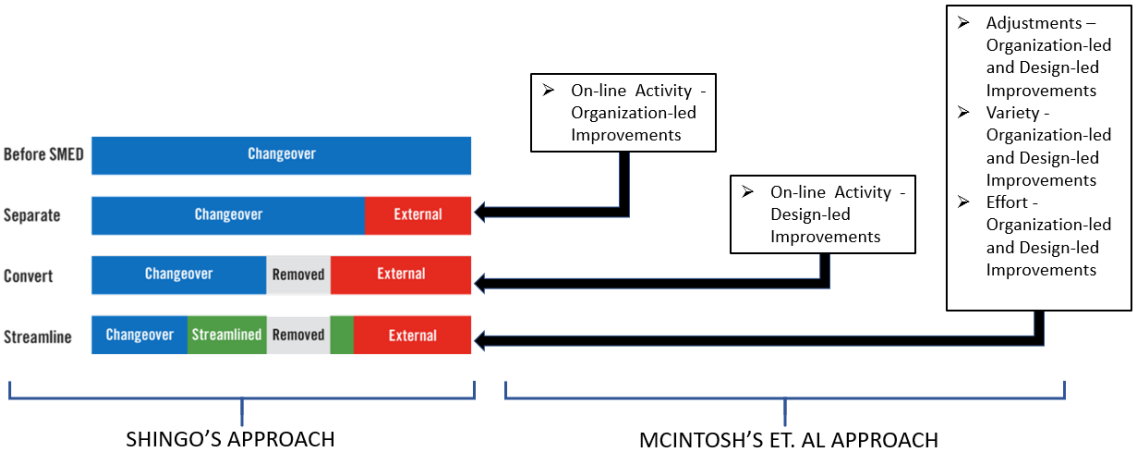


Figure 2 - Shingo and McIntosh approaches on setup time reduction

In Shingo's book, he demonstrates how the results of the SMED application go beyond reducing change times. Practitioners of this method can reap benefits such as increased productivity, increased operator performance, as well as quality and safety (Dillon & Shingo, 1985).

3.5 Total Productive Maintenance

TPM is a management approach that seeks the constant elimination of all forms of waste existing in the productive and administrative areas of the company. This concept was created in the 1960s by Nakajami in Japan and was initially developed to support the maintenance of the equipment and which later extended it is area of intervention to the whole process (Williamson, 2015). This methodology relies upon 6 elements: Productivity, Morale, Quality Delivery Safety and Cost (J. Kumar, Soni, & Agnihotri, 2014). This tool is used in the stage "Interpret and Plan" to analyze the productivity status translating the OEE values, and in the Create stage training the personnel to perform tasks.

3.6 5's

The 5's is a lean method and an improvement system adopted to reduce waste, clean the workspace and improve productivity. It is usually used to facilitate other lean methods. To this end, 5s aims to maintain order in the workplace and uses visual management to increase operational results (Al-Aomar, 2011). During the create stage, 5's techniques were applied to standardize the setup process.

3.7 Error-proofing

Error-proofing methods were introduced by Shingeo Shingo in Japan in the 1960s and are based on the identification and prevention of errors or defects in processes. These methods have several denominations also known as mistake proofing or fool proofing and are often covered in their Japanese designation poka-yoke, meaning "error-proof system" (Tommelein & Ballard, 1999). This method is used in the "Create" phase to create mechanisms to prevent errors.

4. CASE STUDY

The implementation was performed on one of the lines of the packaging department of a pharmaceutical company that produces a wide range of generic drugs for domestic and international market. This line has 2 machines, the blister machine and the packing machine and has team of 6 people working in shifts of two people per shift.

4.1 Analyse

The application of the model was initiated with the definition of value and the identification of the value chain of the project in question (Figure 3). In this case, the "value", regarding the industry in which these changes are inserted, is defined by the modification of the necessary components in a fast way never jeopardizing the health of employees or customers.

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

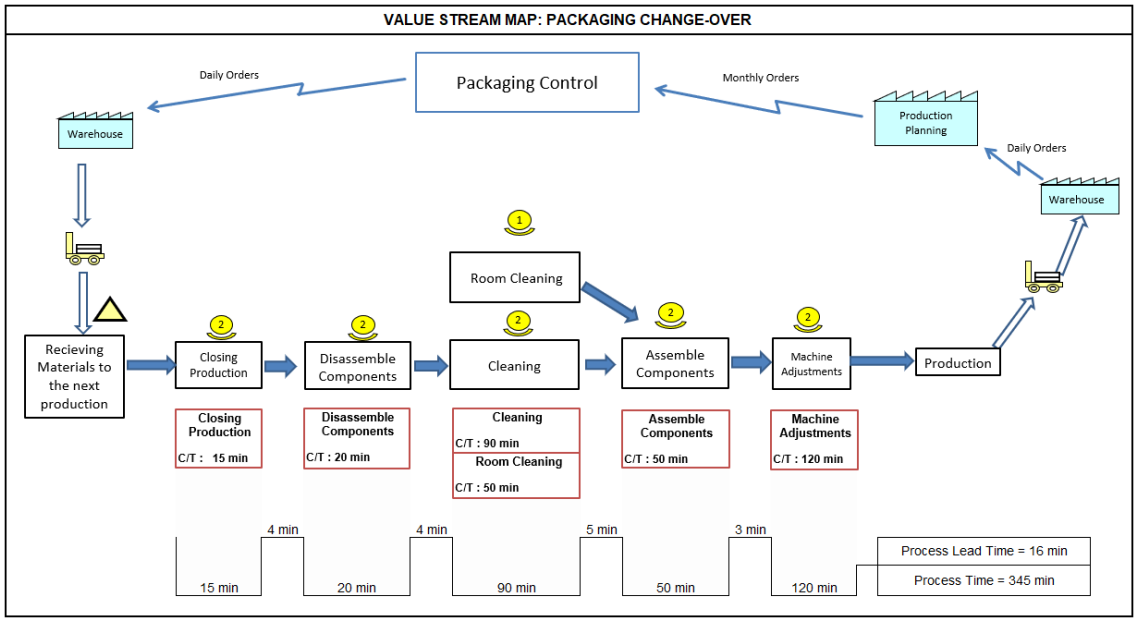


Figure 3 - Value Stream Map Packaging Product Change-over

The second step in this stage consisted in collecting and analyzing the data and the characteristics of each change and the production line. In total, 37 changes were observed, divided into 5 different types of setups according to their nature and named setups type 1, 2.1, 2.2, 2.3 and 3. These setups have different mean times depending in their complexity.

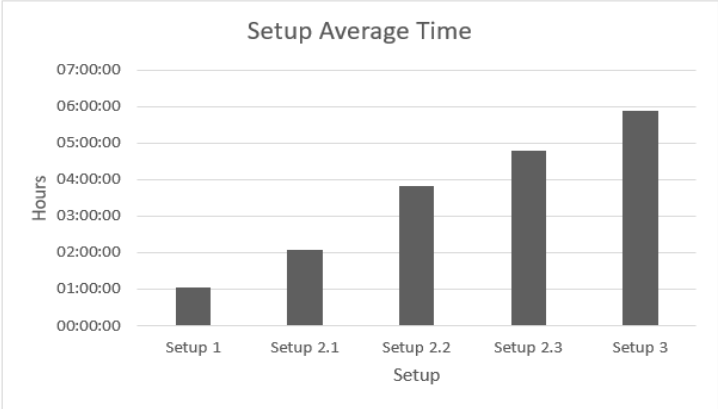


Figure 4 - Average Time of the different setups

This packaging line has 2 machines, the blister line to pack the pills into blisters and the cartoner to pack the blisters into carton boxes. Both machine have the same setup process but with different natures. In this phase, the theoretical and the real times of each activity of the process were analyzed and compared (Figure 5).

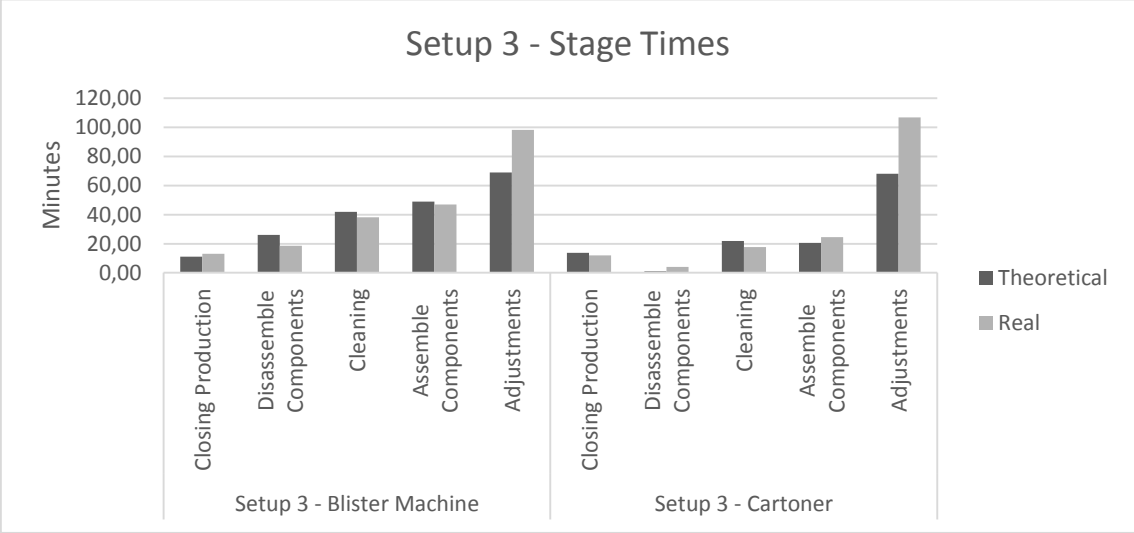


Figure 5 - Time of each stage of a setup 3

The history of the changeovers was also analyzed in the years 2016 and 2017, noting the number of changes, the dispersion of change times, the products, most frequent products, as well as the number of changes between each size of box and leaflet and number of problems during the setups (Table 2).

Table 2 - Nº of changes between leaflet dimensions during 2016 / 2017

Nº OF CHANGES BETWEEN LEAFLET DIMENSIONS (2016 & 2017)																	
	180x210	180x240	180x260	180x300	180x320	180x350	180x370	180x380	180x400	180x420	180x450	180x480	180x500	180x520	180x560	180x600	180x840
180x210	12	1	0	3	0	1	0	1	2	1	1	0	0	0	0	16	5
180x240		3	0	3	0	0	0	0	1	0	0	0	0	0	0	4	1
180x260			2	1	0	1	1	0	1	0	0	0	0	0	0	2	0
180x300				52	3	3	0	2	5	0	1	1	2	0	0	28	4
180x320					45	0	2	1	0	12	0	0	0	0	0	15	1
180x350						5	0	0	2	2	0	0	0	0	0	6	2
180x370							2	0	2	0	0	0	0	0	0	5	1
180x380								3	0	1	0	0	0	0	0	8	1
180x400									21	1	2	0	0	0	7	23	3
180x420										36	1	1	0	1	0	16	1
180x450											12	0	0	1	0	31	1
180x480												0	2	0	0	5	3
180x500													2	0	0	7	1
180x520														25	0	14	0
180x560															5	9	4
180x600																265	16
180x840																	4

4.2 Interpret and Plan

With the first point of the cycle completed, an elaborate interpretation of causes and an improvement plan was defined. The first step in this stage was the analysis of the main causes using a Lean technique called cause-effect diagram explored by Ishikawa (Figure 6).

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

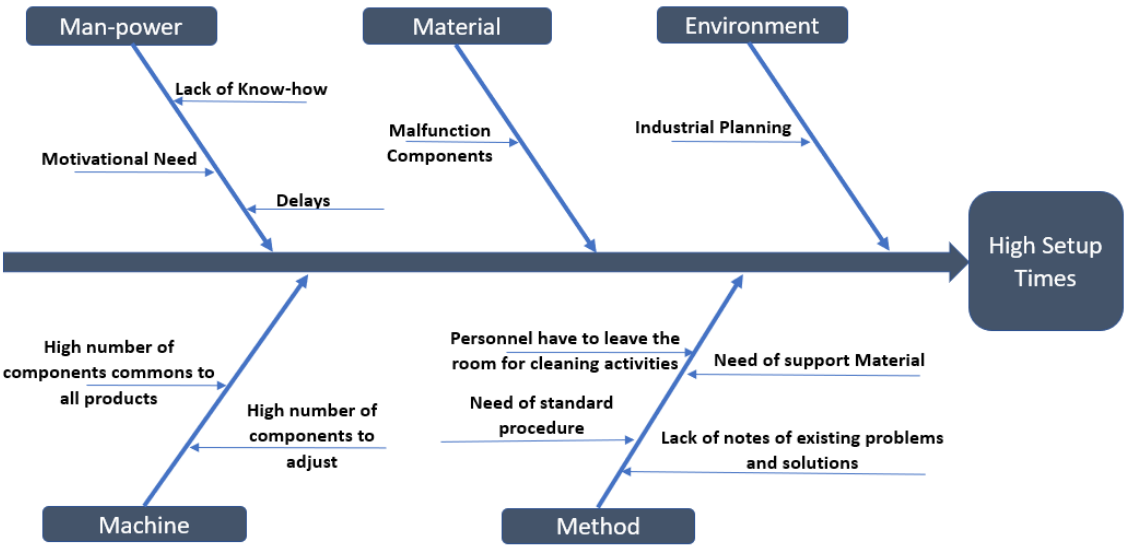


Figure 6 - Ishikawa Diagram of the causes of High Setup Times

With the identification of the main problems, the difficulties were interpreted and by the application of Lean methodologies, 6 solutions were defined:

- Application of 5's methodology: Work Standardization - Create a standard work procedure for the setup activities including a new element. The purpose of this application is to create a standard procedure of the best practice. This new practice relies on executing activities in parallel with the help of a third element.
- Application of SMED methodology: Acquisition of a redundant set of components - The acquisition of redundant set of some components externalize the activity of their cleaning.
- Standardization of the dimensions of leaflets and carton boxes - Reducing the number of different dimensions, without creating any kind of constraint to the costumer, reduces the number of adjustments in the setup. By choosing the dimensions that create the lowest number of stoppages during the production, productivity will increase.
- Use of Total Productive Maintenance Morale methods: Creation of Troubleshooting and tutorials - train the personnel to perform all the functions needed in a setup including adjustments or small maintenance activities.
- Poka-yoke application: Rearrange support material – creation of mechanism in the support material to help the transportation of machine components.
- Application of Error Proofing methodology: Creation of checklists for the preparation of machine components - Create check lists to don't miss any important part creating a set of components needed in the line.

In the table 3 it is presented, according to the McIntosh's SMED approach, the solutions proposed and what difficulty it aims to reduce. The "Creation of Troubleshooting and Tutorials" and the "Standardization of the dimensions of leaflets and carton boxes" do not aim specifically to reduce the time consumed in adjustments but by reducing the effort in performing adjustments activities and by reducing the variety of the dimensions it is expected to short the "Adjustments" activities.

Table 3 - Improvements presented to each difficulty

	Organization-led Improvement	Design-led Improvement
On-line Activity	• Acquisition of a redundant set of components	
Adjustments	• Creation of Troubleshooting and tutorials	• Standardization of the dimensions of leaflets and carton boxes
Variety	• Standard work procedure for the setup activities	• Standardization of the dimensions of leaflets and carton boxes
Effort	• Creation of Troubleshooting and tutorials • Creation of checklists for the machine components preparation	• Rearrange support material

4.3 Create

4.3.1 Standard work procedure for the setup activities

This new practice relies on the execution of the cleaning activities in parallel and in the cooperation between the personnel to perform the activities in a quicker way. The Figure 7 represents a setup before the new approach where each staff cleans their own machine and then they clean the components.

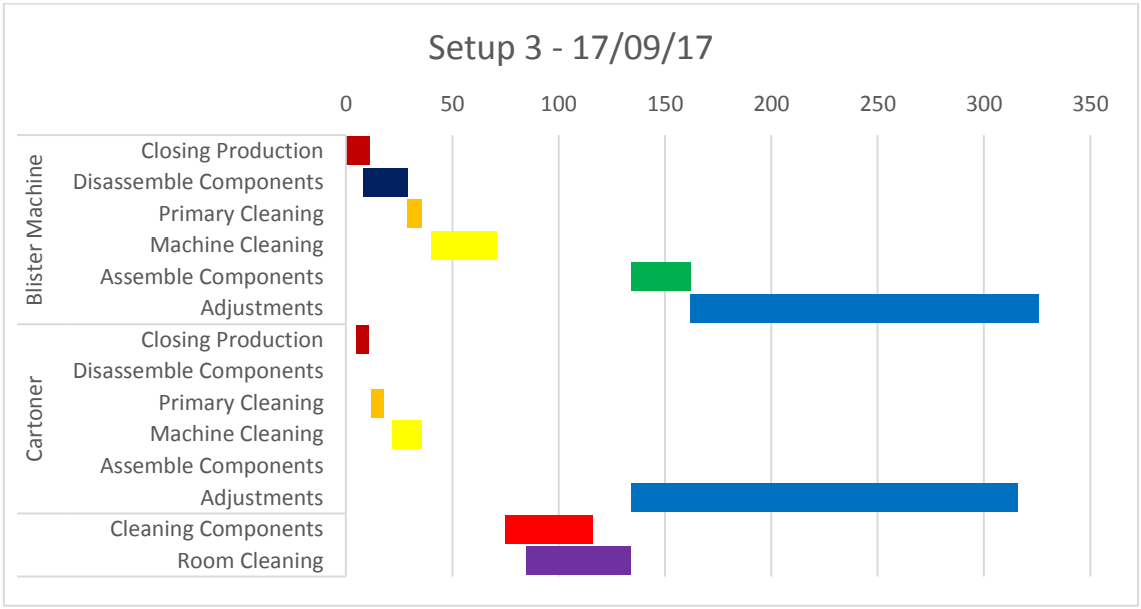


Figure 7 - Gantt Chart of a setup before the procedure standardization

In Figure 8, a setup is presented to observe the new procedure where the cleaning of the machine, the room and the components are performed in parallel, and the personnel execute the cleaning of the machine together to reduce the effort. The difference between these two setups is 54 minutes.

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

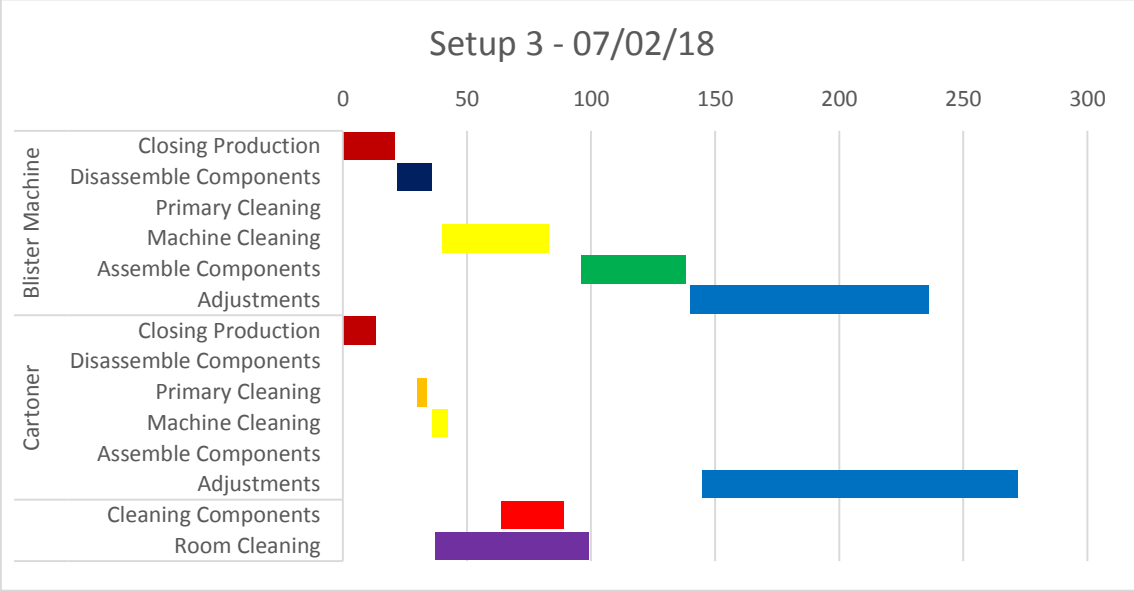


Figure 8 - Gantt Chart of a setup with the procedure standardization

4.3.2 Acquisition of a redundant set of components

The acquisition of a redundant set of components brings an investment of 9922,17€, reducing between 27 to 68 minutes in setups 2.2, 2.3 and 3. This reduction may represent a decrease of 130 hours and 56 minutes of setup time within 12 months. The return of the investment is forecasted to be within 8 months.

4.3.3 Standardization of the dimensions of leaflets and carton boxes

This standardization aims to reduce the number of adjustments required in a setup (Figure 9). During the time studied, this solution would reduce 78 adjustments, wich decreases the setup time in 13 hours and 58 minutes to an average of 10,75 minutes of leaflet adjustment. Regarding that the dimensions chosen are the ones that create the least number of stoppages, this solution also creates an increase of productivity.

Nº OF CHANGES BETWEEN LEAFLET DIMENSIONS (2016 & 2017)

	180x210	180x240	180x260	180x300	180x320	180x350	180x370	180x380	180x400	180x420	180x450	180x480	180x500	180x520	180x560	180x600	180x840
180x210	12	1	0	3	0	1	0	1	2	1	1	0	0	0	0	16	5
180x240		3	0	3	0	0	0	0	1	0	0	0	0	0	0	4	1
180x260			2	1	0	1	1	0	1	0	0	0	0	0	0	2	0
180x300				52	3	3	0	2	5	0	1	1	2	0	0	28	4
180x320					45	0	2	1	0	12	0	0	0	0	0	15	1
180x350						5	0	0	2	2	0	0	0	0	0	6	2
180x370							2	0	2	0	0	0	0	0	0	5	1
180x380								3	0	1	0	0	0	0	0	8	1
180x400									21	1	2	0	0	0	7	23	3
180x420										36	1	1	0	1	0	16	1
180x450											12	0	0	1	0	31	1
180x480												0	2	0	0	5	3
180x500													2	0	0	7	1
180x520														25	0	14	0
180x560															5	9	4
180x600																265	16
180x840																	4

Nº OF CHANGES

	180x240	180x320	180x420	180x600	180x840
180x240	16	6	6	21	6
180x320		103	28	49	5
180x420			75	70	8
180x600				378	25
180x840					4

Figure 9 - Nº of changes between leaflet dimensions with the new approach

4.3.4 Creation of Troubleshooting and tutorials

This implementation has the purpose of fighting one of the problems identified during the observation of the setups, the lack of know-how performing adjustments or some simple maintenance task every time it is needed. Training all personnel with the

best practices will reduce the effort executing the setup activities' time. It will also eliminate the dependency on the technicians reducing the waits for maintenance. In the figure is a graph about the hours spent on delays in waiting for maintenance during 2017. In figure 10 it is possible to notice the difference between the "Production Line E" where all the staff has knowledge about simple maintenance tasks and the remaining production lines where there is completely different reality regarding this theme.

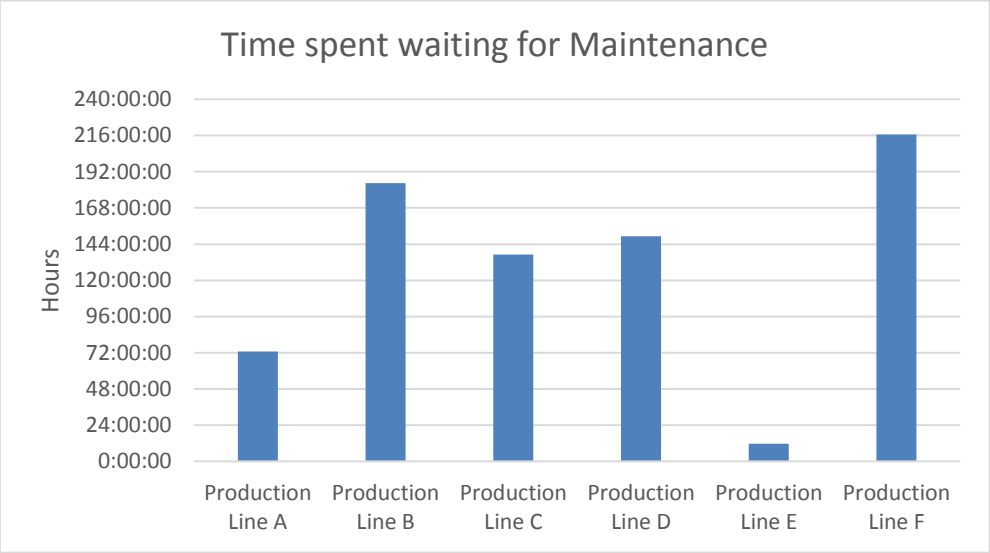


Figure 10 - Time spent on waiting for maintenance during 2017

4.3.5 Rearrange support material

This application of poke-yoke consists in create a side support on the trolleys to carry long components (Figure 11). This solution has the purpose of reducing the effort preparing the production components and to prevent the objects from falling and getting damaged.

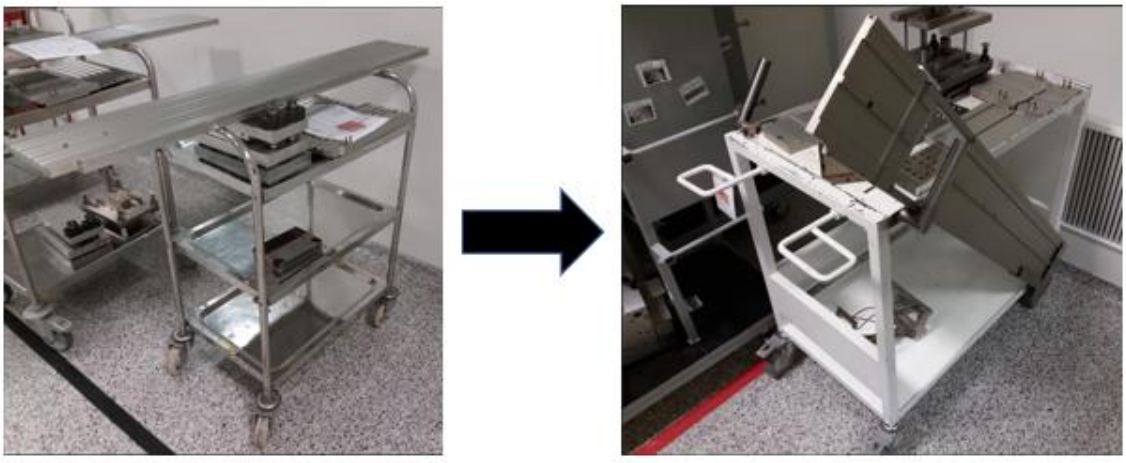


Figure 11 - Side Support on the trolleys

4.3.6 Application of Error Proofing methodology

By creating check-lists for the components needed in the productions it is possible to prevent one of the problems observed during the setups wich is to gather components missing in the preparation. This check list has the purpose of preventing errors during an external task that may create trouble in the performance of the internal activities.

4.4 Verify

Even though are presented six solutions, only the ones who did not required financial investments were applied. This stage only contemplates the implemented solutions which are:

- Standard work procedure for the setup activities

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

- Creation of Troubleshooting and tutorials
- Creation of checklists for the machine components preparation

By creating a sequence of operations, it was possible to standardize the operational method among all operators facilitating the execution of their tasks. This implementation of the operative mode allowed an average time reduction of setups type 3 and 2.3 by 60 minutes.
The standardization of processes decreased the dispersion of the values indicating the real time of the setup times (Figure 12). The training of the operators in this operative model also provided a reduction in the number of waits in 24.5%: from 4,41 occurrences per month to 3,33 occurrences per month.

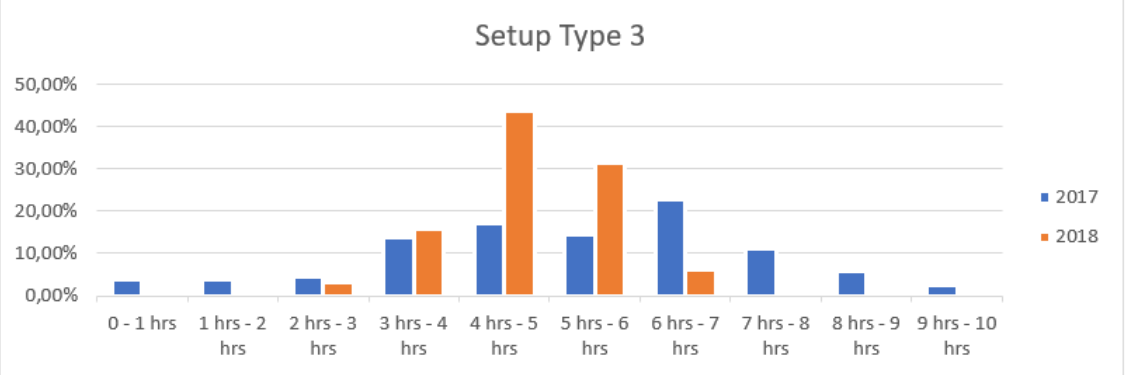


Figure 12 - Dispersion of the setup times in 2017 and 2018

This study provides an example of the application of different Lean tools in an organization which, without financial investment allowed the reduction of changeover time by 20%. This reduction occurred in the space of 3 months. If keeping these data constant, in the period of 12 months. it will reduce 266 hours and 15 minutes; the potential of reduction corresponds to a financial gain of approximately 33603€.
The behavior of the availability of this production is shown in Figure 3 where can be observed a slight increase.

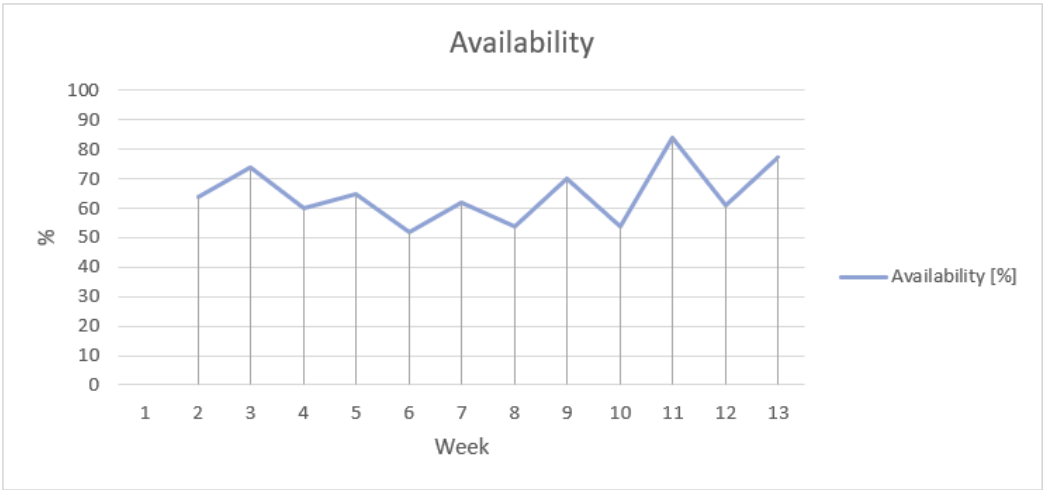


Figure 13 - Availability of the production line during the implementation

CONCLUSIONS

In this study, lean methodologies were applied in a packaging line of the pharmaceutical sector with the purpose of reducing setup times. The application of this study was conducted according to a proposed four-stage cycle. This cycle indicates the lean methodologies for each step demonstrating the efficiently application of Lean tools in the reduction of setup times.
This study resulted in a presentation of 6 proposed solutions. Only the solutions which did not require any investment were applied. Without investments in new equipment or in personnel, within 3 months, this application brought a decrease of the standard deviation and a reduction of 60 minutes of the mean setup time 2.3 and 3. This reduction can be translated in a

decrease of 266 hours and 15 minutes in 12 months, which corresponds to an annually financial gain of 33603€. The application of the remaining solution proposals foresees a reduction, within a period of 12 months, between 136 and 163 hours corresponding to a financial gain between 17164,56€ and 20573,23€ for an investment around 36492€.
The availability of the packaging line suffered a growth with the application of this analysis, which is the main object of the study. It will permit the pharmaceutical plant to manage a higher volume and greater variety of products, and it will reduce the batch size.
This paper has the purpose of providing a methodology that applies a selection of lean tools to reduce the setup time and increment the availability in a field where the production process is strictly influenced by norms like GMP, and in a market that has a great need of flexibility.
The time reduction of the changeover activities may support a company growth and ensure a measurable and long-term competitive advantage.

REFERENCES

Al-Aomar, R. (2011). Applying 5S lean technology: An infrastructure for continuous process improvement. *Academy of Science, Engineering and Technology*.

Bevilacqua, M., Ciarapica, F. E., Mazzuto, G., & Paciarotti, C. (2013). The impact of RFID technology in hospital drug management: An economic and qualitative assessment. *International Journal of RF Technologies: Research and Applications*, *4*.

Boztinaztepe, B., & Canan, F. (2008). Lean Tools for Reducing Production Time and Satisfying Employees - A Case Study.

Culley, S., Owen, G., Mileham, A., & McIntosh, R. (2001). *Improving Changeover Performance*. Butterworth-Heinemann.

Dillon, A. P., & Shingo, S. (1985). A Revolution in Manufacturing: The SMED System.

Ferradás, P. G., & Salonitis, K. (2013). Improving changeover time: A tailored SMED approach for welding cells. *Procedia CIRP*, *7*.

Gwiazda, A. (2006). Quality tools in a process of technical project management. *Journal of Achievements in Materials and Manufactuting Engineering*.

Jeffrey K. Liker. (2006). *The Toyota way: 14 management principles from the world's greatest manufacturer*. *Action Learning: Research and Practice*.

Karam, A.-A., Liviu, M., Cristina, V., & Radu, H. (2018). The contribution of lean manufacturing tools to changeover time decrease in the pharmaceutical industry. A SMED project. *Procedia Manufacturing*.

Kumar, B. S., & Abuthakeer, S. S. (2013). Implementation of Lean Tools and Techniques in an Automotive Industry. *Journal of Applied Sciences*.

Kumar, J., Soni, V. K., & Agnihotri, G. (2014). Impact of TPM implementation on Indian manufacturing industry. *International Journal of Productivity and Performance Management*.

McIntosh, R. I., Culley, S. J., Mileham, A. R., & Owen, G. W. (2000). A critical evaluation of Shingo's 'SMED' (Single Minute Exchange of Die) methodology. *International Journal of Production Research*.

Nagyova, A., Palko, M., & Pacaiova, H. (2015). Analysis and Identification of Nonconforming Products By 5W2H. *9th International Quality Conference*.

Pinto, J. P. (2014). *Pensamento Lean - A filosofia das organizações vencedoras* (6th ed.).

Rauch, E., Damian, A., Holzner, P., & Matt, D. T. (2016). Lean Hospitality-Application of Lean Management Methods in the Hotel Sector. *Procedia CIRP*.

Schmitt, B. (2017). Top 10 Automakers For June 2017: Volkswagen Reigns And Notion Of "Peak Car" Takes A Hit. Retrieved from https://www.forbes.com/sites/bertelschmitt/2017/08/16/top-10-automakers-july-2017-no-peak-car/#28d921c915e5

Seth, D., & Gupta, V. (2005). Application of value stream mapping for lean operations and cycle time reduction: An Indian case study. *Production Planning and Control*.

Sohani, Y. D. N. (2011). Single Minute Exchange of Dies: Literature Review.

Sullivan, W. G., McDonald, T. N., & Van Aken, E. M. (2002). Equipment replacement decisions and lean manufacturing. *Robotics and Computer-Integrated Manufacturing*.

Tommelein, I. D., & Ballard, G. (1999). *Proceedings IGLC. 7th Conference of the International Group for Lean Construction*.

Van Goubergen, D., & Van Landeghem, H. (2002). Rules for integrating fast changeover capabilities into new equipment design. *Robotics and Computer-Integrated Manufacturing*.

Curado, A. G. (2019).
Application of lean methodologies in the reduction of setup times in the pharmaceutical industry. *Millenium*, 2(8), 37-52.
DOI: https://doi.org/10.29352/mill0208.04.00220

Wallace, S. W., & Choi, T. M. (2011). Editorial: Challenges in apparel production planning and control. *Production Planning and Control*.

Williamson, R. (2015). A Tribute to the Father of Total Productive Maintenance.

Womack, J. P. (1990). *The Machine That Changed the World*.

EDUCAÇÃO E DESENVOLVIMENTO SOCIAL

EDUCATION AND SOCIAL DEVELOPMENT

EDUCACIÓN Y DESAROLLO SOCIAL

millenium

EDUCAÇÃO E DESENVOLVIMENTO SOCIAL
EDUCATION AND SOCIAL DEVELOPMENT
EDUCACIÓN Y DESAROLLO SOCIAL

millenium 55

Millenium, 2(8), 55-66.

en

A DINÂMICA DA CRIAÇÃO DO CONHECIMENTO NUMA REDE INTRA-ORGANIZACIONAL

DYNAMICS OF KNOWLEDGE CREATION IN INTRA-ORGANIZATIONAL NETWORKS

LA DINÁMICA DE LA CREACIÓN DEL CONOCIMIENTO EN UNA RED INTRAORGANIZACIONAL

Clotilde Passos[1]

[1] *Universidade Católica Portuguesa, Departamento de Economia, Gestão e Ciências Sociais, Viseu, Portugal*

Clotilde Passos - clotilde.passos@gmail.com

Corresponding Author
Clotilde Passos
Universidade Católica Portuguesa
Campus Viseu
Estrada da Circunvalação
3504-505 Viseu
clotilde.passos@gmail.com

RECEIVED: 16th May, 2018
ACCEPTED: 01th October, 2018

Passos, C. (2019). Dynamics of knowledge creation in intra-organizational networks.
Millenium, 2(8), 55-66.
DOI: https://doi.org/10.29352/mill0208.05.00192

RESUMO

Introdução: O conhecimento tem vindo a evidenciar uma crescente importância na economia, como fator de diferenciação e recurso gerador de competitividade, sendo considerado um grande pilar de desenvolvimento das sociedades actuais. As redes, dentro e entre organizações, como estratégia potenciadora para a partilha do conhecimento, podem ser uma das vias mais importantes que uma organização pode adotar.
Objetivos: Avaliar como ocorre a dinâmica da criação de conhecimento numa rede intra-organizacional.
Métodos: O estudo desenvolve-se segundo uma tipologia descritiva e exploratória e utiliza a metodologia de estudo de caso desenvolvido numa rede de serviços de óptica em Portugal.
Resultados: Os resultados apresentam evidências que o contexto de uma rede intra-organizacional pode proporcionar um ambiente de aprendizagem coletivo, representado sobretudo pela interação que ocorre entre os colaboradores das unidades e por meio de vários "espaços de interação" e criação de conhecimento.
Conclusões: As potencialidades atribuídas ao conhecimento e às redes na promoção de melhores práticas organizacionais justificam o interesse da análise e o contributo do estudo para o meio empresarial.

Palavras-chaves: Criação de conhecimento, redes intra-organizacionais, gestão do conhecimento

ABSTRACT

Introduction: Knowledge has shown growing importance in economy, as a differentiation factor and a competitiveness enhancer, being considered as an important pillar of modern society development. Networks within and between organizations, as a strategy to potentiate knowledge sharing might be considered one of the most important approaches an organization may adopt.
Objectives: To evaluate how the dynamics of knowledge creation occur in an intra-organizational network.
Methods: This study is developed according to a descriptive and exploratory typology and uses the methodology of a case study developed over an optical services network in Portugal.
Results: The results present evidence that the context of an intra-organizational network may favor a collective learning environment, mainly represented by the interaction that occurs between the collaborators of the various units and with the help of several "interaction" and knowledge creation spaces.
Conclusions: The potential attributed to knowledge and networks in promoting the best organizational practices justify the interest of this analysis and the contribution of this study to the business environment.

Keywords: Knowledge Creation; intra-organizational networks; Knowledge management.

RESUMEN

Introducción: El conocimiento ha venido a evidenciar una creciente importancia en la economía, como factor de diferenciación y recurso generador de competitividad, siendo considerado un gran pilar de desarrollo de las sociedades actuales. Las redes, dentro y entre organizaciones, como estrategia potenciadora para el compartir el conocimiento, pueden ser una de las vías más importantes que una organización puede adoptar.
Objetivos: Evaluar cómo ocurre la dinámica de la creación de conocimiento en una red intraorganizacional.
Métodos: El estudio se desarrolla un tipo descriptivo y exploratorio y utiliza la metodología de estudio de caso desarrollado en una empresa de servicios de redes ópticas en Portugal.
Resultados: Los resultados presentan evidencias que el contexto de una red intraorganizacional puede proporcionar un ambiente de aprendizaje colectivo, representado sobre todo por la interacción que ocurre entre los colaboradores de las unidades y por medio de varios "espacios de interacción" y creación de conocimiento.
Conclusiones: Las potencialidades atribuidas al conocimiento ya las redes en la promoción de mejores prácticas organizacionales justifican el interés del análisis y la contribución del estudio al medio empresarial.

Palabras Clave: Creación de conocimiento; redes intraorganizacionales; Conocimiento administrativo.

INTRODUCTION

From the current economic environment, of constant uncertainty and complexity, emerges the inevitability of restructuring the organizations in order to define actuation strategies, regarding resource sharing, in a perspective of inter and intra-

organizational cooperation, with the goal of creating synergies, competitive advantage and sustainability. Drucker (2003) foresaw that the "next society will be a knowledge based society", in that sense, knowledge is a key resource for competitiveness and for the individual and organizational capacity to create, process and transform information and knowledge. The network paradigm has been extended to several disciplines (Vidal, 2017).

Some authors, like Grant (2006), Nonaka, Toyama and Konno (2002) and Sundaresan and Zhang (2017) consider that the ability to create and utilize knowledge is an important source of sustainability and competitive advantage to the company. On this context, inter and intra-organizational networks may be seen as a mechanism which allows organizations to reach their goals. Uzzi and Spiro, (2005); and Ahuja et al., (2016), affirm that networks help spreading the rules, knowledge and other resources. On this line of thought, Tatarynowicz et al., (2016) affirm that networks can become an adequate strategy, since they can improve the competitiveness of organizations, shape behaviors and results and allow organizations obtain competitive advantages.

Even with acknowledged collective capacity and efficiency through networking, however, this phenomenon has been scantly studied (Perrow, 1992) and existing literature about the theme is still scarce.

Nonaka et al., (2002) emphasize that the conditions favorable to the creation of knowledge in an organization involve the method of knowledge conversion (Socialization – Externalization – Combining – Internalization), but for it to actually occur it is fundamental the emersion of a "*ba*", Japanese concept meaning a physical, virtual or mental space within which knowledge is created, shared, and utilized.

Considering these facts, this study´s goal is to evaluate and to understand how the knowledge creation dynamics occur in an intra-organizational network in the optics sector, in Portugal.

Therefore, the study is structured into three parts; after the introduction a theoretical reflection about the studied concepts is made, methodology is resumed, results are presented followed by the discussion, conclusions, contribution, limitations and suggestions for future research.

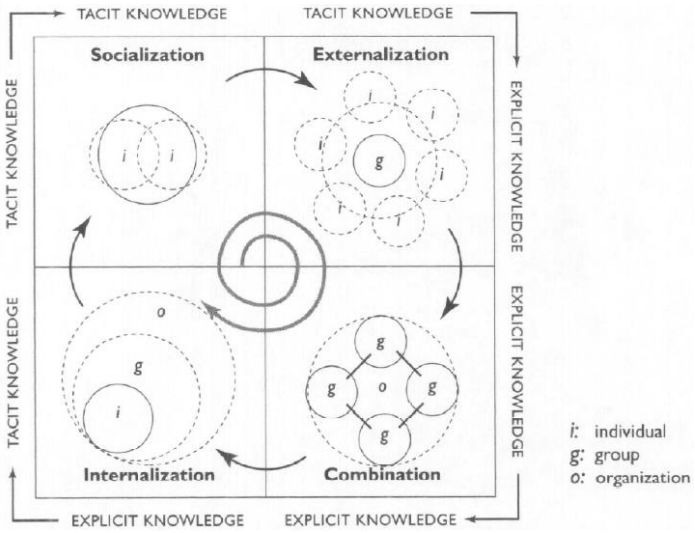
1. THEORETICAL FRAMEWORK

1.1 Knowledge creation in organizations

Nonaka and Takeuchi (1997) support the thesis that knowledge is a high valued asset for the organization and has the following dimensions: it is tacit, because it is strongly related to action, procedures, routines, ideas, values and emotions; it is dynamic, because it is created within social interactions between individuals, groups and organizations; it is humanist, for it is essentially related to human action. According to these authors, knowledge creation assumes an individual learning process resulting from reflection, creativity and questioning, therefore of a constructivist nature. The constructivist theory regards learning and knowledge creation as an active process, where the subject, interacting with others, creates new knowledge. According to Nonaka and Nishiguchi (2001) organizational learning occurs through shared perceptions, knowledge, and mental models, it is built upon past knowledge and experience, that is to say, upon memory and the organizational memory depends on institutional mechanisms such as policies, strategies and explicit models, used in order to retain knowledge.

Individual learning, by itself, does not assure organizational learning, but, without it, however, organizational knowledge does not happen (Nonaka and Takeuchi, 1995). Organizational learning occurs at three levels – individual, group and organizational – which makes knowledge to flow from individual to the group, and then to the organization (Nonaka, 2001). Nonaka and Takeuchi (1995) named "knowledge conversion" the process by which organizations create knowledge, based on the critical assumption that human knowledge is created and expanded through social interaction between tacit and explicit knowledge. This process develops itself in four knowledge conversion modes as presented in Picture 1.

Passos, C. (2019). Dynamics of knowledge creation in intra-organizational networks.
Millenium, 2(8), 55-66.
DOI: https://doi.org/10.29352/mill0208.05.00192



Picture 1 – SECI Model of knowledge forms transformation/conversion
Source: Nonaka and Konno (1998, p.43)

We understand tacit knowledge as personal knowledge, meaning it is difficult to formalize and transmit to others (Nonaka and Takeuchi, 1995; Li and Gao, 2003). Explicit knowledge is the one which is produced from collected data and storage information (Nonaka and Konno, 2001).

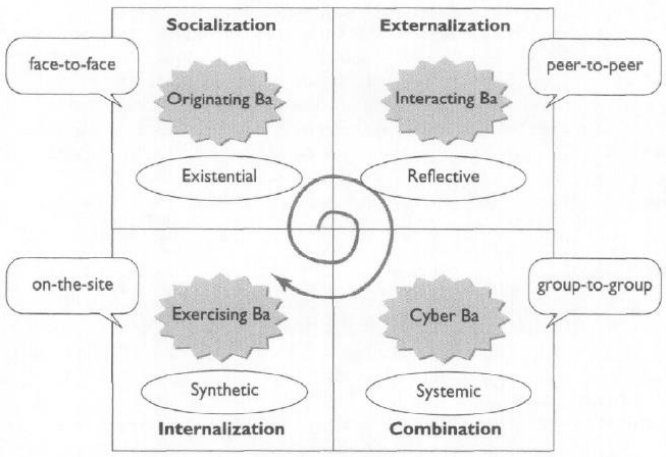
The socializing field occurs from the experience and from shared mental models, on which face-to-face communication creates shared knowledge (Nonaka and Takeuchi, 1997). In the externalization field, conceptual knowledge is created, expressed in metaphors, analogies, concepts, hypothesis or models Nonaka and Konno (2001). The conversion of explicit knowledge into a more complex set of explicit knowledge is called combination. The keys for that process are knowledge communication, diffusion e systematization (Nonaka and Konno, 2001).

Combination occurs in three phases: First is the gathering and integration of knowledge; second is dissemination; and third, editing e processing knowledge (Nonaka and Takeuchi, 1997; Nonaka and Konno, 2001). Internalization process is no more than learning by doing in organizational apprenticeship, with no empirical testing (Gourlay, 2006). Interaction in order to create knowledge starts in a field which facilitates sharing between individuals, generating dialogs and collective reflections, to create new knowledge which will become crystallized through products, management processes or systems (Nonaka et al., 2002) as shown in Picture 2.

Picture 2 – Types of interaction spaces
Source: Adapted from Nonaka et al., (2002)

Knowledge cannot be created in a vacuum, it needs a place, an appropriate and specific context in terms of time, space and relationship between individuals with the necessity of a dynamic interaction, which allows to create, utilize, share and spread knowledge (Nonaka et al., 2002; Kostiainen, 2002).

Nonaka and Nishiguchi (2001) name the aforementioned context as "*ba"*, which serves as the knowledge creation platform. The *ba* concept aims to unify the physical space, within which knowledge is created, shared and used. In this study the expression "interaction space" is used to name the concept of *ba*, which represents all the space which promotes interaction between individuals in a knowledge creation dynamic.
Nonaka et al., (2002) present four groups of "interaction spaces". The first one is knowledge socialization spaces (*originating ba*), on which tacit knowledge, shared through common experiences, is responsible for the emergence of knowledge assets as skills, *know-how*, confidence. Then comes knowledge externalization spaces (*dialoguing ba*), where tacit knowledge is articulated in the form of symbols, images and language, promotes the creation of knowledge assets like *design* and product concept (Nonaka, and Toyama, 2003). The third group refers to specific knowledge systematization spaces (*systematizing ba*), which are responsible by the emergence of knowledge assets like *database*, documents, specifications, manuals, patents and licenses (Nonaka, 2005). At last, knowledge applied to activities, actions and practices or knowledge internalization spaces (*exercising ba*) is responsible for creating knowledge assets like *know-how*, organizational routines and new behavior patterns (Nonaka, 2005) as depicted on picture 3.



Picture 3 – The four characteristics of "*ba"*
Source: Nonaka, Toyama and Konno (2002)

These "interaction spaces" allow knowledge to be socialized through means of face-to-face interaction where individuals share knowledge, feelings, emotions, experiences and mental models.
Provan et al., (2009) and McNamara et al., (2015) developed studies on how the organizational knowledge creation process occurs, effectively, and how the organizational structure influences on the existence of different "interaction spaces". They have reached the conclusion that knowledge works as a contingency variable, which determines the fitness of the organizational structure. Thus, it can be stated that, "interaction spaces" might emerge in work groups, informal circles, occasional meetings, virtual spaces and other moments where relationships occur in shared time and space. So, the question is how to potentiate the creation and utilization of knowledge. Some research point in the direction that the network configured enterprise might be more effective in knowledge creation than the one integrated in creation, transferring and recombining knowledge (Nonaka and Nishiguchi, 2001; Chua, 2002; Tsai, 2002; Spencer, 2003; Jacometti et al., 2015). Results point in the direction of understanding on how a network configuration might support the knowledge creation process.

1.2 Inter and intra-organizational networks
The "inter and intra-organizational networks" have been gaining ground on organizational theory (Ahuja et al., 2012). The inter-organizational relationships exists in several forms, like; *joint ventures*, licensing, *cobranding*, *franchising*, networks, associations and consortiums (Klein and Pereira, 2016).
In the social sciences study field, the term "network" designates a set of directly or indirectly interconnected people or organizations (Kim et al., 2006). In the technical-operational field, the term "network" underscores the idea of flow. In the organizational field, it implies a set of relations between companies, seen as a strategic resource for the organizations to face an uncertainty environment, characterized by competition. Actually, strategic networking within organizations are fundamental communication, interaction, share and knowledge diffusion vectors (Eiriz, 2004; Coviello, 2006).
Gulati (2007), Lefroy and Tsarenko (2013) define organizational networks as a more modern and dynamic way of structural configuration, establishing lasting agreements between two or more companies. Franco et al., (2011) defines networks as a group of organizations which involve exchanging and resource and capabilities sharing, with the goal of achieving a common

Passos, C. (2019). Dynamics of knowledge creation in intra-organizational networks.
Millenium, 2(8), 55-66.
DOI: https://doi.org/10.29352/mill0208.05.00192

strategy. Schiele et al., (2015) affirm that "the company where resources are found lacking will have to obtain them through relationships with others".
Other relevant characteristics are pointed out: gathering of several companies; operate in a specific market segment; establish horizontal and cooperative relationships between its elements; and it is structured according to basic coordination rules. Actually, networks are organizational systems capable of uniting individuals and institutions, in a democratic and participatory way, around a common goal (Franco e Barbeira, 2009).
Beyond that, inter-organizational relationships may increase the opportunities to develop new capabilities and launch new products without the need for large investments (Klein e Pereira, 2016). According to McNamara et al., (2015), they serve to define common goals and to create sustainable relationships. The real contribution of networks, to the participating organizations, is the development of specific organizational capabilities and competences, including aspects which address the improvement of processes (administrative, production, human resources, innovation, marketing and services) and to produce, process and transform information and knowledge into economic assets which grant differentiation, competitiveness and sustainability.

1.3 Creation of knowledge in intra-organizational networks

The intra-organizational network has the potential to create and develop new knowledge (Spencer, 2003; Cudney et al., 2014), to achieve that, there must be a knowledge management system which encourages that type of relation, which must be turned to information flow creation and organization within and between the various organizational levels, aiming to create, increase, develop and share knowledge within the organization. In this sense, knowledge management, might be seen as a set of processes which manages knowledge creation, dissemination and use in order to achieve the organization's goals (Lefroy and Tsarenko, 2013).
Nonaka and Takeuchi (1997) provided evidence of the creation of new knowledge from interaction between individuals, groups and organizations. According to these authors, knowledge comes at an individual level, being expanded through knowledge interaction and socialization dynamics, to an organizational and, onwards, to an inter-organizational level. Thus, knowledge is created solely by individuals; an organization, by itself, cannot create knowledge, but may provide a positive and constructive relationship space between the several elements, in such a way that allows information, knowledge, experiences and competences exchange to create a context to knowledge creation within organizations.
In order for the intra-organizational knowledge creation process to be effective, it is necessary a synergetic and stimulating environment, where emotions, experiences, feelings and mental images are shared beyond the organization borders (Spencer, 2003; Szell et al., 2010).
Tsai (2002), mentions that this environment cannot be produced by the traditional company model.
Therefore, one might say that an intra-organizational network may provide the existence of an effective interaction between people, groups and organizations, intra-organizationally expanding the knowledge created by individuals. This dynamic promotes competences complementarity through which knowledge, practices, values, processes, culture and individual differences are collectively shared with a common goal in sight (Gest et al., 2011).
The creation of knowledge in an organization involves articulated and intentional interconnection between its members, in a way that allows for sharing reasoning and ideas aiming to stimulate the cooperation and collaboration with the goal of promoting organizational performance.
Nonaka and Toyama (2003) refer to "devices" through which individual knowledge is connected and distributed, spreading in a spiral inside the organization. These devices have the function of promoting a continuous and dynamic interaction, which promotes sharing and creation of knowledge. Some studies suggest that networks are amongst the main necessary conditions to endure the pressure and to sustain enough levels of knowledge diversity, in order to prosper in current environments (Uzzi and Spiro, 2005; Franco and Mariano 2007; Gulati et al., 2012; Valente et al., 2015).
Considering this, it can be said that the network configuration might facilitate the appearance of "interaction spaces" which are favorable to the knowledge creation process. To better understand how that dynamic occurs and its potential contribution to the company, evidences from a case study in the optical business area in Portugal were sought, and presented next.

2. METHODS

This study develops through a qualitative topology, it is framed by a descriptive and exploratory line and adopts the methodology of a unique case study, concerning a network in the optical services area in Portugal. According to Yin (2001), in a case study, the goal is to expand and generalize theories (analytical generalization), in order to accomplish an holistic view of the studied phenomenon. Godoy (2006), says that the case study is evaluative.
Since the object of the study is to understand and analyze how does the knowledge creation dynamics works, within an intra-organizational network, the method used was the one proposed by Nonaka et al., (2002) (picture 2), considering as "interaction spaces" the place where the sharing and creation of knowledge occurs.

The analyzed unit in the case study was one store, which is part of an international Optics services network. The group, at a National level, is formed by several stores, 78 of which are franchised, and 77 others which are directly managed by the group (own stores). For this study, only the management and policies practices of own stores are considered. This is an horizontal network, formed by small stores, with an average of 7 workers per store. The choice of the analyzed unit was due to the fact that this group adopts an intra-organizational management network, allowing access to data collection and is adequate to the goal of this study.

Since it is impossible to do a longitudinal study, the retrospective method was adopted, revealing occurred changes and the motives that originated them. The retrospective method allows the interviewed to describe the events and changes, in their own terminology, besides allowing them to propose their own explanations to the facts. Data and information collection, according to Yin (1994), must contemplate various sources, observation, documents analysis, interviews and inquiries, contributing to create evidence chains, which connect gathered data to the purpose of the study. This work was performed through semi-structured interviews, information gathering through web site and direct observation. To conduct the interviews a guide was prepared, according to the idea defended by Lee (1999) and Hartley (1994). The analysis of the interviews was made through the content analysis, according to the set of rules guaranteeing its rigor (Miles and Huberman, 1994), which consists in the interpretation of the data from the lexical analysis, or content analysis.

The analyzed unit (store) has seven workers: The unit responsible, optometrist, salesperson, sales helper and counter employees. A pre-interview was conducted with one of the store's elements (optometrist) which allowed the readjustment of the questions, in order to evaluate and conclude the second goal of the study. Being a retrospective study, one of the inconveniences is that the terminology of the interviewed might be imprecise or inconsistent amongst them. To minimize this problem, the interviewed must encompass the "widest possible of hierarchical positions so that the information can be analyzed" (Glick, Huber et al., 1993). Thus, the empirical evidences were collected from interviews with the seven workers, with an approximate duration of 30 minutes each, during the month of May 2017, by the interviewer. The content of the interviews was noted and, afterwards, a transcript was produced. Additionally, other evidences were collected allowing to create evidence chains, which connect the collected data to the analysis variables. The operationalization of the study occurred from the systematization between conceptual elements, authors, purpose and the corresponding variables according to Table 1.

Table 1 – Operationalization of the research variables

Conceptual Elements	Authors	Variables analysis
Companies networks	Perrow, 1992; Gulati, 2007; Suire and Vicente, 2008 Franco et al., 2011; Lefroy and Tsarenko, 2013 Schiele et al., 2015; Klein and Pereira, 2016 Vidal, 2017	- geographic localization of the stores; - number of stores participating in the network; - business sector (commerce or service); - type of service or product; - coordination instruments; - formalization level of relations between stores in the network (formal *versus* informal); - hierarchy levels of the relations between networks (hierarchy *versus* cooperation); - cooperation level *versus* competition between network stores; - objectives inherent to the formation of the network.
Knowledge creation	Nonaka, 1994; Nonaka and Takeuchi, 1997; Nonaka and Nishiguchi, 2001; Chua, 2002; Nonaka *et al.*, 2002; Spencer, 2003 and Tsai, 2002 Kostiainen, 2002 Sher and Lee, 2004 Provan et al., 2007; Gest et al., 2011 Valente et al., 2015	- types and quantities of "interaction spaces" dedicated to knowledge socialization (fraternizing, touring the stores, other meetings); - types and quantities of "interaction spaces" dedicated to knowledge externalization (formal meetings, collective decision making processes, planning meetings and goals); - types and quantities of "interaction spaces" dedicated to knowledge systematizing (electronic communication, formal documents, data banks, shared management systems); - types and quantities of "interaction spaces" dedicated to knowledge internalization (new concepts and practices of services and management); - trust in information and knowledge sharing; - main knowledge assets created within the network

Source: own making

3. RESULTS

Among the several answers, one of those which got more similar responses refers that the greater gain of stores and collaborators, as a result of the implementation of the knowledge network, was information and experiences sharing. The

Passos, C. (2019). Dynamics of knowledge creation in intra-organizational networks.
Millenium, 2(8), 55-66.
DOI: https://doi.org/10.29352/mill0208.05.00192

shared information which brought more benefits to the network was that concerning services, suppliers, technologies and markets. That information was shared on a social inter-relationship which occurs on an informal basis between collaborators and the commercial director.

Several moments where information and knowledge sharing occurs were identified. According to the orientations of Nonaka et al., (2002), each one of those spaces work as a different set of situations, allowing to promote an effective "platform" to ease the process of knowledge creation between networked stores. "Interaction spaces" were identified from the collected data and are presented in Table 2.

Table 2 – Interaction spaces identified within the optics company network

Interaction spaces	Empirical evidences
National level meetings	"National level meetings occur in smaller numbers, usually to communicate changes in the group, strategies, management directives, services, in order to keep everyone integrated with the set goals."
Monthly meetings	"Every month meetings with the Commercial Director are set, in order to check the achievement of the established goals, the detected difficulties and the definition of new goals". "These meetings also serve to communicate the difficulties and problems related to relationships, tasks performed or problems with clients"
Individual meetings	"Most frequent individual meetings occur with the store responsible or commercial director". "These occur when new information or alerts are communicated. Or when a clarification is necessary". "At these meetings we also give our opinion about the service, or even about the management directives"
Fraternizing	"An interesting moment of information exchange is confraternization (lunches or dinners) at national and district level, attended by all employees and family involved in the network or specific sub segments of it."
	"Given the current national crisis, these meetings frequency have been decreasing overtime". They are always moments of a great deal of experiences and knowledge sharing"
Training / Colloquiums	"With the upcoming of new products or services" These training allows to acquire new knowledge about products or the market, but we also meet new people, colleagues, suppliers and even costumers" "They are always interesting experiences"
Courses and speeches	"Training is mandatory for all store workers" Whenever new products arrive or new service methodologies are implemented, training is provided, given by the group or even by suppliers"
Electronic space	"Throughout the years professional and even friendship ties are created. Informal talks occur through email. The necessity of clarifying about any situation or even asking for information is, usually, made by phone or fax. Information exchange also occur, simply motivated by curiosity or the novelty of the matter. We talk a lot by phone, email and fax".

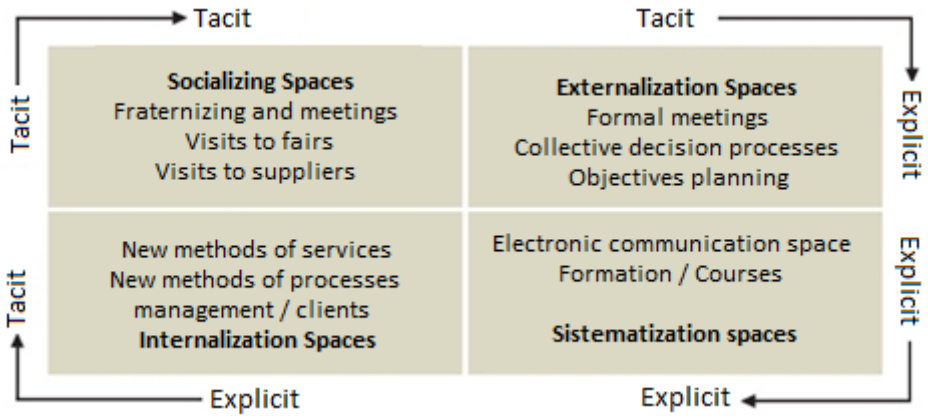
Source: own elaboration

As such, evidences were found that several physical and virtual spaces do exist, easing the knowledge creation process in the studied network. One of these spaces, usually used for communication of tacit knowledge, consists on the visits that the commercial director pays to the stores belonging to the network. Through these visits, improvement possibilities are identified in services delivery, going around technological problems and improving the relationship to clients on a continuous improvement perspective.

This dynamic leads to substantial gains to the store and to the group. Another observed fact is that, with the evolution of the network, the social relations become stronger, as the trust and cooperation levels became more intense.

Meetings with the commercial director occur at least once a month and constitute a relevant knowledge sharing space in the network. It works as a formal forum for the collective decision making process and group and individual goals planning. Goals are established according to a debate, and an individual and group reflection process, in order to produce satisfactory and feasible goals, according to market conditions. At the meetings, after concluding the planned works, a space for sharing experiences is created, favoring an atmosphere of trust and well-being between the director and collaborators. According to these, sometimes "they learn more in those informal talks than in an entire month's work". They find solutions to problems and even to perform their tasks better. This fact corroborates the affirmation from Tsai (2002), who believes that "informal lateral relations, in social interaction form, have a significant effect over shared knowledge". The store, which is the case study, is located at a mall. This context and environment also promotes and facilitates intense social relations, often allowing friendship

and business to happen simultaneously. As an example, fraternizing lunches or dinners occur between collaborators, between collaborators and customers, between collaborators and suppliers and even with elements from other stores.
Fraternizing at regional or national level, like dinners, meetings and others are important moments to solidify trust relationships and, beyond that, enable informal talks about opportunities, challenges and the future of the group. We can see that trust is fundamental to existing cooperation between collaborators and the leadership. This is established, primarily, by informal ways, face to face. Events like visiting products exhibits at fairs allows collaborators to have the opportunity to meet other colleagues, suppliers and customers, share experiences and also reflexing on the tendencies and challenges of the business sector.
The utilization of several electronic resources in the knowledge creation dynamics of the network was observed, such as the examples of email, telephone and fax. These methods are used more frequently and, on the other hand, the internet is less used. This evidence represents a point that must be strengthened in the studied network, in its knowledge creation dynamics, as the following analysis will show. On picture 4 it is proposed a classification of the diverse "interaction spaces" identified in the knowledge creation dynamics of the studied store.



Picture 4 – "Interaction spaces" in the studied optics network
Source: Adapted from Nonaka et al., (2002)

Analyzing pictures 4, the knowledge **socialization spaces** occur in the several visits to fairs, when collaborators directly observe not only the solutions, but also the new and better adopted practices. Besides that, those visits allow fraternizing and other informal encounters, sharing knowledge, emotions and feelings through informal interaction. Knowledge **externalization spaces** occur in formal meetings, objective planning meetings and collective decision making processes. These meetings allow the collaborators, through dialogue and collective reflection, to share ideas and experiences (tacit knowledge), converting them into common concepts, in the form of models, hypothesis and scenarios (explicit knowledge).
Knowledge **systematizing spaces** occur at training and courses, as well as in using the technologies that ease that process. However, the knowledge systematizing resources are defective, mainly on the lack of usage of IT systems by most of the collaborators.
Knowledge **internalization spaces**, which sustain the last phase of the knowledge conversion process, in which the knowledge is internalized and applied in terms of new organizational practices, has given good results, both for the store and for the group. By evaluating different "interaction spaces" of the studied store, a strong context of trust and interaction between the collaborators and leadership (commercial director) is generally observed. That interaction, which occurs mainly informally and face to face, offers a valuable basis for knowledge creation. According to Nonaka e Nishiguchi (2001), the biggest part, if not the whole of the knowledge, is created through an interactive process of experimenting and dialogue involving several individuals. In Soo, Devinney e Midgley's (2002) perspective, to many organizations, the informal communication channels have been a rich source of knowledge, which cannot be found in databases or organization's manuals. The importance of informal interaction is a crucial element for the creation of knowledge, mostly when knowledge is systemic, complex and tacit (Bhagat et al., 2002).

CONCLUSIONS

Before what was exposed one might say that this study is a theoretical rehearse for the formation and understanding of the knowledge creation phenomenon in intra-organizational networks, according to a systemic approach, arising important implications to the present and future managers and proprietors of companies which are inserted into a network.
One might conclude that the effective knowledge creation process, represented by the diverse "interaction spaces", allows the development of essential knowledge assets to the creation of value and competitive differential to the store and to the group.

Passos, C. (2019). Dynamics of knowledge creation in intra-organizational networks.
Millenium, 2(8), 55-66.
DOI: https://doi.org/10.29352/mill0208.05.00192

According to this research results, the fact that the group is working as a network allows new concepts and know-how, a better understanding of the network functioning scenario, knowledge about suppliers and representatives, knowledge about new processes, new technologies and products. One might say that the intangible assets, resulting from learning, are already contributing to the improvement of service delivery processes.

The results allowed us to observe that the social interaction acquired by the network configuration had a positive influence in the store and group's knowledge creation dynamics.

In the network context, several "interaction spaces" emerged where the created knowledge would hardly be attained in isolation.

The existence of formal and informal situations so that the collaborators might share expertise, experience, emotions and know-how, through face to face communication, promoted, amongst themselves at the store and in the group, an intense tacit knowledge sharing environment, and an essential resource to the sustainability, as already mentioned in the literature.

Teece et al., (1994) stress that the learning process is an intrinsically social and collective phenomenon. The conclusions relative to the network effect in promoting and amplifying knowledge creation reinforce some evidences pointed out in the literature.

To be noted is that theorizations outlined in this study, although limited by the used methodology, are intended to contribute to a more widening approach to intra-organizational knowledge creation, going from an endogenous process in the store to an exogenous process, inter-organizational. The study does not refrain from supplying evidences and ideas to the professionals to implement strategies in order to promote the success of their networks. These considerations, although not having a definitive character, stimulate the study about the theme on other methodologies views and typologies, establishing the basis to future researches, in order to deepen the theme and fill the theoretical gaps. Thus, to better understand the phenomenon mixed, qualitative and quantitative, studies are suggested in order to further validate on theorizations and allow for generalization of the same. As Nonaka e Takeuchi, (1997) affirm, "creating new knowledge means to recreate the company and all within in a continuous process of personal and organizational self-renewal".

REFERENCES

Ahuja, G., Soda, G. and Zaheer, A. (2012) ''The genesis and dynamics of organizational networks.'' Organization Science, 23: 434–448.

Bhagat, R. Kedia, B., Harveston, P. and Triandis, H. (2002) Cultural variations in the cross-border transfer of organizational knowledge: an integrative framework. *Academy of Management Review*, v. 27, n. 2, p. 204-21.

Boisot, M., and B. McKelvey (2010) ''Integrating modernist and postmodernist perspectives on organizations: A complexity science bridge.'' Academy of Management Review, 35: 415–433.

Chua, A. (2002) The influence of social interaction on knowledge creation. *Journal of Intellectual Capital*, v. 3, n. 4, p. 375-92.

Coviello, N. (2006) The network dynamics of international new ventures. *Journal of international Business Studies, 37*, 713-731.

Cudney, E. A., Corns, S. M., & Long, S. K. (2014). Improving knowledge sharing in healthcare through social network analysis, *International Journal of Collaborative Enterprise*, 4(1/2),17-33.

Drucker, P. F. (2003) A Administração na próxima sociedade. 3ª. Edição. São Paulo

Eiriz, V. (2004) Dinâmica de relacionamento entre redes inter - organizacionais. *Inovação Organizacional*, *2,* 121–153.

Franco, M., Mainardes, E., Martins, O. (2011) A review of interorganizational networks: Evidence from studiespublished in 2005-2008, Cuad. admon.ser.organ. Bogotá (Colombia), 24 (43): 133-155, julio-diciembre de 2011

Franco, M. e Barbeira, M. (2009) Um Sistema de Gestão de conhecimento como fomentador de Redes Estratégicas Inter-organizacionais**,** RIAE – Revista Ibero-Americana de Estratégia ISSN: 2176-0756.

Franco, M., & Mariano, S. (2007) Information technology repositories and knowledge management processes. *Journal of Information and Knowledge Management Systems, 37*(4), 440-451.

Gest, S.D., Osgood, D.W., Feinberg, M.E., Bierman, K.L. and Moody, J., (2011). Strengthening prevention program theories and evaluations: Contributions from social network analysis. *Prevention Science*, 12 (4), 349-360.

Glick, W., Huber, G. et al. (1993) "Studying Changes in Organizational Design and Effectiveness: Retrospective Event Histories and Periodic Assessments", in Huber, G. e . Glick, W. (Eds.), Organizational Change and Redesign, Oxford: Oxford University Press, p. 411-433.

Godoy, Arilda S; (2006) Pesquisa Qualitativa em estudos organizacionais: paradigmas, estratégias e métodos; São Paulo: Saraiva, 2006

Grant, R. M. (2006) The knowledge-based view of the firm. In D. Faulkner & A. Campbell,

Strategy: a strategy overview and competitive strategy (pp. 203-227). Oxford: University Press.

Gulati, R. (2007). Silo busting: transcending barriers to build high growth organizations. *Harvard Business Review*, *85*(5), 98-108.

Gulati, R., M. Sytch, and A. Tatarynowicz (2012) "The rise and fall of small worlds: Exploring the dynamics of social structure."Organization Science, 23: 449–471.

Hartley, J. (1994) "Case Studies in Organizational Research", in Cassel, C. e Symon, G. (Eds.), Qualitative Methods in Organizational Research, Thousand Oaks, CA: Sage, p. 208-229.

Inkpen, A. C., & Tsang (2005) Creating knowledge through collaboration. *California Management Review*, *39*(1), 123-140.

Jacometti, M.; Gonçalves, S. ;Castro, G. (2015) Institutional Work Y Knowledgein Networkin Interorganizational: A Proposal to Investigate LPAs

Lee, T. (1999) Using Qualitative Methods in Organizational Research, Thousand Oaks, CA: Sage.

Klein, L. Pereira, B. (2016) The survival of inter-organizational networks: a proposal based on resource dependence theory, RAM, REV. ADM. MACKENZIE, *17*(4), SÃO PAULO, SP, JUL./AGO. 2016. SSN 1518-6776 (impresso), ISSN 1678-6971 (*on-line*)

Kostiainen, J. (2002) Learning and the "Ba" in the development network of an urban region. *European Planning Studies*, v. 10, n. 5.

McNamara, P., Pazzaglia, F., & Sonpar, K. (2015). Large-scale events as catalysts for creating mutual

dependence between social ventures and resource providers. *Journal of Management, 20*(10), 1-31.

Michelis, G. (2001) Cooperation and knowledge creation. In: Nonaka, I.; Nishiguchi, T. (Eds.) *Knowledge emergence*. New York: Oxford.

Nonaka, Ikujiro; Takeuchi, Hirotaka (1997) The Knowledge – Creating Company: How Japanese Companies create the Dinamics of Innovation, Oxford University Press, New York.

Nonaka, I., Nishigushi, T. (2001). Knowledge emergence: social, technical and evolutionary dimensions of knowledge creation. New York: Oxford University Press.

Nonaka, I.; Konno, N. (2001) The concept of "Ba": building a foundation for knowledge creation . California Management Review, v.40, n.3, p.40-55, 1998.

Nonaka, I.; Toyama, R.; Konno, (2002) N. SECI, Ba and Leadership: a unified model of dynamic knowledge creation. Long Range Planning, Oxford, v.33, n.1, p.5-34.

Nonaka, I.; Konno, N. (1998). The Concept of "Ba": building a foundation for knowledge creation. California Management Review, Berkerley, v.40, n.3, p.40-54, Spring.

Nonaka, I.; Toyama, R.; Nagata, (2000) A. A Firm as a Knowledge-creating Entity: a new perspective on the Theory of the firm. Industrial and Corporate Change, v.9, n.1, p.1-20.

Nonaka, I.; Toyama, R. (2003). The Knowledge-creating theory revisited: knowledge creating as synthesizing process. Knowledge Management Research and Practice, Hampshire, v.1, n.1, p.2-10, July.

Nonaka, I. (2005). Managing Organizational Knowledge: Theoretical and Methodological Foundations. In K.G. Smith & M.A. Hitt (Eds.), *The Great Minds in Management* (pp.373-393). New York: Oxford University Press.

Perrow, C. (1992) Small-Firm Networks. In: NOHRIA, N.; ECCLES, R. (Eds.) *Networks and organizations: structure, form and action*. Boston: Harvard Business School Press.

Provan, K.G., Fish, A. and Sydow, J. (2007) Interorganizational Networks at the Network Level: A Review of the Empirical Literature on Whole Networks. *Journal of Management*,

33 (3), 479-516. Doi: 10.1177/0149206307302554

Provan, K.G., Huang, K. and Milward, B. 2009. The Evolution of Structural Embeddedness

and Organizational Social Outcomes in a Centrally Governed Health and Human Services Network. *Journal of Public Administration Research and Theory,* 19, 873- 893. Doi: 10.1093/jopart/mun036

Schiele, H., Ellis, S. C., Eßig, M., Henke Jr., J. W., & Kull, T. J. (2015). Managing supplier satisfaction: social capital and resource dependence frameworks. *Australasian Marketing Journal, 23*(2), 132-138.

Sher, J. P., & Lee, C. V. (2004) Information technology as a facilitator for enhancing dynamic capabilities through knowledge managements. *Information and Management*, *41*(8), 933-945.

Soo, C.; Devinney, T.; Midgley, A. (2002) Knowledge management: philosophy, processes and pitfalls. *California Management Review,* v. 44, n. 4.

Spencer, J. W. (2003) Firms' knowledge-sharing strategies in the global innovation system: empirical evidence from the flat panel display industry. *Strategic Management Journal*, v. 24, n. 3, p. 217-33.

Suire, R., and Vicente, J. (2008) Théorie économique des *clusters* et management des réseaux d'entreprises innovantes. *Revue Française de Gestion, 184*, 119-136.

Passos, C. (2019). Dynamics of knowledge creation in intra-organizational networks.
Millenium, 2(8), 55-66.
DOI: https://doi.org/10.29352/mill0208.05.00192

Sundaresan, S. and Zhang, Z. (2017) Incentive Strategies for Facilitating Knowledge Sharing on an Enterprise Social Network, Northeast Decision Sciences Institute 2017 Annual Conference

Szell, M., R. Lambiotte, and S. Thurner (2010) ''Multi-relational organization of large-scale social networks in an online world.'' PNAS, 107: 13636–13641.

Tatarynowicz, T.; Sytch, M.; Gulati, R. (2016) Environmental Demands and the Emergence of Social Structure: Technological Dynamism and Interorganizational Network Forms, Administrative Science Quarterly 2016, Vol. 61(1)52–86

Teece, D.; Rumelt, R.; Dosi, G.; Winter, S(1994) Understanding corporate coherence: theory and evidence. *Journal Economic Behavior*, v. 23, p. 1-30.

Tsai W. (2002) Social structure of "coopetition" within a multiunit organization: coordination, competition, and intraorganizational knowledge sharing. *Organization Science*, v. 13, n. 2, p. 179-90.

Uzzi, B., and J. Spiro (2005) ''Collaboration and creativity: The small world problem.'' American Journal of Sociology, 111: 447–504.

Valente, T.W., Palinkas, L.A., Czaja, S., Chu, K.H. and Brown, C.H., (2015) Social network analysis for program implementation. *PloS One*, 10 (6), e0131712.

Vidal, Ignácio Ramos,(2017) Detecting key actors in interorganizational networks, *Cuadernos de Gestión* Vol. 17 - Nº 2 (2017), pp. 63-86 - ISSN: 1131 – 6837

Yin, R. (1994) Case Study Research - Design and Methods, Thousand Oaks, CA: Sage.Yin, R. K. (2001) *Estudo de caso: planejamento e métodos*. Porto Alegre: Bookman.

CIÊNCIAS AGRÁRIAS, ALIMENTARES E VETERINÁRIAS

AGRICULTURAL SCIENCES, FOOD AND VETERINARY

CIENCIAS AGRÍCOLAS, ALIMENTOS Y VETERINARIA

millenium

CIÊNCIAS AGRÁRIAS, ALIMENTARES E VETERINÁRIAS
AGRICULTURAL SCIENCES, FOOD AND VETERINARY
CIENCIAS AGRÍCOLAS, ALIMENTOS Y VETERINARIA

millenium 09

Millenium, 2(8), 69-78.

en

EFEITO DOS HIDROCOLÓIDES NO REVESTIMENTO E NAS PROPRIEDADES ORGANOLÉTICAS DO RISSOL REGENERADO EM FORNO E COMPARAÇÃO COM O PROCESSO DE FRITURA

HYDROCOLLOIDS EFFECT ON COATING BATTER AND ON ORGANOLEPTIC PROPERTIES OF RISSOL REGENERATED IN OVEN AND COMPARISON WITH DEEP-FRYING PROCESS

EFECTO DE LOS HIDROCOLOIDES EN EL REVESTIMIENTO Y EN LAS PROPIEDADES ORGANOLÉTICAS DEL RISSOL REGENERADO EN HORNO Y COMPARACIÓN CON EL PROCESO DE FRITURA

Ana Patrícia Sousa[1]
Manuela Vaz Velho[1]
Manuel Alves[1]
Rita Pinheiro[1]

[1] Polytechnic Institute of Viana do Castelo, Viana do Castelo, Portugal

Ana Sousa - apatriciasousa@estg.ipvc.pt | Manuela Vaz Velho - mvazvelho@estg.ipvc.pt | Manuel Alves - mruialves@estg.ipvc.pt | Rita Pinheiro - ritapinheiro@estg.ipvc.pt

Corresponding Author
Ana Patrícia Sousa
Instituto Politécnico de Viana do Castelo
Rua Escola Industrial e Comercial de Nun'Álvares, n.º 34
4900-347 Viana do Castelo
apatriciasousa@estg.ipvc.pt

RECEIVED: 29th May 2018
ACCEPTED: 03th January 2019

Sousa, A. P., Velho, M. V., Alves, M. & Pinheiro, R. (2019). Hydrocolloids effect on coating batter and on organoleptic properties of rissole regenerated in oven and comparison with deep-frying process. *Millenium*, 2(8), 69-78.
DOI: https://doi.org/10.29352/mill0208.06.00198

RESUMO

Introdução: A preferência dos consumidores por produtos com baixo teor de gordura continua a aumentar e há uma pressão significativa na indústria para reduzir processos de fritura.
Objetivos: Avaliar o efeito dos hidrocolóides no revestimento do *Rissol*, evitando os processos de pré-fritura ou fritura. Pretende-se também comparar as características físico-químicas do *Rissol* obtido, usando o processo de regeneração em forno e o tradicionalmente frito, e a aceitabilidade do consumidor do *Rissol* regenerado em forno.
Métodos: Hidrocolóides como carboximetilcelulose, hidroxipropilmetilcelulose, metilcelulose, gomas xantana, karaya e alfarroba, foram utilizadas sozinhas no sistema de revestimento, a uma concentração de 0.5% e 1.5%(p/p), assim como a sinergia estabelecida entre pares de gomas com concentração final de 1% (p/p). O *Rissol* foi regenerado em forno (200°C; 18 minutos). Analisou-se o efeito na cor, textura instrumental, humidade e propriedades organoléticas (análise descritiva quantitativa). O teor em proteínas, hidratos de carbono, gordura total, fibra, cloreto de sódio, humidade e a aceitabilidade do consumidor no *Rissol* com 0.5%(p/p) de carboximetilcelulose foi determinado, para comparar os processos de cozimento em forno e fritura.
Resultados: O uso de 0,5%(p/p) de hidrocolóides adicionado ao sistema de revestimento oferece melhores perspetivas para a produção de um produto sem pré-fritura. As formulações com 1,5%(p/p) apresentaram uma aparência visual pior com fissuras e grumos.
Conclusão: O uso de carboximetilcelulose no sistema de revestimento do *Rissol* pode ser uma alternativa para obter um produto com um teor de gordura reduzido e com resultados positivos de aceitação pelo consumidor (com regeneração em forno).

Palavras-chaves: Hidrocolóides; Carboximetilcelulose; solução de revestimento; conteúdo em gordura;

ABSTRACT

Introduction: Consumers' preference for lower fat products continues to increase and there is a significant pressure on industry to reduce the use of frying processes.
Objetives: Evaluate the effect of hydrocolloids on the *Rissol* coating, avoiding the pre-frying or frying processes. It's also intended to compare physicochemical characteristics of the *Rissol* obtained, using oven regeneration process and traditional deep-frying, and consumer's acceptability of oven regenerated *Rissol*.
Methods: Hydrocolloids such as carboxymethylcellulose, hydroxypropylmethylcellulose, methylcellulose, xanthan, karaya and locust bean gums, were used alone in the coating system at concentrations of 0.5% and 1.5%(w/w), as well as the synergies established between pairs of gums to a final concentration of 1%(w/w). Rissol was regenerated in the oven (200°C, 18 minutes) and instrumental texture, colour, moisture and organoleptic properties (quantitative descriptive analysis) were measured.
The contents in protein, carbohydrates, total fat, fibre, sodium chloride and moisture and consumer's acceptability in *Rissol* with 0.5%(w/w) of carboxymethylcellulose were determined, to compare deep-frying and oven cooking processes.
Results: The addition of 0.5%(w/w) of any of hydrocolloids studied to the coating system offers better prospects to produce an oven product without pre-frying. The formulations with 1.5%(w/w) of hydrocolloid showed a poorer appearance with cracks and lumps.
Conclusion: The use of carboxymethylcellulose in coating batter could be an alternative to obtain a product with reduced total fat and with positive consumer acceptability results (with oven regeneration).

Keywords: Hydrocolloids; Carboxymethylcellulose; Batters; fat content

RESUMEN

Introducción: La preferencia de los consumidores por productos con bajo contenido de grasa sigue aumentando y hay una presión significativa en la industria para reducir el uso de procesos de fritura.
Objetivos: Evaluar el efecto de los hidrocoloides en el revestimiento del *Rissol*, evitando los procesos de pre-fritura o fritura. También se pretende comparar las características fisicoquímicas del *Rissol* obtenido, utilizando el proceso de regeneración en horno y el tradicionalmente freído y la aceptabilidad del consumidor del *Rissol* regenerado en horno.
Métodos: Hidrocoloides tales como carboximetilcelulosa, hidroxipropilmetilcelulosa, metilcelulosa, gomas xantana, karaya y algarroba, se usaron solos en el sistema de revestimiento a concentraciones de 0.5% y 1.5% (p/p), así como las sinergias establecidas entre pares de gomas a una concentración final de 1%(p/p). El *Rissol* fue regenerado en horno (200°C, 18 minutos). Se analizó el efecto en el color, textura instrumental, humedad y propiedades organoléticas (análisis descriptivo cuantitativo).
El contenido en proteínas, hidratos de carbono, grasa total, fibra, cloruro de sodio, humedad y la aceptabilidad del consumidor en el *Rissol* con 0.5% (p/p) de carboximetilcelulosa fue determinado, para comparar los procesos de cocción en horno y fritura.

Sousa, A. P., Velho, M. V., Alves, M. & Pinheiro, R. (2019). Hydrocolloids effect on coating batter and on organoleptic properties of rissole regenerated in oven and comparison with deep-frying process. *Millenium*, 2(8), 69-78.
DOI: https://doi.org/10.29352/mill0208.06.00198

Resultados: El uso del 0,5% (p/p) de hidrocoloide agregado al sistema de recubrimiento ofrece mejores perspectivas para la producción de un producto sin pre-fritura. Las formulaciones con 1,5% (p/p) presentaron una apariencia visual peor (fisuras y grumos).
Conclusións: El uso de carboximetilcelulosa en el sistema de revestimiento del *Rissol* puede ser una alternativa para obtener un producto con un contenido de grasa total reducido y con resultados positivos de aceptación por el consumidor (con regeneración del horno).

Palabras Clave: hidrocoloides; carboximetilcelulosa; solución revestimiento; contenido en grasa;

INTRODUCTION

The high consumption of lipids has been related to obesity and other health problems, such as cardiac diseases (Gadiraju et al., 2015). World Health Organization (WHO) encouraged the nutritional reformulation of food products through an articulated action with Food Industry, especially in products with high quantities of sugar, salt and fat (World Health Organization, 2015). In fried foods, the application of batter and breading provides many opportunities to develop flavours and textures (Xue & Ngadi, 2007; Brannan et al., 2014). Batter is a liquid dough containing essentially flour and water, but may contain other ingredients, for example, egg, starch, salt, and others (Fiszman & Salvador, 2003; Yilmaz et al., 2017). The main purpose of batter is to create a layer to which the breading adheres completely. In addition to this function, batter acts as a barrier to prevent moisture loss, thus ensuring an end product with a smooth and juicy inside, and crispy outside (Albert, et al., 2009). Manipulating the coating (batter and breading system) of some foods can be a good method to reduce fat absorption during the frying process, while promoting the retention of water inside (Singthong & Thongkaew, 2009). Polysaccharides, have been shown to be effective in reducing the amount of absorbed oil (Fiszman & Salvador, 2003; Brannan et al., 2014). Hydrocolloids have functions as thickeners, gelling agents, emulsifiers, stabilizers, fat replacers, clarifying agents, flocculating agents, clouding and whipping agents. In addition, they have applications in the areas of edible films and crystallization inhibition (Li & Nie, 2016). The use of hydrocolloids in batters, with the formation of a thin and invisible film, besides reducing oil absorption, it also contributes to the improvement of the viscosity, adhesiveness, mechanical resistance of the crust and pickup control. It also helps in the control and stability of the freezing/defrosting processes and maintains crispness of the fried breaded foods (Fiszman & Salvador, 2003; Varela & Fiszman, 2011). Many hydrocolloids are highly hydrophilic, requiring the adjustment of the solids/water ratio of the formulation, so that the hydrocolloid efficiency is not affected due to competition for the available water (Meyers & Grazela, 2011). Among the hydrocolloids, hydrophilic polymers (e.g., alginates, carrageenans, carboxymethylcelluloses, pectins, xanthan gum) are widely used as film formers because they preserve the texture, flavour, and shelf life of foods. Among the wide variety of gums used as fat barriers, the most studied have been cellulose esters, methylcellulose (MC) and hydroxypropylmethylcellulose (HPMC) (Varela & Fiszman, 2011), which exhibit unique properties of reversible thermal gelation (Xue & Ngadi, 2007; Varela & Fiszman, 2011). When MC or HPMC is added to the coating solution, it gels immediately when the product comes into contact with the hot oil. This property, coupled with its high water retention capacity, allows these gums to protect food from water loss from the substrate and therefore prevent oil absorption during the frying process (Meyers & Grazela, 2011). One factor that has been associated with barrier efficacy is the MC concentration in the coating solution. Sanz et al. (2004a) found that the increase in MC concentration from 1 to 2% (w/w) leads to a lower oil absorption and a greater reduction of moisture on the crust of the coated squid rings, both in pre-frying and in final frying following freezing. The same authors found that another factor that has been related to barrier efficiency is the temperature of the solution at the time of coating application. The application of MC is its use in industrial process of frozen food, which uses its thermogelling properties to eliminate the pre-frying phase (Sanz et al., 2004b). Carboxymethylcellulose (CMC) is another hydrocolloid, derived from cellulose, which has been extensively studied (Varela & Fiszman, 2011). However, it has been reported to be less efficient than MC and HPMC (Varela & Fiszman, 2011). Ajo (2017) investigated the effects of using carrageenan and xanthan thin coating films to reduce oil absorption in fried potato chip-based pellets and concluded that these hydrocolloids significantly reduced oil absorption across all concentrations, and the most effective level of fat reduction was found using 4% (w/w) carrageenan and 0.3% (w/w) xanthan coatings.
Regeneration in the oven at higher temperatures results in better evaluations regarding characteristics such as 'roast', 'roasted' and 'bitter', which can be related to the fact that high temperatures favour the Maillard reactions without, however, implying a decrease in the development of the characteristic flavour (Byrne, Bredie, Mottram, & Martens, 2002).
Rissol is a traditionally consumed food product in Portugal, whose wrapping dough is pre-cooked, stuffed with meat, fish or vegetables and coated with egg (batter) and breadcrumb (breading) before being fried.
The aim of this study was to evaluate the effect of the hydrocolloids on the *Rissol* coating, avoiding the processes of pre-frying or frying. The effect of different hydrocolloids (Carboxymethylcellulose, methylcellulose, hydroxypropylmethylcellulose, xanthan

Sousa, A. P., Velho, M. V., Alves, M. & Pinheiro, R. (2019). Hydrocolloids effect on coating batter and on organoleptic properties of rissole regenerated in oven and comparison with deep-frying process. *Millenium*, 2(8), 69-78.
DOI: https://doi.org/10.29352/mill0208.06.00198

gum, karaya gum and locust bean gum) incorporated in the coating solutions on colour, instrumental texture, moisture and organoleptic properties were analysed. Comparisons of physicochemical characteristics and energetic values of the *Rissol* with CMC in the batter solution, was also studied, using the oven regeneration method and the traditional deep-frying, as well as consumer's acceptability of oven regenerated *Rissol* were also carried out.

1. METHODS

1.1 Sample

1.1.1 Effect of different hydrocolloids in batter solution

Batter solution: xanthan gum (XA), locust bean gum (AL), karaya gum (KA), carboxymethylcellulose (CMC) (Formulab, Ltd), hydroxypropylmethylcellulose (HPMC) (Methocel K4M - Dow Chemical Company) and methylcellulose (MC) (Capri, Ltd.), at concentrations of 0.5% and 1.5% (w/w), were used for each hydrocolloid. The effect of the combination of different hydrocolloid doubles was also analysed at a concentration of 0.5:0.5 - 1% (w/w) - MC + KA, MC + AL, MC + XA, CMC + KA, CMC + AL, CMC + XA. The solutions were prepared by diluting the hydrocolloid in distilled water, and letting it stand for 24 hours before use.
Preparation of the samples: A meat *Rissol*, supplied by a local company, was used as sample. *Rissol* formulation was: 65% dough (60% of wheat flour; 39% of water and 1% of salt) and 35% meat mixture (40% of water, 35% of pork meat and 25% of wheat flour). In the coating system, the sample was dipped (± 20 °C) into the batter liquid solution (hydrocolloid solution). The batter was drained from the sample during 10 seconds. After this, the breading step with breadcrumb was followed. The samples were frozen (-18 ºC) for 1 hour in a quick freezer equipment (Mercatus, Italy) and stored at the same temperature.
Samples were regenerated in a conventional electric oven (FE 2/3, Italy) at 200 °C ± 5 °C for 18 to 20 minutes.

1.1.2 Comparison between two cooking methods

Samples followed the same preparation method described above. A batter solution containing 0.5% of CMC was used in the coating system. After being immersed in the solution, the *Rissol* was breadcrumbs. Subsequently, *Rissol* was regenerated in the oven, at 200 °C for 18 to 20 minutes or deep-fried with sunflower oil at 180 °C for 12 minutes.

1.2 Procedures

1.2.1 Effect of different hydrocolloids in batter solution

A texture profile analysis test (TPA) using a texture analyser (TA.XT2i) equipped with a cylindrical probe (P/4) was used with a force and speed of 0.049 N and 5 mm/s, respectively. A colour analyser (Minolta CR-300), using CIE L* a* b* colour space, recording the values of brightness L*, chromaticity a* (red-green) and b* (yellow-blue). Moisture was determined according to ISO 1442:1997, by gravimetric method, by means of the evaluation of mass loss up to constant weight in an oven (Heraeus, Germany) regulated at 103 ± 2 °C. Five replicates were performed for each sample (T = 20 °C) for texture and six for colour and moisture. A quantitative descriptive analysis (QDA) was performed by a sensory trained panel with 14 elements, involving previous sessions for main descriptors definition, their scale limits as well as verbal anchors, using the very same type of commercial *Rissol* (ISO 8587,2006). The samples were evaluated for general appearance, smell, texture appreciated with cutlery and texture in mouth, with a 9 – point scale anchored at the ends with 1 – little / absent and 9 – very / intense (Stone & Sidel, 2004)

1.2.2 Comparison between two cooking methods

The protein content was determined by the Kjeldahl method, described by ISO 937:1978. A digestion unit (Buchi, Switzerland) and a Vapodest distillation unit (Gerhardt, Germany) were used. The conversion of the total nitrogen content to crude protein content was obtained by the standard conversion factor (6.25). The fat content was determined by the Soxhlet method, described by ISO 1443:1973. The carbohydrates were determined by the 3,5-dinitrosalicylic acid colorimetric method (DNS) (James, 1995). The absorbance were measured on the spectrophotometer (Varian Cary 50, California) at 540 nm. The dietary fibre was determined by the Ceramic Fibre Filter Method, described by AOAC 962.09:1995. The sodium chloride content was determined by Volhard method, described by ISO 1841-1:1996. The moisture was determined following the method described above ISO 1442:1997. All analysis were performed in triplicate. The energy value was calculated according to conversion factors, according to Regulation (EU) 1169:2011.

1.2.3 Consumer acceptance test

The consumers' acceptance was evaluated in a global way, using *Rissoles* with 0.5% CMC (w/w) in the coating system and regenerated in the oven. Consumers (N=73) were originally from the North region of Portugal and select with ages older than 18 years old. A survey was used and consumers would have to identify age and sex. A 5-point hedonic scale ranking from 1= dislike very much to 5= like very much was used to evaluate acceptance (Meilgaard, Civille, & Carr, 1999)

1.3 Statistical analysis

The effect of different hydrocolloids in batter solution (except sensory analysis) were analysed using analysis of variance (*one-way* ANOVA), followed by Tukey's HSD test. Sensory analysis was evaluated by principal component analysis (PCA) with predictive biplots (Alves, 2012). The comparison between two cooking methods was analysed using Student's t-test. Statistical significance was set at *p* values less than 0.05. All analyses were performed with the R Project for Statistical Computing program (www.r-project.org).

2. RESULTS AND DISCUSSION

2.1 Effect of different hydrocolloids in batter solution

Regarding texture results, it was observed that samples with a concentration of 0.5% (w/w) present higher hardness values than those with 1.5% (w/w) (p<0.05) regardless of the type of hydrocolloid used (Figure 1- left). This behaviour may be due to a greater dehydration of the *Rissol* during its regeneration in the oven by the lower concentration of hydrocolloid, 0.5%, making its hardness higher than 1.5% (w/w) of hydrocolloid. Samples with a concentration of 0.5% (w/w) of CMC and HPMC showed the highest hardness values. On the contrary, samples with 1.5% (w/w) of LA revealed the lowest hardness values. Samples with 1.5% (w/w) of hydrocolloids showed not differences in hardness results (p<0.05). It was also observed that the XA gum shows similar values for the hardness for two concentrations (p<0.05). On the contrary, Lazaridou, Duta, Papageorgiou, Belc, & Biliadereis (2007) found in a rheology study on gluten-free bread that XA gum, in concentrations of 1 and 2%, enhances the effect of bread hardness and elasticity . Guarda, Rosell, Benedito, & Galotto (2004), found that a concentration of 0.1% (w/w) of this hydrocolloid increased the hardness in the bread crumb, maintaining that effect constant at higher concentrations. However, it must be pointed out that the food matrix analysed was different from *Rissol*. The results obtained for the elasticity (Figure 2-left) indicated that, in general, the elasticity is higher for samples with 0.5% (w/w) of hydrocolloid, the highest values were with concentrations of 0.5% (w/w) (CMC and AL). The synergies between the hydrocolloid pairs (Figure 1, right), both in the hardness values and in the elasticity values (Figure 2, right) did not revealed any tendency, although there were significant differences between the samples (p<0.05).

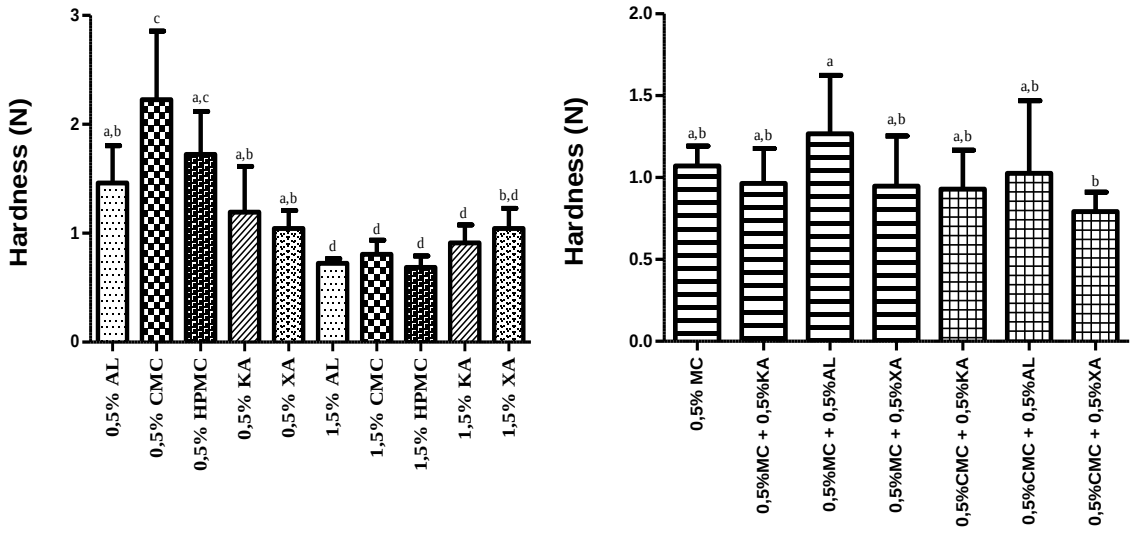


Figure 1 – Effect of the hydrocolloids on the hardness (N) of the *Rissol*, in concentrations (0.5% and 1.5%) (on the left), and in different pairs of hydrocolloids (with the exception of 0.5% MC) (right). Mean values ± standard deviation of five replicates for each sample; values with the same letter are not significantly different (p<0.05)

Sousa, A. P., Velho, M. V., Alves, M. & Pinheiro, R. (2019). Hydrocolloids effect on coating batter and on organoleptic properties of rissole regenerated in oven and comparison with deep-frying process. *Millenium*, 2(8), 69-78.
DOI: https://doi.org/10.29352/mill0208.06.00198

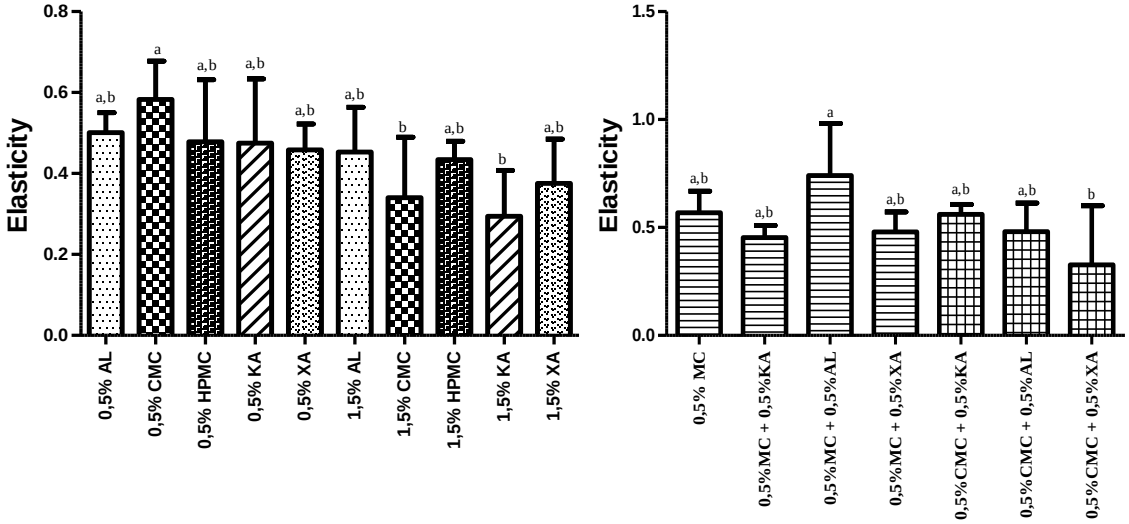


Figure 2 - Effect of the hydrocolloids on the elasticity of the *Rissol*, in concentrations (0.5% and 1.5%) (on the left), and in different pairs of hydrocolloids (with the exception of 0.5% MC) (right). Mean values ± standard deviation of 5 replicates for each sample; values with the same letter are not significantly different (p<0.05).

Colour results showed that there were significant differences between the samples with different hydrocolloids *(p*<0.05) (Table 1) (L*, a*, b*). L* values obtained were between 54.29 (± 3.29) and 62.85 (± 1.15). Samples with 0.5% (w/w) of CMC and 0.5% (w/w) of AL presented the highest values (62.85 and 61.15, respectively) and 0.5% (w/w) of HPMC and 1.5% (w/w) of KA presented the lowest values of all experiments (54.92 and 54.29, respectively). The higher L* values, the lighter are the samples, on the contrary, the lower L* values, the darker are the samples. Regarding other colour parameters, greenness (a*) and yellowness (b*), significant differences between the samples were also found (p<0.05). a* values obtained ranged from 9.23 (± 0.55) to 12.84 (± 0.18). Samples with 0.5% (w/w) of HPMC and 0.5% (w/w) of KA presented the highest values (12.84 and 12.67, respectively) and 0.5% (w/w) of AL and 0.5% (w/w) of CMC presented the lowest values of all experiments (9.23 and 10.09, respectively). b* values obtained ranged from 26.51 (± 0.84) to 31.03 (± 1.09). Samples with 0.5% (w/w) of KA and 0.5% (w/w) of HPMC presented the highest values (31.03 and 29.50, respectively) and 1.5% (w/w) of KA and 0.5% (w/w) of AL presented the lowest values of all experiments (26.51 and 26.59, respectively). The synergies of the hydrocolloid pairs (Table 2) did not show any tendency, although there were significant differences between the samples (*p*<0.05). According to Varela & Fiszman (2011), the final appearance of the breaded product depends entirely on its external characteristics, and, in this case, it is verified that the hydrocolloid does not influence the colour of the product, since the colour is very influenced by the breadcrumb.
Moisture results showed that the values varied between 38.57 (± 2.56) % and 47.22 (± 1.83) % (Table 1), for hydrocolloids 1.5% (w/w) of KA and 1.5% (w/w) of CMC, respectively, although there were no significant differences between the samples *(p*<0.05). The synergies of the hydrocolloid pairs did not show any tendency (Table 2), there were no significant differences between the samples (p<0.05). Gonçalves (2015), found that the L* values decreased significantly with the increase of the regeneration time of commercial chicken nuggets.

Table 1 - Effect of the hydrocolloids on the batter in the parameters L*, a*, b* of the colour, and moisture (%), in concentrations (0.5% and 1.5%). Mean values ± standard deviation of 6 replicates for colour and 3 replicates for moisture; Values followed by the same letter in the same column are not significantly different (p<0.05)

Formulations	Colour			Moisture (%)
	*L**	*a**	*b**	
0.5% AL	61.15 (± 3.01)a,b,f	9.23 (± 0.55)a	26.59 (± 0.96)a,b	42.35 (± 3.76)a
0.5% CMC	62.85 (± 1.15)a,g	10.09 (± 0.21)a,c	27.67 (± 0.46)b,f	43.85 (± 2.08)a
0.5% HPMC	54.92 (± 0.85)cd	12.84 (± 0.18)b	29.50 (± 0.50)c,d	42.84 (± 5.46)a
0.5% KA	57.62 (± 1.60)b,d,f	12.67 (± 0.58)b,d	31.03 (± 1.09)a,d	41.98 (± 2.40)a
0.5% XA	58.41 (± 3.54)a,b,c,d,f	10.62 (± 1.93)a,c	28.13 (± 1.31)a,b,c	45.99 (± 3.05)a
1.5% AL	58.33 (± 2.30)b,c,d,f	11.27 (± 1.00)b,c	28.39 (± 1.10)a,c,e	45.30 (± 1.80)a
1.5% CMC	58.58 (± 2.06)a,b,c,d,f	11.69 (± 0.81)b,c	28.81 (± 1.46)a,c,e	47.22 (± 1.83)a
1.5% HPMC	55.65 (± 0.95)d,e	10.95 (± 0.88)c	27.60 (± 1.67)e,f	45.21 (± 0.96)a
1.5% KA	54.29 (± 3.29)d	11.08 (± 0.50)c,d	26.51 (± 0.84)c,e	38.57 (± 2.56)a
1.5% XA	59.66 (± 2.74)e,f,g	10.98 (± 0.52)c	28.11 (± 1.36)a,b,d,f	44.45 (± 2.82)a

Table 2 - Effect of the hydrocolloids on the batter in the parameters L*, a*, b* of the colour, and moisture (%) in different pairs of hydrocolloids (with the exception of 0.5% MC). Mean values ± standard deviation of 6 replicates for colour and 3 replicates for moisture; Values followed by the same letter in the same column are not significantly different (p<0.05)

Formulations	Colour			Moisture (%)
	*L**	*a**	*b**	
0.5% MC	60.86 (± 1.46)a	10.96 (± 0.65)a	28.96 (± 0.76)a	48.68 (± 1.60)a
0.5% MC + 0.5% KA	59.97 (± 0.73)a,c	11.28 (± 0.70)a	28.99 (± 0.92)a	44.01 (± 2.39)a
0.5% MC + 0.5% AL	55.77 (± 1.64)b	9.75 (± 0.29)b	24.96 (± 0.99)b	46.11 (± 1.00)a
0.5% MC + 0.5% XA	57.66 (± 2.76)b,c	11.30 (± 1.04)a	28.70 (± 1.64)b	44.72 (± 2.73)a
0.5% CMC + 0.5% KA	61.18 (± 1.85)a	11.14 (± 0.42)a	29.20 (± 1.01)a	43.26 (± 2.90)a
0.5% CMC + 0.5% AL	59.26 (± 0.73)a,c	11.53 (± 0.20)a	29.15 (± 0.48)a	46.53 (± 2.02)a
0.5% CMC + 0.5% XA	59.48 (± 1.14)a,c	10.98 (± 0.52)a	28.35 (± 0.51)a	44.72 (± 2.73)a

Regarding the sensory analysis, as shown in Figure 3, the principal components 1 and 2 explain 54% of the total information, reflecting main observed variations among the different samples, in the attributes T1d (texture/hardness on the first bite), mc (chewing - crispness), at (aspect -characteristic texture), cc (characteristic colour) and af (visual aspect - cracks). It can then be said that the panellists have privileged the aspect (characteristic aspect, cracks, and characteristic texture) and chewiness (crispness and hardness on the 1st bite). The characteristic texture aspect correlates positively with the characteristic smell and negatively with visible cracks. It can be seen that CMC gives a more characteristic general appearance, with values between 6 - 6.5, whereas, for example, XA and KA gums give a less characteristic colour (for the 2 concentrations). *Rissol* with MC, CMC + AL, CMC + XA and CMC presented lower hardness on the first bite and less crispness; the use of HPMC demonstrated the same but less extreme behaviour. *Rissol* with MC + XA and AL1 presented a less characteristic appearance, a greater hardness, and greater crispness, whereas with AL2 they are identical with those with AL1, but with a more characteristic appearance.

Sousa, A. P., Velho, M. V., Alves, M. & Pinheiro, R. (2019). Hydrocolloids effect on coating batter and on organoleptic properties of rissole regenerated in oven and comparison with deep-frying process. *Millenium*, 2(8), 69-78.
DOI: https://doi.org/10.29352/mill0208.06.00198

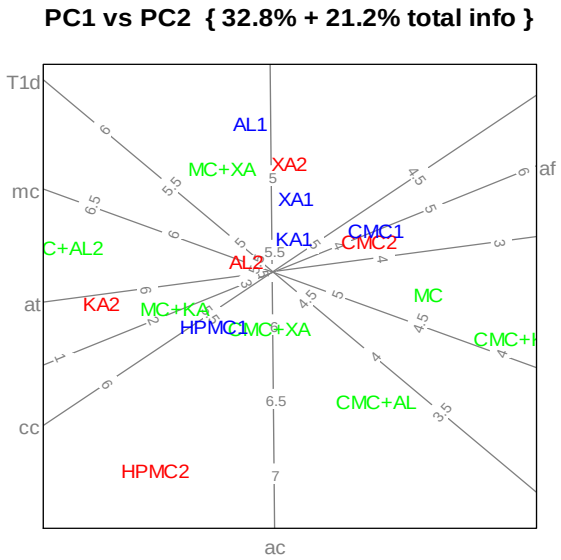


Figure 3 – Predictive PCA biplot of attributes evaluated in sensory analysis. AL1, CMC1, HPMC1, XA1 correspond to 0.5% concentrations. AL2, CMC2, HPMC2, correspond to 1.5% concentrations. The remainder correspond to the different pairs of hydrocolloids. Principal components: T1d (texture/hardness on the first bite), mc (chewing - crispness), at (visual aspect-characteristic texture), cc (characteristic color) and af (visual aspect - cracks).

Coating products are, mainly, evaluated by consumers on the basis of quality attributes including appearance, taste and smell, moisture and fat content, texture and crispness, which is perhaps one of the most important properties which determine consumer acceptance, looking for a product with a crunchy outer layer that contrasts with the soft interior (Piqueras-Fiszman, Varela, & Fiszman, 2013).

2.2 Comparison between two cooking methods: regenerate in the oven and deep-fried

The *Rissol* produced with a coating system with 0.5% (w/w) of CMC was regenerated in oven and in deep-frying. The results of protein content, carbohydrates, dietary fibre, and sodium chloride showed no significant differences between the two regenerated cooking methods (p<0.05) (Table 3). On the contrary, total fat content and moisture presented significant differences between the two cooking methods (p<0.05). The fat content of *Rissol* regenerated by the frying method has 7-fold higher, than the oven regenerated *Rissol*, 15.10 (± 0.00) % and 2.02 (± 0.00) %, respectively. The deep-fried *Rissol* presented lower moisture content, 46.10 (±0.01) %, than the oven regenerated *Rissol*, 51.84 (±0.01) %, meaning that the frying leads to higher water loss than oven process. Same results were found by Gonçalves (2015) who compared the oven regeneration with the frying process of commercial chicken nuggets. According to Varela, Salvador & Fiszman (2008), the regeneration by frying of coating products, fried and frozen leads to products with high moisture in the interior, lower moisture in the outer layer and good crispness. Results showed that oven regenerated *Rissol* contains 167 kcal per 100 g and deep-fried *Rissol* contains 273 kcal per 100 g. These results reflect a 70% decrease in Calories content that the oven regenerated *Rissol* provides, compared to the deep-fried *Rissol.*

Table 3 - Physicochemical characteristics and energetic value of oven regenerated *Rissol* without pre-frying and traditional deep-fried *Rissol*. Mean values ± standard deviation of 3 replicates; Values followed by the same letter in the same column are not significantly different (p<0.05)

	Physicochemical characteristics (%)						Energetic value (kcal)
	Total fat	Protein	Sodium chloride	Carbohydrates	Dietary fiber	Moisture	
Oven method	2.02 (±0.00)a	5.99 (±0.00)a	0.91 (±0.02)a	30.90 (±0.03)a	0.68 (±0.00)a	51.84 (±0.01)a	167
Deep frying method	15.10 (±0.00)b	5.76 (±0.00)a	1.03 (±0.09)a	28.30 (±0.03)a	0.77 (±0.00)a	46.10 (±0.01)b	273

2.2.1 Consumer acceptance test

The consumers' acceptance was evaluated in a global way, using the *Rissol* produced with a coating system with 0.5% (w/w) of CMC and regenerated in the oven. It was found that the majority of consumers scored *Rissol* as "like very much" (50.68%) and "like moderately" (46.58%) (N=73). Only two respondents revealed that "neither like or dislike" (1.37%) and "dislike moderately" (1.37%), and none revealed that "dislike very much". In the study, consumers aged between 18 and 76 participated in the study, with more than half of respondents aged 18-25 years: 18-25 years (61.64%); 26-34 years (17.81%); 35-44 years (13.70%) and 45-76 years (6.85%). Respondents were mostly women (62%).

CONCLUSIONS

Many studies have been done aiming to reduce the fat content in fried products. Developing a coating system capable of suppressing the need for frying or pre-frying a traditional *Rissol*, by replacing it with the oven regeneration, may be an alternative, in order to obtain an acceptable consumer product and nutritionally more suitable. In general, the use of a 0.5% (w/w) of hydrocolloid formulation added to the coating system offers a better alternative for the production of an oven baked product. It was concluded that the formulations with 1.5% (w/w) of hydrocolloid showed a poorer appearance (with cracks and lumps) than the formulations with 0.5% (w/w). The results of the present study showed that carboxymethylcellulose in coating batter of *Rissol* regenerated in oven could be an alternative for reduction of total fat when compared to the traditional deep-fried process.

REFERENCES

Ajo, R. Y. (2017). Application of hydrocolloids as coating films to reduce oil absorption in fried potato chip-based pellets. *Pakistan Journal of Nutrition, 16 (10)*, 805-812. doi:10.3923/pjn.2017.805.812

Albert, A., Perez-Munuera, I., Quiles, A., Salvador, A., Fiszman, S., & Hernando, I. (2009). Adhesion in fried battered nuggets: Performance of different hydrololloids as predusts using tree cooking procedures. *Food Hydrocolloids, 23*, 1443-1448. doi:10.1016/j.foodhyd.2008.11.015

Alves, M. R. (2012). Evaluation of the predictive power of biplot axes to automate the construction and layout of biplots based on the accuracy of direct readings from common outputs of multivariate analyses: 1. application to principal component analysis. *Journal of Chemometrics, 26(5)*. doi:10.1002/cem.2433

AOAC 962.09 (1995). *Official Method Fiber (crude) in Animal Feed.* Official Methods of Analysis of AOAC INTERNATIONAL.

Brannan, R. G., Mah, E., Schott, M., Yuan, S., Casher, K. L., Myers, A., & Herrick, C. (2014). Influence of ingredients that reduce oil absorption during immersion frying of battered and breaded foods. *European Journal of Science and Tecnology 116*, 240-254.

Byrne, D. V., Bredie, W. L., Mottram, D. S., & Martens, M. (2002). Sensory and chemical investigations on the effect of oven cooking on warmed-over flavour development in chicken meat. *Meat sience, 61*, 127-139. doi:10.1016/S0309-1740(01)00171-1

Fiszman, S., & Salvador, A. (2003). Recent developments in coating batters. *Trends in Food Science & Technology, 14*, 399-407. doi:10.1016/S0924-2244(03)00153-5

Gadiraju, T., Patel, Y., Gaziano, J., & Djousse, L. (2015). Fried food consumption and cardiovascular health: A review of current evidence. *Nutrients, 7*, 8424-8430.

Gonçalves, C. M. (2015). Avaliação do impacte dos procedimentos de confecção e regeneração na qualidade de produtos cárneos pré-cozinhados congelados (Tese de mestrado, Universidade Nova de Lisboa, Portugal). https://run.unl.pt/bitstream/10362/16878/1/Goncalves_2015.pdf

Guarda, A., Rosell, C., Benedito, C., & Galotto, M. (2004). Different hydrocolloids as bread improvers and antistaling agents. *Food Hydrolloids, 18*, 241-247. doi:10.1016/S0268-005X(03)00080-8

ISO 1443 (1973). *Determination of total fat content.* Genève: International Organization for Standardization.

ISO 937 (1978). *Determination of nitrogen content.* Genève: International Organization for Standardization.

ISO 1841-1 (1996). *Determination of chloride content-Part1. Volhard method.* Genève: International Organization for Standardization.

ISO 1442 (1997). *Determination of moisture content.* Genève: International Organization for Standardization.

Sousa, A. P., Velho, M. V., Alves, M. & Pinheiro, R. (2019). Hydrocolloids effect on coating batter and on organoleptic properties of rissole regenerated in oven and comparison with deep-frying process. *Millenium*, 2(8), 69-78.
DOI: https://doi.org/10.29352/mill0208.06.00198

ISO 8587 (2006). *Sensory analysis - Methodology - Ranking.* Genève: International Organization for Standardization.

James, S. C. (1995). *Analytical Chemistry of Foods.* Springer-Science+Business Media, B.V.

Lazaridou, A., Duta, D., Papageorgiou, M., Belc, N., & Biliaderis, C. (2007). Efects of hydrocolloids on dough rheology and bread quality parameters in gluten-free formulations. *Journal of Food Engineering, 79*, 1033–1047. doi:10.1016/j.jfoodeng.2006.03.032

Li, J.-M., & Nie, S.-P. (2016). The functional and nutritional aspects of hydrocolloids in foods. *Food Hydrocolloids 53*, 46-61. doi:10.1016/j.foodhyd.2015.01.035

Meilgaard, M., Civille, G. V., & Carr, B. T. (1999). Affective Tests: Consumer Tests and In-House Panel Acceptance Tests. Em M. Meilgaard, G. V. Civille, & B. T. Carr, *Sensory Evaluation Techniques* (3rd ed.). CRC Press.

Meyers, M. A., & Grazela, A. (2011). Funtionality of hydrocolloids in batter coating systems. Em K. Kulp, R. Loewe, K. Lorenz, & J. Gelroth (Edits.), *Batters and Breadings in Food Processing* (2nd ed., pp. 117-138). AACC International, Inc.

Piqueras-Fiszman, B., Varela, P., & Fiszman, S. (2013). How Does the Science of Physical and Sensory Properties Contribute to Gastronomy and Culinary Art? *Journal of Culinary Science & Technology, 11*, 96-109. doi:10.1080/15428052.2012.728983

Sanz, T., Salvador, A., & Fiszman, S. (2004a). Effect of concentration and temperature on proprieties of methylcellulose-added batters. Application to battered, fried seafood. *Food hydrocolloids, 18*, 127-131. doi:10.1016/S0268-005X(03)00050-X

Sanz, T., Salvador, A., & Fiszman, S. (2004b). Innovative method for preparing a frozen, battered food without a prefrying step. *Food Hydrocolloids, 18*, 227-231. doi:10.1016/S0268-005X(03)00067-5

Singthong, J., & Thongkaew, C. (2009). Using hydrocolloids to decrease absorption in banana chips. *LWT - Food Science and Tecnology, 42*, 1199-1203. doi:10.1016/j.lwt.2009.02.014

Stone, H., & Sidel, J. L. (2004). *Sensory Evaluation Practices.* Elsevier Academic Press.

Varela, P., & Fiszman, S. (2011). Hydrocolloids in fried foods. A review. *Food hydrocolloids, 25*, 1801-1812. doi:10.1016/j.foodhyd.2011.01.016

Varela, P., Salvador, A., & Fiszman, S. M. (2008). Methodological developments in crispness assessment: Effects of cooking method on the crispness of crusted foods. *LWT - Food Science and Technology, 41*, 1252-1259. doi:10.1016/j.lwt.2007.08.008

World Health Organization. (2015). *Fact Sheet N.º 394: Healthy diet.* Geneve. Obtido de http://www.who.int

Xue, J., & Ngadi, M. (2007). Thermal properties of batter sytems formulated by combinations of different flours. *LWT - Food Science and Technology, 40*, 1459-1465. doi:10.1016/j.lwt.2006.02.007

Yilmaz, V. M., Sufer, O., & Seher, K. (2017). Effects of temperature and hydrocolloids on the rheological characteristics of coating batters. *Food Measure, 11*, 1159-1166.

CIÊNCIAS AGRÁRIAS, ALIMENTARES E VETERINÁRIAS
AGRICULTURAL SCIENCES, FOOD AND VETERINARY
CIENCIAS AGRÍCOLAS, ALIMENTOS Y VETERINARIA

millenium 79

Millenium, 2(8), 79-87.

en

CARATERIZAÇÃO QUÍMICA, PROPRIEDADES ANTIOXIDANTES E PERFIL FENÓLICO DE CASCA DE PINHEIRO (Pinus pinaster Aiton subsp. atlantica)

CHEMICAL CHARACTERIZATION OF PINE BARK (Pinus pinaster Aiton subsp. atlantica), ANTIOXIDANT PROPERTIES AND PHENOLIC PROFILE OF ITS EXTRACTS

CARACTERIZACIÓN QUÍMICA, PROPIEDADES ANTIOXIDANTES Y PERFIL FENÓLICO DE LA CORTEZA DE PINO (Pinus pinaster Aiton subsp. atlantica)

Catarina da Silva Vieito[1]
Preciosa Pires[2]
Élia Fernandes[2]
Manuela Vaz Velho[2]

1 Polytechnic Institute of Viana do Castelo, Viana do Castelo, Portugal
[2] Polytechnic Institute of Viana do Castelo, School of Technology and Management, Viana do Castelo, Portugal

Catarina da Silva Vieito - cvieito@ipvc.pt | Preciosa Pires - ppires@estg.ipvc.pt | Élia Fernandes - eliaf@estg.ipvc.pt | Manuela Vaz Velho - mvazvelho@estg.ipvc.pt

Corresponding Author
Manuela Vaz Velho
Instituto Politécnico de Viana do Castelo
Rua Escola Industrial e Comercial de Nun'Álvares, n.º 34
4900-347 Viana do Castelo
mvazvelho@estg.ipvc.pt

RECEIVED: 29th May 2018
ACCEPTED: 04th January 2019

Vieito, C. S., Pires, P., Fernandes, E. & Velho, M. V. (2019). Chemical characterization of pine bark (Pinus pinaster Aiton subsp. atlantica), antioxidant properties and phenolic profile of its extracts. *Millenium*, 2(8), 79-87.
DOI: https://doi.org/10.29352/mill0208.07.00197

RESUMO

Introdução: A casca de pinheiro é um resíduo agroindustrial proveniente da indústria madeireira e representa uma fonte de compostos fenólicos. Estes compostos têm várias propriedades benéficas sendo elas antioxidantes, antimicrobianas, anti-inflamatórias, cardiovasculares, entre outras.
Objetivos: O objetivo deste trabalho foi estudar a composição química da casca de *Pinus pinaster* Aiton subsp. *atlantica* e o perfil fenólico dos seus extratos aquosos, etanólicos e hidroetanólicos.
Métodos: Analisou-se o teor de humidade, cinzas, proteínas, gordura total e hidratos de carbono. A casca foi extraída com água, etanol ou uma mistura de ambos num aparelho de Soxhlet e determinou-se o rendimento das extrações, o teor de compostos fenólicos totais (CFT), atividade antioxidante e o perfil fenólico por RP HPLC UV nos extratos com o CFT mais elevado.
Resultados: Os resultados obtidos para a composição química foram: 63,43 de hidratos de carbono, 2,81 de gordura total, 1,60 de proteínas e 1,75 de cinzas, calculados em % m/m de casca seca. O rendimento das extrações foi superior para os solventes etanol e mistura hidroetanólica (17,08 e 17,55% m/m de casca seca, respetivamente). O CFT e a atividade antioxidante foram superiores no extrato hidroetanólico (73,48 mg EAG/g e 108,74 mg EAA/g de casca seca, respetivamente). Na análise do perfil fenólico do extrato hidroetanólico identificou-se o ácido gálico, taxifolin, ácido ferrúlico e quercetina a 280 nm e a catequina no extrato etanólico a 320 nm.
Conclusões: A casca de *P. pinaster* Aiton subsp. *atlantica* é maioritariamente constituída por hidratos de carbono e é rica em extrativos hidroetanólicos e etanólicos, tendo estes elevada atividade antioxidante. O extrato etanólico apresenta concentração de catequina mais elevada comparativamente ao extrato hidroetanólico.

Palavras-chave: casca de pinheiro; propriedades antioxidantes; compostos fenólicos; RP-HPLC-UV.

ABSTRACT

Introduction: Pine bark is an agroindustrial residue from the timber industry and represents a source of phenolic compounds. These compounds have several beneficial properties being antioxidants, antimicrobial, anti-inflammatory, cardiovascular, among others.
Objetives: The aim of this work was to study the chemical composition of the bark from *Pinus pinaster* Aiton subsp. *atlantica* and the phenolic profile of its aqueous, ethanolic and hydroethanolic extracts.
Methods: The moisture content, ash, protein, crude fat and carbohydrates were analysed. The bark was extracted with water, ethanol or a mixture of both in a Soxhlet apparatus and the extraction yield, total phenolic content (TPC), antioxidant activity and phenolic profile by RP-HPLC-UV, in the extracts with higher TPC, were determined.
Results: The results obtained for chemical composition were: 63.43 of carbohydrates, 2.81 of crude fat, 1.60 of proteins and 1.75 of ash, calculated in % w/w of dry bark. The extraction yield was greater for the ethanolic and the hydroethanolic extracts (17.08 and 17.55% w/w dry bark, respectively). The TPC and antioxidant activity were higher in the hydroethanolic extract (73.48 mg GAE/g and 108.74 mg AAE/g dry bark, respectively). Regarding the phenolic profile of the hydroethanolic extract, gallic acid, taxifolin, ferulic acid and quercetin were identified at 280 nm, and catechin was identified in the ethanolic extract at 320 nm.
Conclusion: *P. pinaster* Aiton subsp. *atlantica* bark is mainly constituted by carbohydrates and it is rich in hydroethanolic and ethanolic extractives, being that these have high antioxidant activity. The ethanolic extract presents higher catechin amount when compared to the hydroethanolic extract.

Keywords: pine bark; antioxidant properties; phenolic compounds; RP-HPLC-UV.

RESUMEN

Introducción: La corteza de pino es un residuo agroindustrial proveniente de la industria maderera y representa una fuente de compuestos fenólicos. Estos compuestos tienen varias propiedades benéficas siendo ellas antioxidantes, antimicrobianas, antiinflamatorias, cardiovasculares, entre otras.
Objetivos: El objetivo de este trabajo fue estudiar la composición química de la corteza de *Pinus pinaster* Aiton subsp. *atlantica* y el perfil fenólico de sus extractos acuosos, etanólicos e hidroetanólicos.
Métodos: Se analizó el contenido de humedad, cenizas, proteínas, grasa total y carbohidratos. La corteza se extrajo con agua, etanol o una mezcla de ambos en un aparato de Soxhlet y se determinaron el rendimiento de la extracción, el contenido de compuestos fenólicos totales (CFT), la actividad antioxidante y el perfil fenólico mediante RP-HPLC-UV, en los extractos con CFT más alto.
Resultados: Los resultados obtenidos para la composición química fueron: 63,43 de carbohidratos, 2,81 de grasa total, 1,60 de proteínas y 1,75 de ceniza, calculados en % m/m de corteza seca. El rendimiento de extracción fue mayor para los extractos etanólicos e hidroetanólicos (17,08 y 17,55% m/m de corteza seca, respectivamente). El CFT y la actividad antioxidante fueron

mayores en el extracto hidroetanólico (73,48 mg de EAG/g y 108,74 mg de EAA/g de corteza seca, respectivamente). Con respecto al perfil fenólico del extracto hidroetanólico, se identificaron ácido gálico, taxifolin, ácido ferúlico y quercetina a 280 nm, y se identificó la catequina en el extracto etanólico a 320 nm.
Conclusións: La corteza de *P. pinaster* Aiton subsp. *atlantica* está constituida principalmente por carbohidratos y es rica en extractos hidroetanólicos y etanólicos, ya que estos tienen una alta actividad antioxidante. El extracto etanólico presenta una mayor cantidad de catequina en comparación con el extracto hidroetanólico.

Palabras Clave: corteza de pino; propiedades antioxidantes; compuestos fenólicos; RP-HPLC-UV.

INTRODUCTION

As a consequence of the growing world population, natural resources availability is of current concern. In this sense, the use of materials, such as biomass residues from forests and agriculture, formerly considered waste, appears as a new world tendency. The Portuguese pine sector represents an important component of the total forest economic value (around 17%), being the third most important species after eucalyptus and cork oak (Seabra, Dias, Braga, & de Sousa, 2012). Pine bark is an abundant residue of the wood industry, since it represents 10–20% of the pine tree trunk (Braga et al., 2008). Currently, more than half of the bark is incinerated or landfilled and the remainder is mainly used as a cheap source of energy in saw/pulp mills being that both destinations can lead to environmental problems (Jablonsky et al., 2017). It was observed that bark contains a large fraction of extractives, with some important phytochemical constituents, and lignin, which can be utilized as a renewable source following the worldwide tendency of recovering, recycling and upgrading wastes (Braga et al., 2008). Also, it has low price and long-term stability that together make the usage of this residue highly attractive (Seabra et al., 2012). *P. pinaster* bark extracts have been reported to have several bioactivities including antioxidants, cardiovascular benefits, and anti-diabetic effects (Aspé & Fernandez, 2011; Chupin et al., 2015). The most prominent feature of *P. pinaster* is that it can grow on poor soil that provides minimal nourishment (Tümen, Akkol, Taştan, Süntar, & Kurtca, 2018). Due to large availability of pine bark on a global scale, there is an increasing interest in its use (Ronda, Della Zassa, Biasin, Martin-Lara, & Canu, 2017).
A lot of attention has been recently focused on the addition of polyphenols to foods and biological systems, due to their well-known abilities to scavenge free radicals, i.e. antioxidant power (Pinelo, Rubilar, Sineiro, & Núñez, 2004). They have received considerable attention in the fields of nutrition, health, and medicine owing to their physiological and biological activities, namely antibacterial, antiviral, anticarcinogenic, anti-inflammatory and cardiovascular system diseases' prevention (Seabra et al., 2012). The objective of extracting phenolic compounds from their plant sources is to release these compounds from the vacuolar structures where they are found, either by rupturing plant tissue or by a diffusion process. Usually, a high extraction yield is required for an efficient process, although it will not necessarily ensure a high concentration of bioactive components. Since some of these are very sensitive to oxygen and heat, more care should be taken to prevent their oxidation and thermal degradation. Therefore, the extraction yield and the bioactive components' characteristics should also be considered when an extraction method is selected (Aspé & Fernandez, 2011). Also, when the main goal is to apply these bioactive components in foods or nutraceutical products, the extraction solvent must be suitable for human consumption.
Therefore, the aim of the present study was to assess the chemical composition of bark from *Pinus pinaster* Aiton subsp. *atlantica* and evaluate the antioxidant activity of the aqueous, hydroethanolic and ethanolic extracts. Furthermore, its chromatographic profile by reverse phase high-performance liquid chromatography (RP-HPLC) was studied.

1. METHODS

1.1 Sample preparation

Pine bark (*P. pinaster* Aiton subsp. *atlantica*) was collected in Minho region, Northwest of Portugal, from trees aged 15 years. The inner bark was separated from the outer bark and the latter cut into pieces was oven dried to reach equilibrium humidity at 40 °C for 72 hours. The dried outer bark was ground by using a mixer (Termomix TM31, Vorwerk, Germany) for 20 s and sieved at an amplitude of 0.2 for 1 min to select the particles with a diameter between 200 and 850 µm. All analyses and extractions, except for moisture, were performed on outer dried pine bark. The inner bark was not analysed until the date of this article.

1.2 Reagents

Ethanol 96% was purchased from Aga (Prior Velho, Portugal). 2,2-Diphenyl-1-picrylhydrazyl (DPPH) and gallic acid monohydrate were purchased from Sigma-Aldrich (Steinheim, Germany). Folin-Ciocalteu reagent, ascorbic, boric and phosphoric acid were purchased from Merck (Darmstact, Germany). Sodium carbonate anhydrous, potassium sulfate, Kjeldahl catalyst, hydrogen peroxide and sodium hydroxide were purchased from Panreac (Barcelona, Spain). Methanol was purchased from Jt Baker

Vieito, C. S., Pires, P., Fernandes, E. & Velho, M. V. (2019). Chemical characterization of pine bark (Pinus pinaster Aiton subsp. atlantica), antioxidant properties and phenolic profile of its extracts. *Millenium*, 2(8), 79-87.
DOI: https://doi.org/10.29352/mill0208.07.00197

(Deventer, Holland). Petroleum ether and sulfuric acid were purchased from Fisher Scientific (Loughborough, UK). The reagents were of analytical grade, except the ones used for HPLC analysis that were HPLC grade.

1.3 Chemical composition

Pine bark was analysed for moisture, ash, proteins, fat and carbohydrates contents using the AOAC procedures (AOAC, 1995). The moisture was determined by drying in an oven at 103 °C until constant weight (AOAC 930.04); the ash content was determined by incineration at 550 °C (AOAC 930.05); the crude protein content (N x 6.25) of the samples was estimated by Kjeldahl method (AOAC 978.04); the crude fat was determined by extracting a known weight of ground sample with petroleum ether, using a Soxhlet apparatus (AOAC 920.39) and the total carbohydrates were determined by the 3,5-dinitrosalicylic acid reaction with reducing sugars present in the sample after hydrolysis with hydrochloric acid and neutralization (Miller, 1959).

1.4 Extraction of pine bark and evaluation of its antioxidant properties

Different solvent types were tested with Soxhlet extraction. A total of 12.5 g of ground pine bark was put into a cartridge inside a Soxhlet apparatus. Then, 220 mL of solvent (water, hydroethanolic mixture (1+1) or ethanol) was added to the flask and refluxed over four hours as the minimal indicating time for the official AOAC method for crude fat. The extract was collected and completed to 250 mL with the respective solvent and named as PW, PWE and PE, corresponding to the aqueous, hydroethanolic and ethanolic extracts, respectively.

1.4.1 Extraction yield

Extraction yield (% w/w) is a measure of the solvent efficiency to extract specific components from the original material, defined as the amount of solid extract recovered in dry mass compared with the initial amount of dry bark. The extraction yield was calculated measuring an exact sample volume (V_1=25 mL) from the total extract (V_2=250 mL) and the volume was reduced at 35 °C until it was obtained a dry solid residue. Finally, it was dried at 103 °C until constant weight.

$$Extraction\ yield\ (\%\ w/w) = \frac{w_2 - w_1}{w_{sample} \times \left(\frac{V_1}{V_2}\right)} \times 100$$

Where w_1 is the empty recipient weight, w_2 is the recipient weight plus the dried extract weight and w_{sample} is the sample amount weighed to perform the extraction.

1.4.2 Total phenolic content

Total phenolic content was estimated by Folin-Ciocalteu colorimetric assay according to the procedure previously described by Lafka, Sinanoglou, and Lazos (2007) based on Gutfinger (1981) and the results were expressed as mg of gallic acid equivalents (GAE) per g of dry sample.

1.4.3 Antioxidant activity

The antioxidant activity in the extracts was determined by the DPPH (2,2-diphenyl-1-picrylhydrazyl) radical scavenging method used by Deng, Penner, and Zhao (2011) based on Brand-Williams, Cuvelier, and Berset (1995). A volume of 2 mL of diluted extract stock solution (in methanol) was mixed with 6 mL of DPPH in methanol and allowed to stand at room temperature, in the dark, for 30 minutes prior to measuring the solution absorbance at 517 nm. The control was a DPPH solution containing absolute methanol instead of the sample. The antioxidant activity was based on the measurement of the reducing ability of pine extracts towards the radical DPPH. The results were obtained as mg of ascorbic acid equivalent (AAE) per g of dry bark. The standard curve was prepared with ascorbic acid at 0, 1, 2.5, 5, 7.5, 10, 20, 30, 35 and 40 mg/L.

1.5 Polyphenols identification (RP-HPLC-UV analysis)

Sample preparation

A tenfold dilution (1/10) in methanol of the extract was performed. The solution was homogenized and filtered with disposable 0.2 µm syringe filters (Whatman, UK) prior to injection.

LC conditions

Qualitative identification of polyphenols in pine bark extracts was carried out using an HPLC method developed in-house. The system used a reverse phase column, Betasil C18 (4.6 x 250 mm, 5 µm particle size, Thermo Scientific, USA). The mobile phase comprised 1% phosphoric acid in water, methanol and water. Gradient elution was as follows: 0-2 min, 80/0/20; 2-5 min, 65/15/20; 5-10 min, 50/30/20; 10-15 min, 45/35/20; 15-25 min, 30/50/20; 25-30 min, 20/60/20; 30-35 min, 0/80/20; 35-45 min, 0/90/10; 45-65 min, 0/100/0; 65-70 min 30/50/20. The flow rate was 1.0 mL/min. The wavelength for UV detection was set at 280 and 320 nm.

The experiments were performed on a Hewlett Packard Series 1100 HPLC system (Agilent Technologies, Waldbronn, Germany), equipped with a G1322A degasser, G1311A quaternary pump, G1314A UV-Vis detector and a G1328A manual injector. The software used was HP ChemSation for LC Rev. A.06.03 [509].

1.6 Statistical analysis

All the assays were carried out in triplicate. The results were expressed as mean values and standard deviation (SD). A correlation between the total phenolic content and antioxidant activity values was calculated and it was obtained a correlation coefficient (R^2). The results were analysed using one-way analysis of variance (ANOVA) followed by Tukey's HSD test (p<0.05). This treatment was carried out using IBM SPSS Statistics for Windows, version 25.0 (IBM Corp, New York, USA).

2. RESULTS AND DISCUSSION

2.1 Chemical composition

There are little recent papers about the chemical composition of pine bark, namely the maritime pine grown in Portugal. Also, the composition of bark depends on the age of the tree, the location and the growing conditions, among other factors, as observed by Vázquez, Antorrena, and Parajó (1987). In this work the two parts of pine bark (outer and inner) were divided to be studied in separate. However, this work only presents the results for the outer pine bark fraction (Table 1). The moisture content of the pine bark studied was 12.82% w/w. The dry bark composition was: carbohydrates 63.43% w/w, fat 2.81% w/w, proteins 1.60% w/w and ash 1.75 % w/w. Vázquez et al. (1987) extracted dry *P. pinaster* bark with organic apolar solvents obtaining yields of 0.9% w/w with ether and 2.5% w/w with hexane and benzene extraction, which is similar to the fat content determined in this work (2.81% w/w). Other authors (Fradinho et al., 2002; Vázquez et al., 1987) studied dry maritime pine bark and obtained lower values for ash, namely 0.5 and 0.8% w/w, respectively. In this work the remaining value calculated by difference (30.41% w/w; Table 1) includes other components such as cellulose, lignin and suberin, among others.

Table 1 – Chemical composition values of dry pine bark from *P. pinaster* Aiton subsp. *atlantica*.

Parameter	Amount (% w/w)
Ash	1.75 ± 0.03
Proteins	1.60 ± 0.04
Fat	2.81 ± 0.002
Carbohydrates	63.43 ± 3.95
Other components	30.41

Pine bark moisture = 12.82 ± 0.01 % w/w.

2.2 Extraction of pine bark and evaluation of its antioxidant properties

In papers published to date different solvent systems and extraction techniques have been used for the extraction of polyphenols from pine bark. The yield, total phenolic content and antioxidant activity of the extracts is highly dependent on these factors. Water, ethanol, methanol, aqueous alkaline solutions, acetone, dichloromethane, are some of the solvents commonly used to extract phenolic compounds from pine bark (Aspé & Fernandez, 2011; Ćurković-Perica, Hrenović, Kugler, Goić-Barišić, & Tkalec, 2015; Fradinho et al., 2002).

In Table 2 are presented results of extraction yield, total phenolic content and antioxidant properties of *P. pinaster* Aiton subsp. *atlantica* bark extracts. PWE and PE extracts did not show significant differences regarding extraction yield, with 17.55 and 17.08% w/w dry bark being obtained, respectively (p<0.05). Fradinho et al. (2002) have reported a similar value, namely 13.5% w/w dry bark for the successive Soxhlet extractions with ethanol and water in *P. pinaster* bark grown in Portugal. However, the PWE extract showed a higher total phenolic content (73.48±1.84 mg GAE/g dry bark) and therefore, higher antioxidant activity (108.74±2.02 mg AAE/g dry bark, Table 2, R^2=0.983, p<0.05). Water extracts presented the lowest values for all the parameters analysed. Pinelo et al. (2004) observed the same behaviour when extracting *P. pinaster* sawdust with water, denoting that water was not a good solvent for extracting phenolics. However, mixing water and ethanol can improve the rate of extraction by causing the raw material to bloat, enabling the solvent to easily enter the solid particles (Gertenbach, 2002).

Pine bark extracts showed an expressive antioxidant capacity when compared with red fruits. In a recent study (Seraglio et al., 2018), it was found that in *Myrtaceae* fruits, the highest level DPPH value is 50.66 mg AAE/g dry fruit, while in this study the hydroethanolic extract is 108.74 mg AAE/g dry pine bark.

Vieito, C. S., Pires, P., Fernandes, E. & Velho, M. V. (2019). Chemical characterization of pine bark (Pinus pinaster Aiton subsp. atlantica), antioxidant properties and phenolic profile of its extracts. *Millenium*, 2(8), 79-87.
DOI: https://doi.org/10.29352/mill0208.07.00197

Table 2 – Extraction yield, total phenolic content and antioxidant activity of *P. pinaster* Aiton subsp. *atlantica* bark extracts.

Sample	Extraction yield (% w/w dry bark)	Total phenolic content (mg GAE/g dry bark)	Antioxidant activity (mg AAE/g dry bark)
PW	$7.84^{a} \pm 0.56$	$50.09^{a} \pm 4.70$	$82.24^{a} \pm 4.65$
PWE	$17.55^{b} \pm 0.16$	$73.48^{b} \pm 1.84$	$108.74^{b} \pm 2.02$
PE	$17.08^{b} \pm 0.23$	$63.38^{c} \pm 1.26$	$95.58^{c} \pm 0.55$

Note: Means (n=3) with different uppercase letters in the same column are significantly different ($p<0.05$). GAE – Gallic acid equivalent, AAE – ascorbic acid equivalent.

2.3 Polyphenols identification (RP-HPLC-UV analysis)

Polyphenols are among the most widespread class of metabolites in nature, and their distribution is almost ubiquitous (Pereira, Valentão, Pereira, & Andrade, 2009). Biogenetically, phenolic compounds proceed of two metabolic pathways: the shikimic acid pathway where, mainly, phenylpropanoids are formed and the acetic acid pathway in which the main products are the simple phenols (Belščak-Cvitanović, Durgo, Huđek, Bačun-Družina, & Komes, 2018). Flavonoids and stilbenes are the majority of natural-occurring phenolics (Pereira et al., 2009).

Chromatography is needed to obtain more detailed information on polyphenol profiles than is provided by spectrophotometric methods (Sáyago-Ayerdi, Mercado-Mercado, Ramos-Romero, Torres, & Pérez-Jiménez, 2016).

Considering the results obtained in the 3.2 section, as the hydroethanolic and ethanolic extracts showed better antioxidant properties, they were studied by RP-HPLC-UV. Figure 1 shows the base-peak chromatogram of phytochemical compounds in pine bark hydroethanolic extract obtained by reverse phase with the corresponding retention times. Tentative assignment of these phytochemical compounds was obtained by comparing with the retention time of pure standards (Table 3). For each of the five phytochemicals presented in Table 3, a second chromatogram was obtained after addition of a known amount to the original pine bark extract. In the chromatogram with added standard it could be observed which peak increased its intensity. This methodology helps to clarify the assignment as for example, with compounds assigned with similar RT, taxifolin (22.9 min) and ferulic acid (23.6 min).

The HPLC-UV phytochemical profile recorded at 280 nm includes, by elution order: gallic acid, taxifolin, ferulic acid and quercetin. Catechin was detected at 320 nm and exhibits an intense signal in the ethanolic extract (Figure 2), but not in the hydroethanolic one. Braga et al. (2008) also observed an higher content of catechin in the ethanolic extract, however at 280 nm.

Although the pine bark water or ethanolic extracts have not been completely elucidated, the main constituents described in the literature for a commercialized standardized extract, Pycnogenol, indicates that it contains catechin, epicatechin and taxifolin (Packer, Rimbach, & Virgili, 1999).

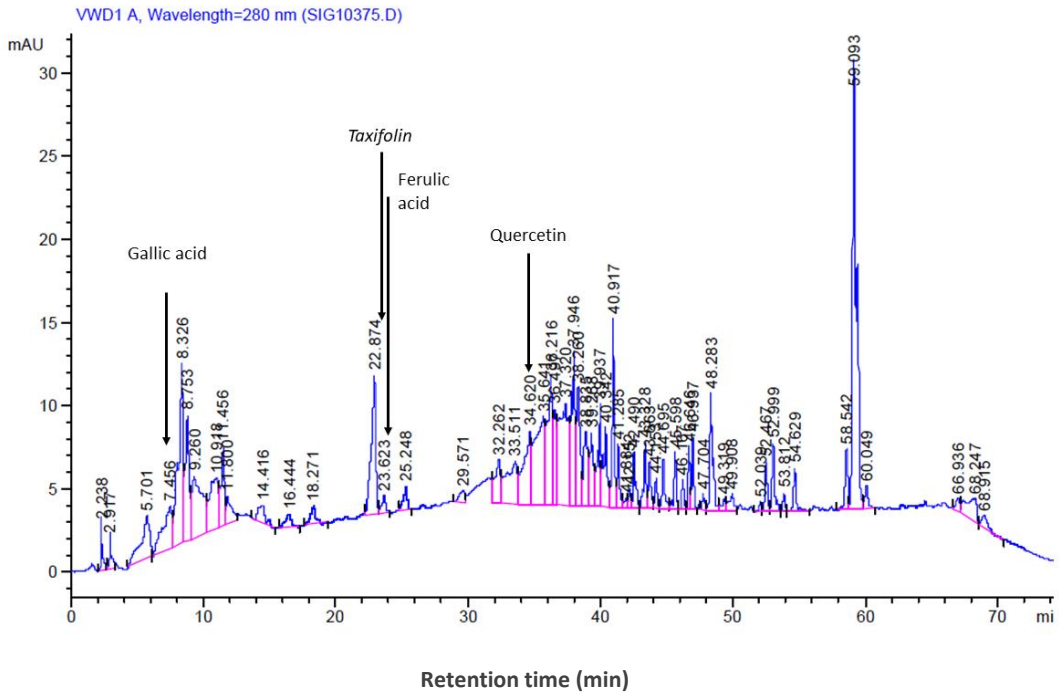
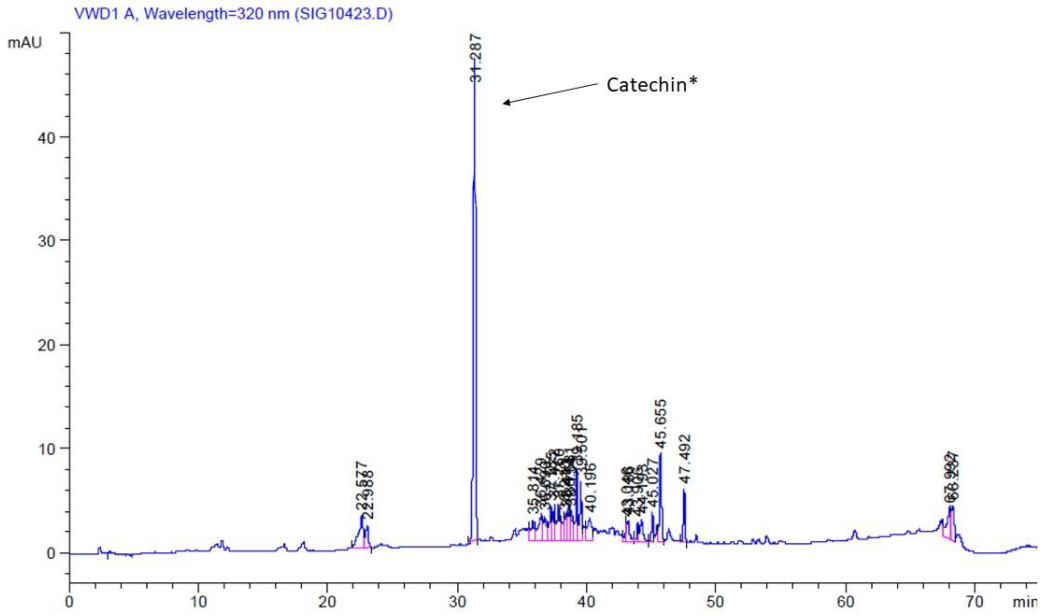


Figure 1 – Chromatogram of the pine bark hydroethanolic extract recorded at 280 nm, tenfold dilution in methanol.



Retention time (min)

Figure 2 – Chromatogram of the pine bark ethanolic extract recorded at 320 nm, tenfold dilution in methanol.

Table 3 – Tentative assignment of phytochemical compounds detected by HPLC-UV in pine bark hydroethanolic extracts.

Tentative assignment	Retention time (min)	Wavelength (nm)
Gallic acid	7.5	280
Taxifolin	22.9	280
Ferulic acid	23.6	280
Quercetin	34.6	280
Catechin	31.3	320

CONCLUSIONS

The bark of *P. pinaster* Aiton subsp. *atlantica* is composed of carbohydrates (63.43% w/w), fat (2.81% w/w), proteins (1.60% w/w), ash (1.75% w/w) and other components (30.41% w/w). Pine bark is rich in hydroethanol and ethanol extractable phytochemicals, namely 73.48 and 63.38 mg GAE/g dry bark, respectively. The pine bark extracts have high levels of antioxidant properties (82-109 mg AAE/g dry pine bark), higher than some red fruits known to be rich in antioxidant properties. The ethanolic extract presents higher concentration of catechin compared to the hydroethanolic extract. Further studies should be developed to fully confirm the chemical composition of *P. pinaster* Aiton subsp. *atlantica* bark extracts.

ACKNOWLEDGEMENTS

The accomplishment of this work was possible due to the SUDOE INTERREG IVB REDVALUE SOE1/P1/E0123 research grant.

REFERENCES

AOAC. (1995). Official Methods of Analysis of AOAC INTERNATIONAL (16th ed., Vol. II): The Scientific Association Dedicated to Analytical Excellence.

Vieito, C. S., Pires, P., Fernandes, E. & Velho, M. V. (2019). Chemical characterization of pine bark (Pinus pinaster Aiton subsp. atlantica), antioxidant properties and phenolic profile of its extracts. *Millenium*, 2(8), 79-87.
DOI: https://doi.org/10.29352/mill0208.07.00197

Aspé, E., & Fernandez, K. (2011). The effect of different extraction techniques on extraction yield, total phenolic, and anti-radical capacity of extracts from Pinus radiata Bark. Industrial Crops and Products, 34(1), 838-844. doi: 10.1016/j.indcrop.2011.02.002

Belščak-Cvitanović, A., Durgo, K., Huđek, A., Bačun-Družina, V., & Komes, D. (2018). Overview of polyphenols and their properties. In C. M. Galanakis (Ed.), Polyphenols: Properties, Recovery, and Applications (pp. 3-44). Cambridge, United Kingdom: Woodhead Publishing.

Braga, M. E. M., Santos, R. M. S., Seabra, I. J., Facanali, R., Marques, M. O. M., & de Sousa, H. C. (2008). Fractioned SFE of antioxidants from maritime pine bark. The Journal of Supercritical Fluids, 47(1), 37-48. doi: 10.1016/j.supflu.2008.05.005

Brand-Williams, W., Cuvelier, M. E., & Berset, C. (1995). Use of a free radical method to evaluate antioxidant activity. LWT - Food Science and Technology, 28(1), 25-30. doi: https://doi.org/10.1016/S0023-6438(95)80008-5

Chupin, L., Maunu, S. L., Reynaud, S., Pizzi, A., Charrier, B., & Charrier-El Bouhtoury, F. (2015). Microwave assisted extraction of maritime pine (Pinus pinaster) bark: Impact of particle size and characterization. Industrial Crops and Products, 65, 142-149. doi: 10.1016/j.indcrop.2014.11.052

Ćurković-Perica, M., Hrenović, J., Kugler, N., Goić-Barišić, I., & Tkalec, M. (2015). Antibacterial Activity of Pinus pinaster Bark Extract and its Components Against Multidrug-resistant Clinical Isolates of Acinetobacter baumannii. Croatica Chemica Acta, 88(2), 133-137. doi: 10.5562/cca2548

Deng, Q., Penner, M. H., & Zhao, Y. Y. (2011). Chemical composition of dietary fiber and polyphenols of five different varieties of wine grape pomace skins. Food Research International, 44(9), 2712-2720. doi: 10.1016/j.foodres.2011.05.026

Fradinho, D. M., Neto, C. P., Evtuguin, D., Jorge, F. C., Irle, M. A., Gil, M. H., & Pedrosa de Jesus, J. (2002). Chemical characterisation of bark and of alkaline bark extracts from maritime pine grown in Portugal. Industrial Crops and Products, 16(1), 23-32. doi: https://doi.org/10.1016/S0926-6690(02)00004-3

Gertenbach, D. D. (2002). Solid-liquid extraction technologies for manufacturing nutraceuticals. In J. Shi, G. Mazza, & M. L. Maguer (Eds.), Functional Foods: Biochemical and Processing Aspects. Boca Raton, Florida, USA: CRC Press.

Gutfinger, T. (1981). Polyphenols in olive oils. Journal of the American Oil Chemists Society, 58(11), 966-968. doi: 10.1007/bf02659771

Jablonsky, M., Nosalova, J., Sladkova, A., Haz, A., Kreps, F., Valka, J., . . . Surina, I. (2017). Valorisation of softwood bark through extraction of utilizable chemicals. A review. Biotechnology Advances, 35(6), 726-750. doi: https://doi.org/10.1016/j.biotechadv.2017.07.007

Lafka, T. I., Sinanoglou, V., & Lazos, E. S. (2007). On the extraction and antioxidant activity of phenolic compounds from winery wastes. Food Chemistry, 104(3), 1206-1214. doi: 10.1016/j.foodchem.2007.01.068

Miller, G. L. (1959). Use of Dinitrosalicylic Acid Reagent for Determination of Reducing Sugar. Analytical Chemistry, 31(3), 426-428. doi: 10.1021/ac60147a030

Packer, L., Rimbach, G., & Virgili, F. (1999). Antioxidant activity and biologic properties of a procyanidin-rich extract from pine (Pinus maritima) bark, pycnogenol. Free Radical Biology and Medicine, 27(5-6), 704-724. doi: 10.1016/s0891-5849(99)00090-8

Pereira, D., Valentão, P., Pereira, J., & Andrade, P. (2009). Phenolics: From Chemistry to Biology. Molecules, 14(6), 2202-2211. doi: 10.3390/molecules14062202

Pinelo, M., Rubilar, M., Sineiro, J., & Núñez, M. J. (2004). Extraction of antioxidant phenolics from almond hulls (Prunus amygdalus) and pine sawdust (Pinus pinaster). Food Chemistry, 85(2), 267-273. doi: 10.1016/j.foodchem.2003.06.020

Ronda, A., Della Zassa, M., Biasin, A., Martin-Lara, M. A., & Canu, P. (2017). Experimental investigation on the smouldering of pine bark. Fuel, 193, 81-94. doi: https://doi.org/10.1016/j.fuel.2016.12.028

Sáyago-Ayerdi, S. G., Mercado-Mercado, G., Ramos-Romero, S., Torres, J. L., & Pérez-Jiménez, J. (2016). Analysis and Characterization of Polyphenol Extracts. In J. Cuevas Valenzuela, J. R. Vergara-Salinas, & J. R. Perez-Correa (Eds.), Advances in technologies for producing food-relevant polyphenols (pp. 27). Boca Raton, Florida: CRC Press.

Seabra, I. J., Dias, A. M. A., Braga, M. E. M., & de Sousa, H. C. (2012). High pressure solvent extraction of maritime pine bark: Study of fractionation, solvent flow rate and solvent composition. The Journal of Supercritical Fluids, 62, 135-148. doi: 10.1016/j.supflu.2011.10.016

Seraglio, S. K. T., Schulz, M., Nehring, P., Della Betta, F., Valese, A. C., Daguer, H., . . . Costa, A. C. O. (2018). Nutritional and bioactive potential of Myrtaceae fruits during ripening. Food Chemistry, 239, 649-656. doi: https://doi.org/10.1016/j.foodchem.2017.06.118

Tümen, İ., Akkol, E. K., Taştan, H., Süntar, I., & Kurtca, M. (2018). Research on the antioxidant, wound healing, and anti-inflammatory activities and the phytochemical composition of maritime pine (Pinus pinaster Ait). Journal of Ethnopharmacology, 211, 235-246. doi: https://doi.org/10.1016/j.jep.2017.09.009

Vázquez, G., Antorrena, G., & Parajó, J. C. (1987). Studies on the utilization of Pinus pinaster bark - Part 1: Chemical constituents. Wood Science and Technology, 21(1), 65-74. doi: 10.1007/bf00349718

CIÊNCIAS AGRÁRIAS, ALIMENTARES E VETERINÁRIAS
AGRICULTURAL SCIENCES, FOOD AND VETERINARY
CIENCIAS AGRÍCOLAS, ALIMENTOS Y VETERINARIA

millenium 89

Millenium, 2(8), 89-99.

en

INFLUÊNCIA DOS SOLVENTES DE EXTRAÇÃO NO CONTÉUDO EM COMPOSTOS BIOACTIVOS E ACTIVIDADE ANTIOXIDANTE DE FLORES DE AMORES-PERFEITOS

EXTRACTION SOLVENTS' INFLUENCE ON THE CONTENT OF BIOACTIVE COMPOUNDS AND ANTIOXIDANT ACTIVITY OF PANSIES

INFLUENCIA DE LOS SOLVENTES DE EXTRACCIÓN EN EL CONTENIDO DE COMPUESTOS BIOACTIVOS Y ACTIVIDAD ANTIOXIDANTE DE LAS FLORES DOS PENSAMIENTOS

Luana Fernandes[1, 2, 3]
José Alberto Pereira[4]
Jorge Saraiva[5]
Susana Casal[6]
Elsa Ramalhosa[7]

[1] Instituto Politécnico de Bragança, Escola Superior Agrária, Centro de Investigação de Montanha (CIMO), Bragança, Portugal.
[2] Universidade de Aveiro, Departamento de Química, Aveiro, Portugal.
[3] Universidade do Porto, Faculdade de Farmácia, Laboratório de Bromatologia e Hidrologia (LAQV@REQUIMTE) Porto, Portugal
[4] Instituto Politécnico de Bragança, Escola Superior Agrária, Bragança, Portugal
[5] Universidade de Aveiro, Departamento de Química, Aveiro, Portugal
[6] Universidade do Porto. Faculdade de Farmácia, Porto, Portugal
[7] Instituto Politécnico de Bragança, Escola Superior Agrária, Centro de Investigação de Montanha (CIMO), Bragança, Portugal

Luana Fernandes - luaopus@hotmail.com | José Alberto Pereira - jpereira@ipb.pt | Jorge Saraiva - jorgesaraiva@ua.pt | Susana Casal - sucasal@ff.up.pt | Elsa Ramalhosa - elsa@ipb.pt

Corresponding Author
Elsa Ramalhosa
Instituto Politécnico de Bragança
Campus de Santa Apolónia
5300-253 BRAGANÇA
elsa@ipb.pt

RECEIVED: 29th May 2018
ACCEPTED: 04th January 2019

Fernandes, L., Pereira, J. A., Saraiva, J., Casal, S. & Ramalhosa, E. (2019).
Extraction solvents' influence on the content of bioactive compounds and antioxidant activity of pansies. *Millenium*, 2(8), 89-99.
DOI: https://doi.org/10.29352/mill0208.08.00205

RESUMO

Introdução: Amores-perfeitos (*Viola×wittrockiana*) são uma fonte rica de antioxidantes naturais com efeitos benéficos para a saúde humana.
Objetivos: O objetivo do presente estudo foi investigar a influência dos solventes (água, metanol e água:acetona (6:4, v/v)) na extracção de compostos bioactivos e actividade antioxidante de extractos de amores-perfeitos.
Métodos: Os compostos bioactivos analisados foram os seguintes: flavonóides, taninos hidrolisáveis e antocianinas monoméricas, bem como, os fenóis totais através da avaliação da capacidade redutora total. A actividade antioxidante foi avaliada pelos métodos de sequestro do radical livre 2,2-difenil-1-picrilhidrazilo (DPPH) e poder redutor. Uma análise de componentes principais (PCA) foi realizada para diferenciar os extractos dos amores-perfeitos.
Resultados: Os solventes que produziram extractos com os maiores teores de taninos hidrolisáveis e capacidade redutora total foram os de metanol e água: acetona (6:4, v/v). Para extrair os maiores teores de antocianinas monoméricas deve-se usar metanol (5.93 mg Cy 3-glu/ g de massa seca), enquanto que para extrair flavonóides, a água:acetona (6:4, v/v) é preferível (115 mg QE/g de massa seca). A água mostrou ser o solvente menos eficaz, obtendo-se extractos com a menor actividade antioxidante. Para além disso, os extractos de metanol e água:acetona foram claramente separados dos aquosos através da análise por PCA.
Conclusões: Os resultados mostraram que a extracção de compostos bioactivos e a actividade antioxidante de flores de amores-perfeitos são influenciadas pelo solvente usado.

Palavras-chave: Amores-perfeitos; Solventes; Actividade antioxidante; Compostos bioactivos.

ABSTRACT

Introduction: Pansies (*Viola×wittrockiana*) are a rich source of natural antioxidants with beneficial effects on human health.
Objetives: The aim of our study was to investigate solvents' influence (water, methanol, water:acetone (6:4, v/v)) on the extraction of bioactive compounds and antioxidant activity of pansies extracts.
Methods: The bioactive compounds analyzed were the following: flavonoids, hydrolysable tannins and monomeric anthocyanins, as well as total phenols by the total reducing capacity assay (TRC). The antioxidant activity was evaluated by 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity and reducing power assays. A Principal Component Analysis (PCA) was performed to differentiate pansies extracts.
Results: The solvents that yielded extracts with the highest contents of hydrolysable tannins and TRC were methanol and water:acetone (6:4, v/v). To extract the highest contents of monomeric anthocyanins, methanol should be used (5.93 mg Cy 3-glu/g flower, d.w), while for flavonoids, water:acetone (6:4, v/v) was the preferred yielding an extract with 115 mg QE/g flower d.w. Water turned out to be the least effective solvent, giving extracts with the lowest antioxidant activity. In addition, methanol or water:acetone extracts were clearly distinguished from aqueous ones through a PCA analysis.
Conclusions: Our results show that the bioactive compounds and antioxidant activity of pansies' extracts are affected by the solvent used.

Keywords: Pansies; Solvents; Antioxidant activity; Bioactive compounds.

RESUMEN

Introducción: Los pensamientos (*Viola x wittrockiana*) son una rica fuente de antioxidantes naturales con efectos beneficiosos sobre la salud humana.
Objetivos: El objetivo del presente estudio fue evaluar la influencia de los solventes (agua, metanol y agua:acetona (6:4, v/v) en la extracción de compuestos bioactivos y actividad antioxidante de extractos de pensamientos.
Métodos: Los compuestos bioactivos analizados fueron flavonoides, taninos hidrolizados y antocianinas monoméricas. También se analizaron los fenoles totales por el ensayo de capacidad reductora total. La actividad antioxidante fue evaluada por los métodos: secuestro del radical libre 2,2-difenil-1-picrilhidrazilo (DPPH) y poder reductor. El análisis de componentes principales (PCA) fue realizado para diferenciar los extractos de los pensamientos.
Resultados: Los solventes que produjeron extractos con los mayores contenidos de taninos hidrolizados y capacidad reductora total fueron el metanol y agua:acetona (6:4, v/v). Los extractos con mayores contenidos de antocianinas monoméricas se obtuvieron utilizando metanol (5.93 mg Cy 3-glu/g peso seco). Para los flavonoides el agua: acetona (6:4, v / v) fue el solvente que hay dado los mejores valores de extracción (115 mg QE / g peso seco). El agua fue el solvente menos eficaz, obteniéndo se extractos con menor actividad antioxidante. Además, los extractos de metanol y agua:acetona se separaron claramente de los acuosos mediante el análisis por PCA.

Conclusións: Los resultados mostraron que la extracción de compuestos bioactivos y la actividad antioxidante de los pensamientos son influenciadas por el solvente utilizado.

Palabras Clave: Pensamientos; Solventes; Actividad antioxidante; Compuestos bioactivos

INTRODUCTION

Garden pansies (*Viola × wittrockiana Gams.*) are plants of complex hybrid origin, involving at least three species, namely *Viola tricolor*, *Viola altaica*, and *Viola lutea* (Vukics et al., 2008). They are popular ornamental plants, differing in size and color, depending on the variety (Weryszko-Chmielewska and Sulborska, 2012). These flowers are mentioned as edible, so they can be found on the market in special packages (Kelley et al. 2003), which are used for garnish, salads, soups, desserts and drinks.
Some studies have been performed in pansies, concerning their antioxidant activity and polyphenolic composition (Hase et al., 2005; Vukics et al., 2008; Gamsjaeger et al., 2011; Skowyra et al., 2014; González-Barrio et al., 2018). Nowadays, plant polyphenols (ex. flavonoids, tannins and anthocyanins) are increasing attention due to their antioxidant properties and documented effects in the prevention of various oxidative stress associated diseases, such as cancer, obesity and diabetes (Dai and Mumper, 2010). So, these active compounds from flowers can be used as drugs, as well as ingredients (to enhance flavor, color, etc.) in food and cosmetics (Jun, 2013).
The most common method to obtain these compounds from plants is solvent extraction. Polar solvents, such as methanol and ethanol, are frequently employed for the recovery of polyphenols from a plant matrix (Kuźma et al., 2014). In detail, reported solvents and extraction conditions used in pansies include: methanol (70%, v/v) in an ultrasonic bath at room temperature for 20 min (Vukics et al., 2008); methanol (100%, v/v) at 25 °C for 24 h (Rop et al., 2012); water (100%, v/v), ethanol (50%, v/v) and acidic ethanol (50%, v/v) at 4 °C for 24 h, under stirring (Skowyra et al., 2014); methanol containing 0.1% HCl at 4 °C for 24 h with gentle shaking (Li et al. 2014a); and methanol (80%, v/v) containing 1% HCl, at 4 °C for 12 h (Benvenuti et al., 2016). So, the aim of this work was to further analyze the effect of using solvents with a wide range of polarities on different bioactive compounds extraction and antioxidant activity of *Viola × wittrockiana*. The solvents studied were water, methanol, and water:acetone (6:4, v/v). The extracts obtained were compared with respect to their flavonoids, hydrolysable tannins and monomeric anthocyanins contents, as well as their total reducing capacity (TRC) and antioxidant activity evaluated by the reducing power and scavenging effect on 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical assays.

1. METHODS

1.1 Flower

Fresh pansies (*Viola × wittrockiana*) of different colors were collected from the greenhouse of School of Agriculture, Polytechnic Institute of Bragança, during the year 2015 (Figure 1). After harvest, flowers were transported immediately to the laboratory under refrigeration. On their arrival, flowers were immediately frozen and lyophilized (Scanvac, Coolsafe, Lynge, Denmark) for 2 days. Afterwards they were ground to a homogeneous powder, which was stored protected from light and moisture until further analyses.

Figure 1 – Pansies of different colors studied in the present work.

Fernandes, L., Pereira, J. A., Saraiva, J., Casal, S. & Ramalhosa, E. (2019).
Extraction solvents' influence on the content of bioactive compounds and antioxidant activity of pansies. *Millenium*, 2(8), 89-99.
DOI: https://doi.org/10.29352/mill0208.08.00205

1.2 Chemicals and reagents

Methanol and acetone were obtained from Fisher Scientific (Leicestershire, UK). Gallic acid and DPPH (2,2-diphenyl-1-picrylhydrazyl) were from Sigma–Aldrich (St. Louis, MO, USA), while Folin–Ciocalteu reagent and sodium carbonate were obtained from Panreac Quimica SA (Barcelona, Spain). All reagents were of analytical grade. Milli-Q system (Millipore Corp., Molsheim, France) ultra-pure water was used throughout this research.

1.3 Extraction conditions

The extraction with different solvents was based on the method described by Li et al. (2014b), with slight modifications. A 1:20 mass/volume ratio was used on all extractions. Dried powder (1 g) of a pansies' mixture was extracted with 20 mL of different solvents (methanol, water and water:acetone (6:4, v/v)) at 37 °C for 30 min under agitation (900 rpm) (IKA, RCT Model B, Staufen, Germany). The methanol and water:acetone extracts were filtered and concentrated in a rotary evaporator, in order to remove methanol and acetone, respectively. Then, all solutions were frozen and placed in the freeze-drier for 2 days to obtain the extracts. The extracts were weighted and dissolved in the same solvent used in the extractions to a concentration of 50 mg extract/mL and covered with aluminium foil under freezing until further analysis. All extractions were performed in triplicate.

1.4 Total flavonoids

Total flavonoids content was determined by the method described by Viuda-Martos et al. (2011), with slight modifications. To pansies extracts (1 mL) was added 0.3 mL $NaNO_2$ (5%, m/v) and, after 5 min, 0.3 mL $AlCl_3$ (10%, m/v) was mixed. After 6 minutes, 2 mL NaOH (1M) was added. The absorbance was read at 510 nm and flavonoids were quantified using a standard curve of quercetin (10-160 µg/mL). The results were expressed in mg of quercetin/g flower, dry weight (mg QE/g flower, dry weight (d.w.)). All measurements were performed in triplicate.

1.5 Hydrolysable tannins

The content of hydrolysable tannins was determined by the method described by Elfalleh et al. (2012). Flower extracts (1 mL), 5 mL 2.5% KIO_3 was added and stirred for 10 seconds. The absorbance was measured at 550 nm. The blank corresponded to the solvent used in each sample. Different concentrations of tannic acid (0.025 to 1.6 g/L) were used for calibration. Results were expressed in mg of tannic acid equivalent/g flower, dry weight (mg TAE/g flower, d.w.). All measurements were performed in triplicate.

1.6 Total monomeric anthocyanins

The total monomeric anthocyanins contents in the pansy extracts were estimated by the pH differential method, following the methodologies used by Bchir et al. (2012) and Rajasekar et al. (2012). The method consisted in using two buffer systems: potassium chloride buffer, pH 1.0 (0.025 M) and sodium acetate, pH 4.5 (0.4 M). The flower extracts (250 µL) were diluted with pH 1.0 and pH 4.5 buffers in 25 mL flasks and allowed to stand for 30 minutes at room temperature. Subsequently, the absorbance readings were made on a UV-Visible spectrophotometer (Thermo, Genesys 10 UV) at the wavelengths of 510 and 700 nm (A_{510} and A_{700}), being A determined by the equation: $A = (A_{510\ nm} - A_{700\ nm})_{pH\ 1.0} - (A_{510\ nm} - A_{700\ nm})_{pH\ 4.5}$. The monomeric anthocyanin pigment concentration was calculated as cyanidin-3-glucoside, being the concentration determined by the equation: Monomeric anthocyanin pigment (mg Cy 3-glu/L) = $A \times MW \times DF \times 1000/(\varepsilon \times 1)$, where A = absorbances difference, MW= molecular weight (449.2), DF = dilution factor, and ε = molar absorptivity (26,900). The results were then converted into flowers dry weight. All measurements were performed in triplicate.

1.7 Total Reducing Capacity

The Total Reducing Capacity (TRC) of each sample was determined by the Folin-Ciocalteu method, described by Falcão et al. (2007). To 8 mL of the different pansy extracts solutions was added 500 µL of Folin-Ciocalteu reagent. The blank and standards were prepared similarly, replacing the sample by the solvent used in the extraction and standards, respectively. After 3 to 8 minutes, 1.5 mL saturated sodium carbonate solution was added. After two hours, the absorbance values were read at 765 nm. A calibration curve was obtained with gallic acid (0.25 to 5 mg/L) and the results expressed on mg gallic acid equivalent (GAE)/g flower, d.w. All measurements were performed in triplicate.

1.8 Determination of Antioxidant Activity

1.8.1 DPPH (2,2-diphenyl-1-picrylhydrazyl) radical scavenging activity

DPPH radical scavenging activity was determined by the procedure described by Delgado et al. (2010), with some modifications. A 0.0024 g mass of DPPH was dissolved in 100 mL of methanol to obtain a solution 6.09×10^{-5} mol/L. The pansy extract solutions were diluted with the solvent used during extraction and 300 µL of these solutions was added to 2.7 mL of DPPH methanolic

solution. After 1 hour in the dark at room temperature, the absorbance was read at 517 nm. Antioxidant activity was expressed by the percentage of scavenging effect according to the formula in Eq. 1:

$$DPPH\ radical\ scavenging\ effect\ (\%) = \frac{A_{DPPH} - A_{Sample}}{A_{DPPH}} \times 100 \quad (1)$$

A_{DPPH} was the absorbance of the DPPH solution and A_{Sample} the absorbance in the presence of the sample. The blanks were made with the different solutions used in the extractions. The extract concentration providing 50% DPPH radical scavenging effect (EC_{50}) was calculated from the graph of DPPH radical scavenging effect (%) *versus* extract concentration. All measurements were performed in triplicate.

1.8.2 Reducing power

The reducing power of the extracts was determined by the procedure described by Delgado et al. (2010). To 1.0 mL of pansies extracts solutions at different concentrations was added 2.5 mL of phosphate buffer 0.2 M (pH 6.6) and 2.5 mL of $K_3[Fe(CN)_6]$ 1% (m/v). After shaking, the mixture was incubated at 50 °C for 20 minutes. After, 2.5 mL of 10% trichloroacetic acid (m/v) was added with further stirring. A volume of 2.5 mL of the mixture was transferred to another test tube, to which 2.5 mL distilled water and 0.5 mL $FeCl_3$ 0.1 % (m/v) were added. The absorbance values were read at 700 nm. From the graph Abs (700 nm) *versus* extract concentration, the EC_{50} values were determined corresponding to the extract concentration that gave an absorbance of 0.5. Each solution was analyzed in triplicate.

1.9 Statistical analysis

SPSS Statistic software, version 18.0 (SPSS Inc., Chicago, USA), was used for the statistical treatment of the data. Analyses of variance (ANOVA) or ANOVA Welch were carried out to evaluate if there were significant differences ($p < 0.05$) between samples. Additionally, significant post hoc analyses were performed (Tukey HSD test if variances in the different groups were identical or Games-Howell test if they were not). The homogeneity of variance was tested by Levene's test. The correlations between variables were determined by Pearson correlation coefficient. A Principal Component Analysis (PCA) was performed to differentiate pansies extracts. The variables considered were the Total Reducing Capacity, hydrolysable tannins, flavonoids and anthocyanin contents, as well as, the EC_{50} values of the DPPH and Reducing Power assays.

2. RESULTS AND DISCUSSION

2.1 Extraction yield

The extraction yields for pansies after applying different solvents are presented in Table 1. The extraction yield for methanol was higher (42.0%) than for the other two solutions, namely water (36.9%) and water:acetone (39.9%).

2.2 Flavonoids

Acording to Skowyra et al. (2014), flavonoids represent the main group of phenolic compounds in pansies. In the present work the flavonoid contents extracted from pansies ranged between 46.9 and 114.7 mg QE/g flower (d.w.) (Table 1), depending on the solvent. The highest content was obtained with water:acetone, approximately 2.4 times more than with water. These results were similar to Guinot et al. (2008), who reported that the organic solvent-water extracts (ethanol:water, 3:7, v/v) of marigold flowers had higher levels of total flavonoids than the solution obtained through the traditional decoction method (distilled water for 10 min). Moreover, Liu et al. (2009) also found that acetone extracts of lychee flowers had higher flavonoids contents than their water extracts.

Fernandes, L., Pereira, J. A., Saraiva, J., Casal, S. & Ramalhosa, E. (2019).
Extraction solvents' influence on the content of bioactive compounds and antioxidant activity of pansies. *Millenium*, 2(8), 89-99.
DOI: https://doi.org/10.29352/mill0208.08.00205

Table 1 - Flavonoids, hydrolysable tannins, monomeric anthocyanins, TRC, EC_{50} DPPH and EC_{50} Reducing Power values for pansies*.

Parameters	Solvent		
	Water	Water:acetone (6:4, v/v)	Methanol
Extraction yield (%)	36.9±0.1[a]	39.9±0.5[b]	42.0±0.3[c]
Flavonoids (mg QE/g flower, d.w.)	46.9±0.7[a]	114.7±1.0[c]	99.6±2.2[b]
Hydrolysable tannins (mg TAE/g flower, d.w.)	6.77±0.65[a]	12.8±1.9[b]	17.4±2.7[b]
Monomeric anthocyanins (mg Cy 3-glu/g flower, d.w.)	3.19±0.31[a]	2.59±0.09[a]	5.93±0.18[b]
TRC (mg GAE/g flower, d.w.)	27.2±2.5[a]	42.8±1.8[b]	39.3±2.7[b]
EC_{50} DPPH (mg extract/mL)	0.22±0.01[c]	0.15±0.01[a]	0.17±0.01[b]
EC_{50} Reducing power (mg extract/mL)	0.60±0.01[b]	0.33±0.01[a]	0.36±0.08[a]

*Values are expressed as: Mean±Standard deviation. Values with the same letter in the same line are not statistically different (p>0.05).

2.3 Hydrolysable tannins

Significant differences on hydrolysable tannins contents were detected between pansies extracts (Table 1), ranging from 6.77 to 17.4 mg TAE/g flower, d.w.. Methanol and water:acetone (6:4, v/v) were the best solvents to extract hydrolysable tannins in pansies. According to Mueller-Harvey (2001), methanol tends to be the best solvent for tannins of low molecular weight or if the tissues contain large amounts of enzymes; however, acetone is often the preferred solvent, as it is less likely to react with hydrolysable tannins than water or methanol.

Several solvent systems have been used, namely methanol, ethanol, acetone, water and their combination for tannins' extraction. For example, Hagerman (1988) studied the extraction of tannins from fresh and preserved leaves of three species of trees and reported that more tannins were extracted with aqueous acetone than with aqueous or acidic methanol. On the other hand, Elfalleh et al. (2012) investigated pomegranate flowers and concluded that methanol extracted more hydrolysable tannins than water (148.24 versus 57.04 mg TAE/g, d.w.). Both authors obtained similar results to ours. So, methanol and acetone:water combinations are good solvent systems to extract hydrolysable tannins.

2.4 Monomeric anthocyanins

The total monomeric anthocyanins contents of pansies extracts analyzed in the present study varied significantly among the solvents used in the extraction, ranging from 2.59 (water:acetone) to 5.93 (methanol) mg Cy 3-glu/g flower, d.w.

Skowyra et al. (2014) obtained values for total monomeric anthocyanins subjected to water extraction between 4.11 to 21.50 mg of malvidin glucoside equivalents (ME)/g of freeze-dried weight. Furthermore, the extracts obtained with the three solvents had different colors (Figure 2). This fact may be the result of different physical properties (solubility) and different types of co-extracted pigments, as suggested by Boonsong et al. (2011). Furthermore, it might be due to the extraction of different anthocyanins by the solvents with different polarities (water =1.00; methanol=0.76 and water:acetone (6:4, v/v)=0.74). Concerning pH, the three solvents presented similar values (water = 6.47; water:acetone (6:4, v/v) = 6.96 ; methanol= 6.41). Abarca-Vargas et al. (2016) also obtained extracts of *Bougainvillea × buttiana* Holttum and Standl, (var. Rose) with different colors when using solvents with different polarities, as well as Bertan et al. (2014) when studying the effect of the ethanol concentration in the solvent on the antioxidant properties of extracts of boldo (*Peumus boldus*).

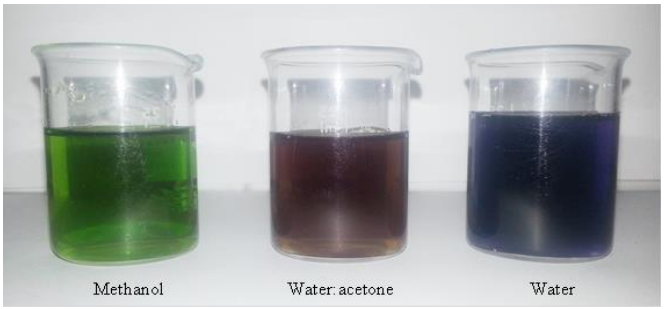


Figure 2 – Pansies extracts with different solvents: methanol, water:acetone (6:4, v/v) and water.

2.5 Total Reducing Capacity

The total reducing capacity (TRC) of the pansies extracts obtained in the present work are presented in Table 1, varying from 27.2 to 42.8 mg GAE/g flower, d.w.. Naczk and Shahidi (2006) mentioned that the extraction of phenolic compounds in plant materials is influenced by their chemical nature and interactions with other plant components such as carbohydrates and proteins. These interactions may lead to the formation of complexes that may be quite insoluble. In the present work, water:acetone and methanol yielded no statistically different amounts, but the highest value of TRC was obtained with water:acetone. According to Uma et al. (2010) and Kuźma et al. (2014), aqueous acetone solutions (48 and 70%, respectively) are the most effective solvent for extraction of polyphenols from henna leaves (*Lawsonia inermis*) and parsley leaves (*Petroselinum crispum*), which is in accordance with our results. On contrary, Shabir et al. (2011), evaluated different extraction solvents in Gold Mohar flowers and detected that 80% methanol gave the highest value of total phenolics contents, as well as Ahmad et al. (2011), who studied flowers of akk and reported that higher values were obtained with 80% ethanol. These different results can be explained because different flowers may synthesize and accumulate different compounds or different amounts of a particular compound, which in turn affects the TRC of the flowers extracts produced. Our results of water extraction was lower (27.2 mg GAE/g flower d.w.) than reported by Skowyra et al. (2014), who obtained a range of values between 120.56 (yellow pansies) to 419.28 (red) mg GAE/g of freeze-dried weight, after applying different extraction conditions.

2.6 Antioxidant activity

2.6.1 DPPH radical scavenging activity

The antioxidant activity determined by the DPPH method for the pansies extracts studied in present work showed significant differences between them (Figure 3A and Table 1). The DPPH free radical scavenging activity increased with extract concentration and the water:acetone (6:4, v/v) solvent was the one with the highest values (Figure 3A). The antioxidant activity was also expressed on EC_{50} values, which indicate the concentration of the extract required to decrease the DPPH radical concentration by 50% (Table 1). Thus, a low EC_{50} value is indicative of a high antioxidant activity. Water:acetone extracts exhibited the lowest EC_{50} value (0.15 mg extract/mL), while water extracts resulted in the highest (0.22 mg extract/mL). These results agree with Kuźma et al. (2014), who reported that higher antioxidant activity were obtained with aqueous organic solvents than with the respective absolute organic solvents. Furthermore, these results can be explained by the polarity because by increasing the proportion of water to the solvent, the polarity of the mixture also increases.

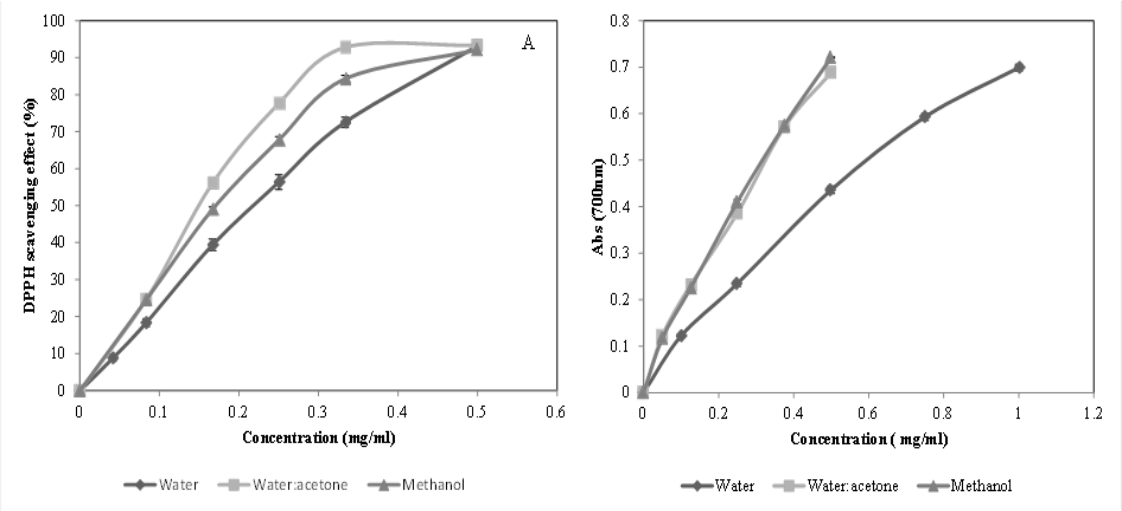


Figure 3 - DPPH radical scavenging activity (%) (A) and reducing power (Abs 700 nm) (B) *versus* pansies extracts concentration.

2.6.2 Reducing Power

The measurement of the reducing power can also be used to express the antioxidant activity of the plant extracts. In this assay, ferric ions are reduced to ferrous ions with change in color from yellow to bluish green (Ahmand et al., 2011). The intensity of the color depends on the reducing potential of the antioxidant compounds present in the extract. The reducing power data of the different extracts followed the similar trends as were observed for hydrolysable tannins and TRC assay, compounds responsible for the antioxidant activity in the extracts. So, methanol and water:acetone (6:4, v/v) extracts showed the lowest values of EC_{50} reducing power, while water extracts had the highest (Table 1). Furthermore, in the present work the reducing power was tested at different extract concentrations and reducing power increased with the extract concentration (Figure 3B).

Fernandes, L., Pereira, J. A., Saraiva, J., Casal, S. & Ramalhosa, E. (2019).
Extraction solvents' influence on the content of bioactive compounds and antioxidant activity of pansies. *Millenium*, 2(8), 89-99.
DOI: https://doi.org/10.29352/mill0208.08.00205

2.7 Correlations between Total Reducing Capacity, monomeric anthocyanins, flavonoids, hydrolysable tannins and antioxidant activity

The Pearson correlation coefficients determined between TRC, monomeric anthocyanins, flavonoids, hydrolysable tannins and antioxidant activity (EC_{50} values of DPPH and Reducing Power assays) are presented in Table 2. Significant positive correlations were found between TRC and flavonoids (0.968), as well as hydrolysable tannins (0.789), showing the important role of these compounds in TRC. As expected, negative correlations were detected between TRC and the EC_{50} values of DPPH (-0.971) and Reducing Power (-0.907), as these properties are inversely correlated. Concerning antioxidant activity, a significant negative correlations were found between flavonoids and EC_{50} DPPH values (-0.992) and EC_{50} reducing power (-0.953), related with the antioxidant potential of these compounds.

Table 2 - Pearson correlation coefficients for Total Reducing Capacity, monomeric anthocyanins, flavonoids, hydrolysable tannins and EC_{50} values of DPPH radical scavenging activity and Reducing Power assays.

	Monomeric anthocyanins	Flavonoids	Hydrolysable tannins	EC_{50} DPPH	EC_{50} Reducing Power
Total Reducing Capacity	0.003	0.968**	0.789*	- 0.971**	-0.907**
Monomeric anthocyanins	___	0.018	0.676	0.71	-0.014
Flavonoids	___	___	0.751*	- 0.992**	-0.953**
Hydrolysable tannins	___	___	___	-0.699*	-0.708**
EC_{50} DPPH	___	___	___	___	0.946**

Correlation is significant at **p < 0.01, *p < 0.05

2.8 Principal Component Analysis

A Principal Component Analysis (PCA) was applied to find possible clusters within the extracts prepared with the solvents studied in the present work, which may differ in bioactive compounds and antioxidant activity. The scores of the first two principal components are presented in Figure 4. The first two principal components took into account 99.6% (PC1 = 97.5% and PC2 = 2.1%, respectively) of the total variation. PC1 was mainly correlated positively to EC_{50} DPPH and EC_{50} Reducing Power, and negatively to TRC, hydrolysable tannins and flavonoids. PC2 was mainly correlated positively to monomeric anthocyanins. In PC1, aqueous extracts had positive scores due to their high values of EC_{50} DPPH and Reducing Power, as well as, low values of TRC, hydrolysable tannins and flavonoids, as stated in Table 1. So, when extracting pansies with water, the extracts obtained had the lowest antioxidant activity and the lowest contents of bioactive compounds. On contrary, the methanolic extracts had positive scores on PC2 due to their high values of monomeric anthocyanins. As stated in previous sections, the methanolic extracts were those with the highest values of hydrolysable tannins and monomeric anthocyanins. Concerning the water:acetone (6:4, v/v) solution, these extracts showed the highest antioxidant activity and flavonoids contents.

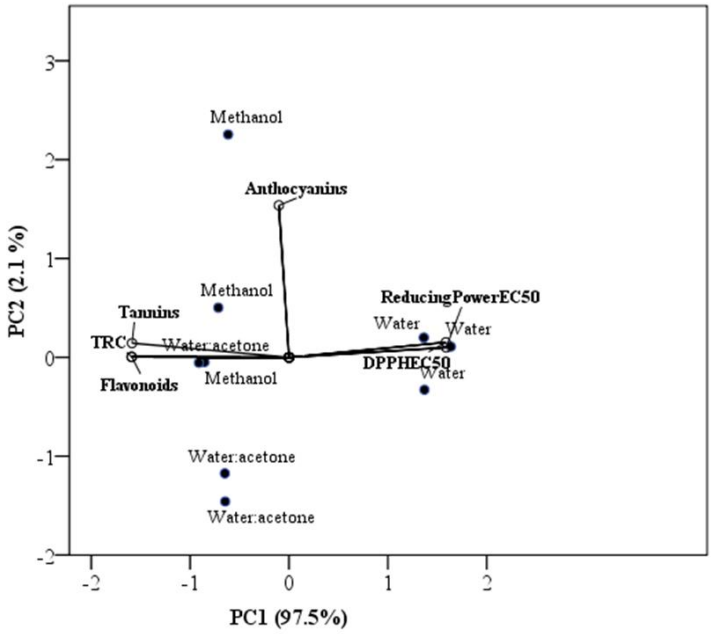


Figure 4 - Principal component analysis plot to flavonoids, monomeric anthocyanins, hydrolysable tannins, TRC and EC_{50} values of DPPH radical scavenging activity and Reducing Power assays.

CONCLUSIONS

The influence of the solvent (water, methanol, water:acetone (6:4, v/v)) on bioactive compounds extraction and antioxidant properties of pansies was demonstrated. The highest flavonoids content was obtained with water:acetone (6:4, v/v), while methanol extract had the highest value of monomeric anthocyanins. Concerning hydrolysable tannins and TRC, both methanol and water:acetone (6:4, v/v) extracts had similar contents. Aqueous extracts presented the lowest antioxidant activity. These results demonstrated the possibility of *Viola × wittrockiana* flowers to be a promising source of natural antioxidants for the food and pharmaceutical industries.

REFERENCES

Abarca-Vargas, R., Malacara, C.F.P., & Petricevich, V.L. (2016). Characterization of chemical compounds with antioxidant and cytotoxic activities in *Bougainvillea x buttiana* holttum and standl, (var. rose) extracts. *Antioxidants*, 5, 2-11. DOI: 10.3390/antiox5040045

Ahmad, N., Anwar, F., Hameed, S., & Boyce, M.C. (2011). Antioxidant and antimicrobial attributes of different solvent extracts from leaves and flowers of akk [*Calotropis procera* (Ait.) Ait. F.)]. *Journal of Medicinal Plants Research*, 5, 4879-4887.

Bchir, B., Besbes, S., Karoui, R., Attia, H., Paquot, M., & Blecker, C. (2012). Effect of air-drying conditions on physico-chemical properties of osmotically pre-treated pomegranate seeds. *Food and Bioprocess Technology*, 5, 1840-1852. DOI: 10.1007/s11947-010-0469-3

Benvenuti, S., Bortolotti, E., & Maggini, R. (2016). Antioxidant power, anthocyanin content and organoleptic performance of edible flowers. *Scientia Horticulturae*, *199*, 170–177. DOI: 10.1016/j.scienta.2015.12.052

Bertan, D.W., Makishi, G.L.A., Arantes, K.S., & Sobral, P.J.A. (2014). Caracterização de extratos de folhas de boldo-do-Chile produzidos com soluções hidroetanólicas. SIICUSP 2014 – 22º Simpósio Internacional de Iniciação Científica e Tecnológica da USP.

Boonsong, P., Laohakunjit, N., & Kerdchoechuen, O. (2011). Detection of pigments and natural colorants from thai herbal plants for possible use as coloring dyes. *Hortscience,* 46(2), 265-272.

Dai, J., & Mumper, R.J. (2010). Plant phenolics: extraction, analysis and their antioxidant and anticancer properties. *Molecules,* 15, 7313-7352. DOI: 10.3390/molecules15107313.

Fernandes, L., Pereira, J. A., Saraiva, J., Casal, S. & Ramalhosa, E. (2019).
Extraction solvents' influence on the content of bioactive compounds and antioxidant activity of pansies. *Millenium*, 2(8), 89-99.
DOI: https://doi.org/10.29352/mill0208.08.00205

Delgado, T., Malheiro R., Pereira, J.A., & Ramalhosa, E. (2010). Hazelnut (*Corylus avellana* L.) kernels as a source of antioxidants and their potential in relation to other nuts. *Industrial Crops and Products*, 32, 621-626. DOI: 10.1016/j.indcrop.2010.07.019

Elfalleh, W., Hannachi, H., Tlili, N., Yahia, Y., Nasri, N., & Ferchichi, A. (2012). Total phenolic contents and antioxidant activities of pomegranate peel, seed, leaf and flower. *Journal of Medicinal Plants Research*, 6, 4724-4730. DOI: 10.5897/JMPR11.995

Falcão, A.P., Chaves, E.S., Kuskoski, E.M., Fett, R., Falcão, L.D., & Bordignon-Luiz, M.T. (2007). Total polyphenol index, total anthocyanins and antioxidant activity of a model system of grape jelly. *Ciência e Tecnologia de Alimentos*, 27, 637-642. DOI: 10.1590/S0101-20612007000300032

Gamsjaeger, S., Baranska, M., Schulz, H., Heiselmayere, P., & Musso, M. (2011). Discrimination of carotenoid and flavonoid content in petals of pansy cultivars (*Viola × wittrockiana*) by FT-Raman spectroscopy. *Journal of Raman Spectroscopy,* 42, 1240–1247. DOI: 10.1002/jrs.2860

Guinot, P., Gargadennec, A., Valette, G., Fruchier, A., & Andary, C. (2008). Primary flavonoids in marigold dye: extraction, structure and involvement in the dyeing process. *Phytochemical Analysis*, 19, 46–51. DOI: 10.1002/pca.1014

González-Barrio, R., Periago, M.J., Luna-Recio, C., Javier, G.F., Navarro-González, I. (2018). Chemical composition of the edible flowers, pansy (*Viola wittrockiana*) and snapdragon (*Antirrhinum majus*) as new sources of bioactive compounds. *Food Chemistry*, 252, 373–380. DOI:10.1016/j.foodchem.2018.01.102

Hagerman, A.E. (1988). Extraction of tannin from fresh and preserved leaves. *Journal of Chemical Ecology*, 14, 453-461. DOI: 10.1007/BF01013897.

Hase, N., Matsuura, S., & Yamaguchi, M. (2005). HPLC evaluation of anthocyanins and flavonols in relation to the flower color of pansy (*Viola× wittrockiana* Gams). *Horticultural Research Japan,* 4, 125-129. DOI: 10.2503/hrj.4.125

Jun, X. (2013). High-Pressure processing as emergent technology for the extraction of bioactive ingredients from plant materials. *Critical Reviews in Food Science and Nutrition*, 53, 837-852. DOI: 10.1080/10408398.2011.561380.

Kelley, K.M., Cameron, A.C., Biernbaum, J.A, & Poff, K.L. (2003). Effect of storage temperature on the quality of edible flowers. *Postharvest Biology and Technology*, 27, 341-344. DOI: 10.1016/S0925-5214(02)00096-0

Kuźma, P., Drużyńska, B., & Obiedziński, M. (2014). Optimization of extraction conditions of some polyphenolic compounds from parsley leaves (*Petroselinum crispum*). *Acta Scientiarum Polonorum Technologia Alimentaria*, 13, 145-154. DOI: 10.17306/J.AFS.2014.2.4

Li, Q., Wang, J., Sun, H.-Y., & Shang, X. (2014a). Flower color patterning in pansy (*Viola×wittrockiana* Gams.) is caused by the differential expression of three genes from the anthocyanin pathway in acyanic and cyanic flower areas. *Plant Physiology and Biochemistry*, 84, 134-141. DOI: 10.1016/j.plaphy.2014.09.012.

Li, A-N., Li, S., Li, H-B., Xu, D-P., Xu, X-R., & Chen, F. (2014b). Total phenolic contents and antioxidant capacities of 51 edible and wild flowers. *Journal of Functional Foods,* 6, 319-330. DOI: 10.1016/j.jff.2013.10.022

Liu, S., Lin, J., Wang, C., Chen, H., & Yang, D. (2009). Antioxidant properties of various solvent extracts from lychee (*Litchi chinenesis* Sonn.) flowers. *Food Chemistry,* 114, 577-581. DOI: 10.1016/j.foodchem.2008.09.088

Mueller-Harvey, I. (2001). Analysis of hydrolysable tannins. *Animal Feed Science and Technology*, 91, 3–20. DOI: 10.1016/S0377-8401(01)00227-9

Naczk, M., & Shahidi, F. (2006). Phenolics in cereals, fruits and vegetables: occurrence, extraction and analysis. *Journal of Pharmaceutical and Biomedical Analysis,* 41, 1523–1542. DOI: 10.1016/j.jpba.2006.04.002

Rajasekar, D., Akoh, C.C., Martino, K.G., & MacLean, D.D. (2012). Physico-chemical characteristics of juice extracted by blender and mechanical press from pomegranate cultivars grown in Georgia. *Food Chemistry*, 133, 1383-1393. DOI: 10.1016/j.foodchem.2012.02.025

Rop, O., Mlcek, J., Jurikova, T., Neugebauerova, J., & Vabkova, J. (2012). Edible flowers - a new promising source of mineral elements in human nutrition. *Molecules*, *17*, 6672-6683. DOI: 10.3390/molecules17066672.

Shabir, G., Anwar, F., Sultana, B., Khalid, Z.M., Afzal, M., Khan, Q.M., & Ashrafuzzaman, M. (2011). Antioxidant and antimicrobial attributes and phenolics of different solvent extracts from leaves, flowers and bark of Gold Mohar [*Delonix regia* (Bojer ex Hook.) Raf.]. *Molecules*, 16, 7302-7319. DOI: 10.3390/molecules16097302.

Skowyra, M., Calvo, M.I., Gallego, M.G. Azman, N.A.M. & Almajano, M.P. (2014). Characterization of phytochemicals in petals of different colours from *Viola × wittrockiana* Gams. and their correlation with antioxidant activity. *Journal of Agricultural Science,* 6, 93-105. DOI: 10.5539/jas.v6n9p93

Uma, D.B. Ho C.W., & Aida W.M.W. (2010). Optimization of extraction parameters of total phenolic compounds from Henna (*Lawsonia inermis*) leaves. *Sains Malaysiana*, 39, 119 -128.

Viuda-Martos, M., Ruiz-Navajas, Y., Fernández-López, J., Sendra, E., Sayas-Barberá, E., & Pérez-Álvarez, J.A. (2011). Antioxidant properties of pomegranate (*Punica granatum* L.) bagasses obtained as co-product in the juice extraction. *Food Research International*, 44, 1217-1223. DOI: 10.1016/j.foodres.2010.10.057

Vukics, V., Kery, A., & Guttman, A. (2008). Analysis of polar antioxidants in heartsease (*Viola tricolor* L.) and garden pansy (*Viola × wittrockiana* Gams.). Journal of *Chromatographic Science*, 46, 823-827. DOI: 10.1093/chromsci/46.9.823

Weryszko-Chmielewska, E., & Sulborska, A. (2012). Diversity in the structure of the petal epidermis emitting odorous compounds in *Viola × wittrockiana* Gams. *Acta Scientiarum Polonorum Hortorum Cultus,* 11, 155-167.

CIÊNCIAS DA VIDA E DA SAÚDE

LIFE AND HEALTH SCIENCES

CIENCIAS DE LA VIDA Y LA SALUD

millenium

CIÊNCIAS DA VIDA E DA SAÚDE
LIFE AND HEALTH SCIENCES
CIENCIAS DE LA VIDA Y LA SALUD

millenium 103

Millenium, 2(8), 103-111.

en

PROMOÇÃO DA INDEPENDÊNCIA FUNCIONAL EM IDOSOS INSTITUCIONALIZADOS

PROMOTION OF FUNCTIONAL INDEPENDENCE INSTITUTIONALIZED ELDERLY

PROMOCIÓN DE LA INDEPENDENCIA FUNCIONAL EN ANCIANOS INSTITUCIONALIZADOS

Carla Mendes[1]

[1] *Centro Hospitalar Médio Tejo, EPE - Hospital Nossa Senhora da Graça, Tomar, Portugal*

Carla Mendes - carlmamendes@hotmail.com

Corresponding Author
Carla Mendes
Centro Hospitalar do Médio Tejo, E.P.E
Av. Maria de Lourdes de Mello Castro – Ap. 118
2304-909 Tomar
carlmamendes@hotmail.com

RECEIVED: 11th December 2018
ACCEPTED: 21th January 2019

Mendes, C. (2019). Promotion of functional independence institutionalized elderly.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

RESUMO

Introdução: Com o envelhecimento verifica-se um declínio das funções físicas e cognitivas da pessoa idosa, o que leva ao comprometimento da sua independência funcional (F.I.).
Objetivo: Comparar a independência funcional de dois grupos de idosos com características inicialmente semelhantes, residentes numa Estrutura Residencial para Idosos, sedentários, sendo um dos grupos submetido a um programa de exercício físico (P.E.P.).
Métodos: Estudo quantitativo, de natureza quase-experimental. Recorreu-se a uma amostra de 20 idosos divididos em dois grupos semelhantes, experimental e controlo, de dez elementos cada. Um dos grupos praticou um P.E.P., que incluiu treino de força muscular e treino aeróbio, durante 12 semanas, 5 dias por semana, a uma intensidade moderada. Utilizou-se a Medida de Independência Funcional (FIM) para avaliar a independência funcional dos idosos num primeiro momento e, posteriormente num segundo momento, isto é, após a implementação do P.E.P. ao grupo experimental. A análise dos dados foi efetuada com recurso à estatística descritiva e inferencial, nomeadamente com o teste exato de Fisher, Mann-Whitney e Wilcoxon, considerando $p<0,05$.
Resultados: No final do estudo o grupo experimental melhorou a sua independência funcional nos subníveis "banho", "banheira e duche" e "escadas", mantendo todos os outros, enquanto o grupo controlo manteve a independência funcional em todos os subníveis avaliados.
Conclusões: A prática de exercício físico permite manter e/ou melhorar a independência funcional dos idosos.

Palavras-chave: idoso; exercício físico; independência.

ABSTRACT

Introduction: With aging there is a decline in the physical and cognitive function of the elderly, which leads to impairment of the Functional Independence (F.I.) of the elderly person.
Objective: To compare functional independence of two elderly groups with initially similar characteristics, living in a residential structure for the elderly, one of the groups being submitted to a Physical Exercise Program (P.E.P.)
Method: Quantitative, quasi-experimental study. A sample of 20 elderly people divided into two similar groups, experimental and control, of ten elements each. One of the groups practiced P.E.P., which included muscle strength training and aerobic training for 12 weeks, 5 days a week, at moderate intensity. The Functional Independence Measure (FIM) was used to evaluate the functional independence of the elderly at first, and then in second moment, that is, after the implementation of P.E.P. to the experimental group. Data analysis was performed using descriptive and inferential statistics, namely with Fisher's exact test, Mann-Whitney and Wilcoxon, considering $p <0.05$.
Results: At the end of the study the experimental group improved its functional independence in the "bath", "bath and shower" and "stairs" sub-levels, while maintaining all others, while the control group maintained functional independence in all evaluated sub-levels.
Conclusion: The practice of physical exercise allows to maintain and/or improve functional independence of the elderly.

Keywords: elderly; physical exercise; independence.

RESUMEN

Introducción: Con el envejecimiento se verifica un declive de las funciones físicas y cognitivas de la persona mayor, lo que lleva al compromiso de la independencia funcional (F.I.) de la misma.
Objetivos: Comparar la independencia funcional de dos grupos de ancianos con características inicialmente similares, residentes en una estrutura residencial para ancianos, sedentarios, siendo uno de los grupos sometido a un programa de ejercicio físico (P.E.P.).
Métodos: Estudio cuantitativo, de naturaleza cuasi-experimental. Se recurrió a una muestra de 20 ancianos divididos en dos grupos similares, experimental y control, de diez elementos cada uno. Uno de los grupos practicó un P.E.P., que incluyó entrenamiento de fuerza muscular y entrenamiento aeróbico, durante 12 semanas, 5 días a la semana, a una intensidad moderada. Se utilizó la Medida de Independencia Funcional (FMI) para evaluar la independencia funcional de los ancianos en un primer momento y, posteriormente en un segundo momento, es decir, después de la implementación del P.E.P. al grupo experimental. El análisis de los datos fue efectuado con recurso a la estadística descriptiva e inferencial, en particular con la prueba exacta de Fisher, Mann-Whitney y Wilcoxon, considerando $p <0,05$.
Resultados: Al final del estudio el grupo experimental mejoró su independencia funcional en los subniveles "baño", "bañera y ducha" y "escaleras", manteniendo a todos los demás, mientras que el grupo control mantuvo la independencia funcional en todos los subniveles evaluados.
Conclusións: La práctica de ejercicio físico permite mantener y/o mejorar la independencia funcional de los ancianos.

Palabras Clave: anciano; ejercicio físico; independencia.

INTRODUCTION

Aging is a progressive, universal and multifactorial process that occurs in a heterogeneous way, that is, each person ages in a unique and particular way. It is mainly in the Third Age, the stage where significant changes in physical, psychic and social conditions that condition the elderly person's adaptation to the environment and, consequently, their functional independence and quality of life (Park et al., 2014). The advances achieved with the industrial revolution have altered people's lifestyles, improving their quality of life, health and, consequently, longevity. In recent decades there has been an increase in the elderly population in industrialized countries and, in Portugal, about 20% of the population is over 65 years old (Portada, 2014). The woman, who until now was the traditional caregiver of the elderly in the family, began to enter the labor market (Barros et al., 2016), which led to a social, cultural, economic and family structure adjustment.

The advances achieved with the industrial revolution have altered people's lifestyles, improving their quality of life, health and, consequently, longevity. In recent decades there has been an increase in the elderly population in industrialized countries and, in Portugal, about 20% of the population is over 65 years old (Portada, 2014). The woman, who until now was the traditional caregiver of the elderly in the family, began to enter the labor market (Barros et al., 2016), which led to a social, cultural, economic and family structure adjustment.

Thus, as we see an increase in the average life expectancy, we are also witnessing an increase in the number of residential structures for the elderly, which assume the responsibility of caring for the elderly, dependent or independent, who can not remain with the family or in their own homes, providing them with housing, food, health care and social interaction (Carneiro et al., 2012).

In general, in the residential structures for the elderly, the activities developed by the elderly who live there are usually few, which is associated with the aging process and will condition their functional capacity. In this sense, the sedentarism common to all the elderly leads them to the loss of the autonomy and, to an increasing difficulty in performing the tasks of the day to day (Barros et al., 2016). The fragility of the elderly is a situation characterized by the decline of the various systems of the body and therefore their physical and mental capacities decrease, increasing their vulnerability to health problems (Fairhall et al., 2013). Functional decline is thus a factor to combat in this age group as a means of promoting health, increasing the sense of well-being, as well as reducing health costs.

In this context, was practized a P.E.P. to a group of elderly people living in a home of the Third Age in central Portugal as a means to promote their functional independence. Thus, this study aims to analyze the F.I. of the elderly, sedentary, before and after participation in a P.E.P. and then compare it with another group with initially similar characteristics but, who did not practice P.E.P..

1. PHYSICAL ACTIVITY AND FUNCTIONAL INDEPENDENCE IN THE ELDERLY

In the last century the increase of the elderly population worldwide has worried society in general and health professionals in particular, which has led to the development of health promotion and wellness programs for this age group (Crocker et al. Chow et al., 2014).

In this dynamic, by the analyzed studies, it was verified that the participation of the elderly in a P.E.P. may be a way to combat the sedentary lifestyle and thus maintain the independence of the elderly as long as possible. The training of components of physical fitness as a way of maintaining autonomy is a factor that plays a relevant role throughout life, that is, leads to a reduction of non-transmissible diseases, fewer falls, fewer health costs, as well as improvement of self-concept and self-esteem (Park et al., 2014).

The practice of physical exercise is seen as a way that leads the elderly to maintain or even improve their autonomy, as well as a way to prevent physical and mental illnesses, facilitating the adaptation of the elderly to the environment (Crocker et al. 2013).

Park et al. (2014) analyzed several studies on the effects of P.E.P. on the depressive symptoms, the quality of life and, the self-esteem, concluding that the practice of physical exercise improved all the variables studied, in this way the authors recommend the practice of physical exercise by the elderly.

Also Crocker et al. (2013) carried out an analysis of several studies whose objective was to analyze the effects of P.E.F. on the activities of daily living in elderly people living in nursing homes. The authors came to the conclusion that the independence of the elderly was improved through P.E.P., although they do not know which interventions are most appropriate to apply to this age group.

Also Gonçalves et al. (2010) analyzed the relationship between physical fitness and functional capacity of elderly people living in a residential structure for the elderly, verifying that F.I. and physical fitness are related, because the better the physical fitness (coordination, muscular strength, aerobic capacity, agility and coordination), the better the performance of daily activities.

Physical exercise consists of the planning, structuring and systematization of the corporal movements executed with the objective of maintaining or improving the physical capacity of the elderly (Borges, 2015; Breet et al., 2015).

One of the important factors for the elderly is the maintenance of their autonomy (Chow et al., 2014), that is, the ability to mobilize in the environment and to independently perform their basic activities of daily living (Rocha et al., 2017). In this sense the search for their health, social interaction and for pleasure leads the elderly to participate in P.E.P. (Castro et al., 2016).

Mendes, C. (2019). Promotion of functional independence institutionalized elderly.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

P.E.P. can be performed by training one or more components of physical fitness, although training involving more than one component of physical fitness in the same session is a still recent method when used to induce effects on health-related variables and maintenance of independence in the elderly population (Rocha et al., 2017).

2. METHODS

This study is characterized as a quantitative research, with a quasi-experimental design, with two groups: one experimental and the other control, each group consisting of 10 elements, where the experimental group was submitted to a program of physical exercise.

2.1 Participants

To select participants in the study FIM was applied to all the elderly living in a residential structure for the elderly, inserted in rural areas in central Portugal, which were to have, complete independence, modified independence or supervision in each sub-level of the six representative levels - self-care; sphincter control; mobility; locomotion; Communication; social cognition. In addition, they should be sedentary (this is not exercising for at least two years), which do not present with diseases such as: stroke, Parkinson's, disturbances of the body balance, visual deficit that makes it impossible to see and, uncontrolled clinical conditions such as hypertension or diabetes mellitus. Like this, 20 elderly were selected, which were later divided into two groups of 10 elements each, experimental and control, in a random way, in order to compare two groups initially similar, but that after the participation of the experimental group in a P.E.P., at the end of the study may present different characteristics.
Relatively to age in the experimental group, 40% were between 75 and 80 years old, 30% were less than 75 years old, and 30% were over 80 years old. The average age was 77.40 years. In the control group, 50% were between 75 and 80 years, 20% less than 75% and 30% more than 80 years. The average age of this group was 78 years. Most of the elements are female, 70% in the experimental group and 60% in the control group. As for schooling, 60% in the experimental group and 50% in the control group did not attend school, while the remaining elements had the first cycle of Basic Education. Comparing the groups, there were no statistically significant differences in the evaluated characteristics - age ($p = 0,543$), sex ($p = 1,000$) and schooling ($p = 1,000$).

2.2 Instruments

Participants were assessed by the FIM scale at two different times. At first, when all the elderly residents in the institution were evaluated and, in a second moment, this one that was after the experimental group to have practiced P.E.P.. It is necessary to take into account that the experimental group before starting the exercise program had to evaluate the muscular strength to adapt the load to be used in it, to teach to execute the movements with the correct technique and to use the scale of subjective perception of Borg effort (used to assess effort tolerance).
The FIM is a scale that has been translated adapted to the Portuguese population by Lains (1991). This instrument aims to assess what the person actually can do, as well as determine the help a person needs to perform motor and cognitive tasks. The FIM allows the evaluation of six levels: self-care, control of sphincters, mobility, locomotion, communication and social cognition, which are subdivided into 18 sub levels. Each sub-level receives the score from 1 to 7, which ranges from total dependence to complete independence. Each level is analyzed by the sum of the sub-levels that constitute it. The total value of the FIM corresponds to the sum of the value of the sub-levels in each level and ranges from 18 to 126 points.
The Cronbach alpha coefficient was determined to evaluate the homogeneity of the items that make up each of the dimensions of the FIM and, of all items in the set, obtaining the values presented in table 1. In the opinion of Maroco and Garcia-Marques (2006) values equal to or greater than 0,60 are acceptable, especially when the number of items is reduced. Thus, internal consistency can be said to be acceptable.

Table 1 – Cronbach alpha coefficients obtained for FIM

Levels	Nr. Of Items	1st evaluation	2nd evaluation
Self-care	6	0,445	0,528
Sphincter control	2	0,176	0,089
Mobility	3	0,000	0,000
Locomotion	2	0,375	0,480
Comunication	2	0,000	0,000
Social cognition	3	0,850	0,828
Total FIM	18	0,732	0,782

2.3 Dependent variable and independent variable

The dependent variable is the F.I. of the elderly, operationalized and measured by FIM, in the following items: Self-care (A-feeding, B-personal hygiene, C- D-wear upper half, E-wear half lower, F-use toilet); Control of sphincters (G-bladder, H-intestine); Mobility (I-bed, chair, wheelchair, J-toilet, K-bathroom / shower); Locomotion (L-marching / wheelchair; M-stairs); Communication (N-understanding, O-expression); Social cognition (P- social interaction, Q-resolution of problems, R- memory); Total FIM.

The independent variable is P.E.P. for the elderly. P.E.P. had a duration of 12 weeks, having been practiced 5 days a week (from Monday to Friday), always starting with a warming-up, passing through the fundamental part, and ending with the return to calm.

The warming-up period lasted 10 minutes, with no intensity, two sets of 10 repetitions of each exercise. This exercise consisted in the training movements for the upper limbs (oscillatory movements in the anteroposterior direction and crossing and at the level of the chest, in the orthostatic position and, to wander), lower limbs (gait, alternating type of gait - fast and slow, the latter being accompanied by movements of the upper limbs) and stretching exercises lumbar and hamstrings.

The fundamental part practiced on Mondays, Wednesdays and Fridays consisted of training of muscular strength and aerobic during 40 minutes. Muscle strength training was performed on the basis of the maximum weight test (Kim et al., 2002) of a maximal repetition (1RM), that is, 100% of 1 RM corresponds to the maximum weight that the person can handle certain motion. Based on this first evaluation it was adequate the weight used for the elderly to perform the planned exercises - initially 60% of 1RM and, later, from the ninth week 70% of 1RM.

Thus, the elderly performed muscular strength exercises that were: "lift and sit in a chair without arms, without hands", "flexion of the thigh", "lateral elevation of the upper limbs" and "extension of the thigh" with a intensity of 60% to 70% of 1 RM, two sets of each exercise, starting with 8 repetitions, to 10 repetitions from the ninth week, with a rest interval of 1 minute between sets, at a moderate speed, the which in Borg's subjective perceived exertion scale corresponds to 12 to 13, and then walked, gradually adding movements to the upper limbs and, up and down stairs, with an intensity of 12 to 15, according to the subjective perception of effort scale Borg.

On Tuesdays and Thursdays, the old people walked, initially on regular and later irregular terrain (sidewalk and sloping terrain), at different speeds and, up and down stairs with a maximum duration of 30 minutes.

Finally, the "return to calm", with a duration of 10 minutes, was practiced from Monday to Friday, without intensity, which began with a brief walk lasting for 1 minute at a slow pace and later stretching of the upper limbs, stretching of the sural triceps and stretching of the quadriceps, two sets of 10 repetitions, lasting 10 to 30 seconds, with an interval of 20 seconds between each exercise.

2.4 Ethical procedure

The study was approved by the institution where it was implemented on 5th Septembe, 2011 and, took into account five principles / rights that allow to protect the rights and freedoms of the people who participated in it, namely: right to self-determination, right to privacy, right to anonymity and confidentiality, right protection against discomfort and injury, and the right to fair and equitable treatment. In addition, the participants signed the informed consent form, which proves their knowledge and authorization to participate in the study.

As for the data collection, these were collected at first in March 2012 and then a second time in June 2012, after the experimental group had performed the P.E.P..

2.5 Techniques of data analysis and degree of confidence

We used the Statistical Package for the Social Sciences (SPSS) in version 20.0 to organize and systematize the information contained in the data and obtain descriptive and inferential results. Throughout the study statistical and statistical techniques (Fisher, Mann-Whitney and Wilcoxon) were used. In all tests, the value of 0.050 was set as the limit of significance.

3. RESULTS AND DISCUSSION

Maintaining functional independence, even in old age, is an essential condition for the elderly. In this sense, in table 2 we can analyze the values presented by the elements of the experimental group and the control group in the two moments of evaluation.

Mendes, C. (2019). Promotion of functional independence institutionalized elderly.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

Table 2 – Values obtained in the experimental group and in the control group in the two moments of evaluation.

		First evaluation		Second evaluation		Test of Wilcoxon
		Mean	Median	Average	Median	
Experimental Group	Self-care	6,63	6,67	6,73	6,83	P=0,014
	Sphincters	6,55	7,00	6,55	7,00	P=1,000
	Mobility	6,63	6,67	6,87	7,00	P=0,008
	Locomotion	6,25	6,00	6,65	6,50	P=0,011
	Comunication	6,65	6,75	6,80	7,00	P=0,083
	Social cognition	6,40	6,33	6,53	6,50	P=0,102
	Total FIM	6,54	6,53	6,70	6,69	P=0,007
Control Group	Self-care	6,58	6,50	6,58	6,50	P=1,000
	Sphincters	6,75	7,00	6,60	6,75	P=0,083
	Mobility	6,43	6,33	6,40	6,33	P=0,317
	Locomotion	6,35	6,50	6,35	6,50	P=1,000
	Comunication	6,80	7,00	6,75	7,00	P=0,317
	Social cognition	6,37	6,33	6,37	6,33	P=1,000
	Total FIM	6,54	6,53	6,51	6,50	P=0,059

The comparative analysis of the values obtained shows that, after participation in a P.E.P., the elderly tended to reveal a greater capacity for self-care. The comparison between the two moments of evaluation revealed statistically significant differences between these two moments (p = 0.014) in the experimental group. In the study of each of the levels that constitute this level, it was verified that the statistically significant difference occurred only in the level of "bath" (p = 0.025), and the elderly improved their level of independence. However, the elderly also improved another sub-level, but this was not found to be statistically significant, which was self-care "dressing the lower half of the body."

As for self-care "bathing" the elderly mostly had a need for supervision, where they needed to prepare the bathroom utensils, as well as being stimulated to wash the entire body from neck to feet with the exclusion of back, in the shower, to a situation where the majority no longer needed to be encouraged to perform this self-care, but it took longer to do so, and they did it in a chair with arms. The elderly have thus moved from a need for "supervision or preparation" to a need for "modified independence".

The analysis concerning the mobility level, the statistically significant difference lies in the sub-level use of "bath, shower" (p = 0.001). In this capacity the elderly mostly passed on a need for "modified independence" in which to enter and exit the shower took longer than normal and did not do so in total security, to a situation of "complete independence", where they entered and get out of the shower safely. Only one elderly woman went from a "supervision or preparation" situation where she needed to be encouraged to enter and exit the shower and required the presence of another person for a "modified independence" situation where she no longer needed of another person who encourages her to get in and out of the shower to a situation where she did but took some time.

As for locomotion, the statistically significant difference was found in "stairs". Here the elderly improved their ability to go up and down stairs (p = 0.014). In this sub-level "stairs", the elderly passed from a "supervision or preparation" condition in which they only climbed a flight of stairs in the presence of another person, because only then did they feel safe, for a condition of "modified independence", that is , they already ascend the flight of stairs without the presence of another person and, mostly use the handrail or, from a situation of "modified independence" to a condition of "complete independence", that is, they climb the flight of stairs without be necessary use the handrail.

Analyzing the obtained results, it was verified that in the control group there were no statistically significant differences between the moments of evaluation, that is, this group of elderly remained functional independence during the period of the study, while the experimental group improved in some sub levels to their F.I. and kept the others. This study allowed us to verify that participation in a P.E.P. allows maintaining and / or improving components essential to the F.I. of the institutionalized elderly person.

Similar to our study, Rocha et al. (2017) demonstrated in their study that the elderly in the experimental group showed significant improvements in functional capacity compared to the elderly in the control group. These authors performed an experimental study with elderly women, divided into two groups - experimental group and control group. The elderly of the experimental group underwent a training program of muscle strength and cardiovascular endurance for 20 weeks. Muscle strength training was performed three times a week, three sets of 8 to 10 repetitions, with a range of 1 to 2 minutes between sets and exercises, lasting 30 minutes, using a load of 70% to 85% of 1 RM. For cardiovascular training they used the step, with a frequency of 3 times a week, for thirty minutes, at an intensity between 70% and 89% of the reserve heart rate. Comparing the groups, it was verified that the elderly women in the experimental group showed a significant improvement in the instrumental

activities of daily living according to the lawton scale (p = 0.07), while the elderly women in the control group had a significant reduction in functional capacity according to the Katz scale (p <0.01).
Nogueira (2018) also carried out a study with 23 elderly people aged between 65 and 94 years, where they participated in a P.E.P. for 12 weeks, performed 3 times a week, each session lasting 60 minutes. The elderly practiced muscular strength training - 3 sets of 8 repetitions, with intensity of 50% of 1 RM in the first 6 weeks and 70% of 1 RM in the following weeks, with 1 minute interval between sets and exercises. At the end of the program, there were no statistically significant differences in their ability to perform self-care - walking, transferring, turning, elevating, toilet use, feeding, dressing and undressing however the values obtained showed a slight tendency for improvement.
Also Tiggemann et al. (2016) applied a muscle strength training program to women aged 60-75 years, which included 6 exercises for 12 weeks at an intensity of 13 to 18 according to Borg's subjective perception of effort scale. In the end, the authors found that participants had improved their functional performance only in the "step up" test.
Regarding sphincter control, communication and social cognition, there were no statistically significant differences between the two moments of evaluation.
Regarding sphincter control, the study is in agreement with the literature. According to Cândido et al. (2017) to improve urinary continence through exercise should be performed specific exercises for the pelvic floor muscles, as well as to improve bowel continence according to Ferreira et al (2012) through physical exercise it is also necessary to train the muscles of the pavement pelvic However, our study did not include the training of these muscles.
As for the communication, our study did not bring benefits to the participants, only allowed to maintain them. Training in group exercise, according to Bentancourt (2008), will promote social contacts and, consequently, may improve the ability to communicate, namely that the person is able to understand and express himself better. However, in our study, although the elderly performed the exercises in groups, each element performed them alone, there being no mutual interaction between the elements, which may be the reason for not obtaining gains at this level.
Also in the social cognition component there were no improvements, the program only allowed to maintain them (p = 0.012). With aging, changes in brain function occur. Short-term memory (the process used to acquire and retain new information) decreases with advancing age; however, long-term memory (knowledge acquired over the years) is little changed. According to Santos et al (2006), the elderly who participate in P.E.F., or other groups that require interaction between them, cognitive decline tends to be smaller.
Fernandez-Mayoralas et al (2015) carried out a study in long-term care institutions for the elderly, in which the elderly residents in these institutions were subdivided into three groups: active, moderately active and inactive. The authors concluded that moderately active elderly people with a certain degree of deterioration of physical and / or mental health are the ones who most benefit from physical activity programs that promote physical and mental health, thus delaying the decline cognitive impairment. In our study, the elderly did not present cognitive deficits, which may be the reason for no improvement at this level.

CONCLUSIONS

The present study shows that the P.E.P practiced by elderly residents in a residential structure for the elderly was an added value for them, since there were positive changes for some levels evaluated. Comparing this group to another group with similar characteristics, it was verified that the latter did not suffer alterations in the evaluated components.
Aging with family members is becoming a less common reality, with Third Age households playing an increasingly important role in this phase of people's lives. However, to combat the sedentary lifestyle of the elderly population, a feature widely observed in these institutions. In this dynamic, the practice of physical exercise was not only a way to combat sedentarism, but a means to improve functional independence.
Maintaining the physical and cognitive functionality is essential for the elderly, so that they can self-care, move in the environment and relate to other people, because all these attitudes are essential for achieving good quality of life.
However, the reduced size of the sample does not allow for generalization of results for populations with similar characteristics, and may overestimate the results. Future studies should include a larger sample.
Another limitation was the fact that the program did not bring benefits to the dimensions of communication and social cognition. In this dynamic, future studies should include the interaction between the group's elements in order to promote leisure and socialization.
Finally, there were few studies with institutionalized elderly people who participated in a physical exercise program training more than one component of physical fitness, just as none used a program that was instituted by us, which made it difficult to compare the results.

CONFLICTS OF INTEREST

I declare that there are no conflicts of interest.

Mendes, C. (2019). Promotion of functional independence institutionalized elderly.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

ACKNOWLEDGEMENT

I thank the institution where the study was made - D. Fernanda Marques Foundation, as well as the entire team that collaborated with me in the preparation of this work.

REFERENCES

Barros, T., Santos, A., Gonzaga, J., Lisboa, M., & Brand, C. (2016). Capacidade funcional de idosos institucionalizados: revisão integrativa. *ABCS HEALTH SCIENCES,* 41(3), 176-180. doi:10.7322/abcshs.v41i3.908

Betancourt, C. (2008). Evaluación funcional y anciano frágil. *Revista Cubana de Enfermería*, *24*(2), Retrieved from http://scielo.sld.cu/scielo.php?script=sci_arttext&pid=S0864-03192008000200005&lng=es&tlng=en.

Borges, L., Meurer, S., Borges, R., Gerage, A., & Benedetti, T. (2015). What is the intensity of exercise sessions of a physical exercise program for older adults? *Revista Brasileira de Cineantropometris e Desempenho Humano*, 17(5), 527-538. doi:10.5007/1980-0037.2015v17n5p527

Breet, L., Traynor, V., & Stapley, P. (2015). Effects of physical exercise on health and well-being of individuals living with a demencia in nursing homes: a systematic review. *Journal of the American Medical Directors Associaion*, 17(2), 104-116.

Cândido, F., Matnei, T., Galvão, L., Santos, V., Santos, M., Sarris, A., & Sobreiro, B. (2017). Incontinência urinária em mulheres: breve revisão de fisiopatologia, avaliação e tratamento. Visão Acadêmica, 18(3), 67-80.

Carneiro, F., Brasileiro, I., Vasconcelos, T., Arruda, V., Florêncio, R., & Moreira, T. (2012). Independência funcional de idosas residentes em instituições de longa permanência. *Acta Fisiat,* 19(3), 156-160.

Castro, M., Lima, L., & Duarte, E. (2016). Jogos recreativos para a terceira idade: uma análise a partir da percepção dos idosos. *Revista Brasleira de Ciências do Esporte,* 38(3), 283-289.

Chow, H., Chen, H., & Lin, L. (2014). Association between out-of-homes trips and older adults functional fitness. *Geriatrics Gerontology Internacional*, 14, 596-604.

Crocker, T., Young, J., Forster, A., Brown, L., Ozer, S., & Greenwood, D. (2013). The effects of physical rehabilitation on activities of daily living in older residents of long-term care facilities: systematic review with meta-analysis. *Age and Ageing,* 42, 682-688.

Fairhall, N.J., Sherrington, C., & Cameron, I. (2013). Mobility training for increasing and functioning in older people with frailty. *Cochrane Database of Systematic Reviews,* 5, 1-13. doi:10.1002/14651858.CD010494

Fernández-Mayoralas, G., Pérez, F., Martínez-Martín, P., Prieto-Flores, M., Rodríguez-Blázquez, C., Martín-García, S., Rojo-Abuín, J., & Forjaz, M. (2015). Active ageing and quality of life: factors associated with participation in leisure activities among institutionalized older adults, with and without dementia. Aging & Mental Health, 19(11), 1031-1041. doi: 10.1080/13607863.2014.996734

Ferreira, L., Marino, L. & Cavenaghi, S. (2012). Intervenção fisioterapêutica na incontinência fecal no idoso. *Arquivos Brasileiros de Ciências da Saúde*, 37(3), 168-172.

Gonçalves, L.H., Silva, A.H., Mazo, G.Z., Benedetti, T.R., Santos, S.M., Marques, S., … Rezende, T. (2010). O idoso institucionalizado: avaliação da capacidade funcional e aptidão física. *Cad. Saúde Pública*, 26(9), 1738-1746.

Kim, P.S., Mayhem, J., & Peterson, D. (2002). A modified YMCA bench press test as a predictor of 1 repetition maximum bench press strength. *Journal of strength and conditioning*, 16(3), 440-445.

Kolbe-Alexander, T., Pacheco, K., Tomaz, S., Karpul, D., & Lambert, E. (2015). The relationship between the built environment and habitual levels of physical activity in South African older adults: a pilot study. *BMC Public Health*, 15, 518-527. doi.10.1186/s12889-015-1853-8

Laíns, J. (1991). *Guia para o uso do Banco Uniformizado de Dados para Reabilitação Médica – MIF. Versão Portuguesa de Guide for the Uniform Data System for Medical Rehabilitation, Adult FIM (1984).* Produção Sandoz.

Maroco, J., & Garcia-Marques, T. (2006). Qual a fiabilidade do alfa de Cronbach? Questões antigas e soluções modernas? *Laboratório de Psicologia,* 4(1), 65-90.

Martins, E., Fernandes, R., & Mendes, F. (2017). Bem-estar subjetivo e atividade física em pessoas idosas. *Millenium*, 2(ed espec nº2), 65-72.

Nogueira, I. (2017). *Impacte de um programa de exercício físico na capacidade funcional de idosos institucionalizados.* (Dissertação de Mestrado, Escola Superior de Enfermagem do Porto). Retrieved from https://comum.rcaap.pt/bitstream/10400.26/21630

Park, S.H., Han, K., & Kang, C., (2014). Effects of exercise programs on depressive symptoms, quality of life, and self-esteem in older people: A systematic review of randomized controlled trials. *Applied Nursing Research*, 27(4), 219-226.

Pordata (2014). *Indicadores de envelhecimento segundo os Censos em Portugal.* Retrieved from http://www.pordata.pt/Portugal/Indicadores+de+envelhecimento+segundo+os+censos++-525

Rocha, C., Guimarães, A., Borba-Pinheiro, C., Santos, C., Moreira, M., Mello, D., & Dantas, E. (2017). Efeito de 20 semanas de treinamento combinado na capacidade funcional de idosas. *Revista Brasileira de Ciências do Esporte,* 39(4), 442-449.

Santos, C., Rossetti, C., & Ortega, A. (2006). O funcionamento cognitvo de idosos e de adolescentes num contexto de jogos e regras. *Estudos Interdisciplinares sobre envelhecimento*, 9(3),53-74.

Tiggemann, C., Dias, C., Radaelli, R., Massa, J., Bortoluzzi, R., Schoenell, M., Noll, M., Alberson, C., & Kruel, L. (2016). Effect of traditional resistance and power training using rated perceived exertion for enhancement of muscle strenght, power, and functional performance. *Official Jornal of the American Aging Association*, 38(42), 1-12.

CIÊNCIAS DA VIDA E DA SAÚDE
LIFE AND HEALTH SCIENCES
CIENCIAS DE LA VIDA Y LA SALUD

millenium 103

Millenium, 2(8), 103-111.

pt

PROMOÇÃO DA INDEPENDÊNCIA FUNCIONAL EM IDOSOS INSTITUCIONALIZADOS

PROMOTION OF FUNCTIONAL INDEPENDENCE INSTITUTIONALIZED ELDERLY

PROMOCIÓN DE LA INDEPENDENCIA FUNCIONAL EN ANCIANOS INSTITUCIONALIZADOS

Carla Mendes[1]

[1] *Centro Hospitalar Médio Tejo, EPE - Hospital Nossa Senhora da Graça, Tomar, Portugal*

Carla Mendes - carlmamendes@hotmail.com

Autor Correspondente
Carla Mendes
Centro Hospitalar do Médio Tejo, E.P.E
Av. Maria de Lourdes de Mello Castro – Ap. 118
2304-909 Tomar
carlmamendes@hotmail.com

RECEBIDO: 11 de dezembro de 2018
ACEITE: 21 de janeiro de 2019

Mendes, C. (2019). Promoção da independência funcional em idosos institucionalizados.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

RESUMO

Introdução: Com o envelhecimento verifica-se um declínio das funções físicas e cognitivas da pessoa idosa, o que leva ao comprometimento da sua independência funcional (F.I.).
Objetivo: Comparar a independência funcional de dois grupos de idosos com características inicialmente semelhantes, residentes numa Estrutura Residencial para Idosos, sedentários, sendo um dos grupos submetido a um programa de exercício físico (P.E.P.).
Métodos: Estudo quantitativo, de natureza quase-experimental. Recorreu-se a uma amostra de 20 idosos divididos em dois grupos semelhantes, experimental e controlo, de dez elementos cada. Um dos grupos praticou um P.E.P., que incluiu treino de força muscular e treino aeróbio, durante 12 semanas, 5 dias por semana, a uma intensidade moderada. Utilizou-se a Medida de Independência Funcional (FIM) para avaliar a independência funcional dos idosos num primeiro momento e, posteriormente num segundo momento, isto é, após a implementação do P.E.P. ao grupo experimental. A análise dos dados foi efetuada com recurso à estatística descritiva e inferencial, nomeadamente com o teste exato de Fisher, Mann-Whitney e Wilcoxon, considerando $p<0,05$.
Resultados: No final do estudo o grupo experimental melhorou a sua independência funcional nos subníveis "banho", "banheira e duche" e "escadas", mantendo todos os outros, enquanto o grupo controlo manteve a independência funcional em todos os subníveis avaliados.
Conclusões: A prática de exercício físico permite manter e/ou melhorar a independência funcional dos idosos.

Palavras-chave: idoso; exercício físico; independência.

ABSTRACT

Introduction: With aging there is a decline in the physical and cognitive function of the elderly, which leads to impairment of the Functional Independence (F.I.) of the elderly person.
Objective: To compare functional independence of two elderly groups with initially similar characteristics, living in a residential structure for the elderly, one of the groups being submitted to a Physical Exercise Program (P.E.P.)
Method: Quantitative, quasi-experimental study. A sample of 20 elderly people divided into two similar groups, experimental and control, of ten elements each. One of the groups practiced P.E.P., which included muscle strength training and aerobic training for 12 weeks, 5 days a week, at moderate intensity. The Functional Independence Measure (FIM) was used to evaluate the functional independence of the elderly at first, and then in second moment, that is, after the implementation of P.E.P. to the experimental group. Data analysis was performed using descriptive and inferential statistics, namely with Fisher's exact test, Mann-Whitney and Wilcoxon, considering $p <0.05$.
Results: At the end of the study the experimental group improved its functional independence in the "bath", "bath and shower" and "stairs" sub-levels, while maintaining all others, while the control group maintained functional independence in all evaluated sub-levels.
Conclusion: The practice of physical exercise allows to maintain and/or improve functional independence of the elderly.

Keywords: elderly; physical exercise; independence.

RESUMEN

Introducción: Con el envejecimiento se verifica un declive de las funciones físicas y cognitivas de la persona mayor, lo que lleva al compromiso de la independencia funcional (F.I.) de la misma.
Objetivos: Comparar la independencia funcional de dos grupos de ancianos con características inicialmente similares, residentes en una estrutura residencial para ancianos, sedentarios, siendo uno de los grupos sometido a un programa de ejercicio físico (P.E.P.).
Métodos: Estudio cuantitativo, de naturaleza cuasi-experimental. Se recurrió a una muestra de 20 ancianos divididos en dos grupos similares, experimental y control, de diez elementos cada uno. Uno de los grupos practicó un P.E.P., que incluyó entrenamiento de fuerza muscular y entrenamiento aeróbico, durante 12 semanas, 5 días a la semana, a una intensidad moderada. Se utilizó la Medida de Independencia Funcional (FMI) para evaluar la independencia funcional de los ancianos en un primer momento y, posteriormente en un segundo momento, es decir, después de la implementación del P.E.P. al grupo experimental. El análisis de los datos fue efectuado con recurso a la estadística descriptiva e inferencial, en particular con la prueba exacta de Fisher, Mann-Whitney y Wilcoxon, considerando $p <0,05$.
Resultados: Al final del estudio el grupo experimental mejoró su independencia funcional en los subniveles "baño", "bañera y ducha" y "escaleras", manteniendo a todos los demás, mientras que el grupo control mantuvo la independencia funcional en todos los subniveles evaluados.
Conclusións: La práctica de ejercicio físico permite mantener y/o mejorar la independencia funcional de los ancianos.

Palabras Clave: anciano; ejercicio físico; independencia.

INTRODUÇÃO

O envelhecimento é um processo progressivo, universal e multifatorial, que ocorre de forma heterogênea, isto é, cada pessoa envelhece de uma forma única e particular. É essencialmente na Terceira Idade, a fase onde surgem alterações significativas a nível físico, psíquico e social que condicionam a adaptação da pessoa idosa ao meio ambiente e, por conseguinte, a sua independência funcional e qualidade de vida (Park et al., 2014).

Os avanços alcançados com a revolução industrial alteraram o estilo de vida das pessoas, melhorando a sua qualidade de vida, saúde e, consequentemente a longevidade. Nas últimas décadas verificou-se um aumento da população idosa nos países industrializados e, em Portugal, cerca de 20% da população tem mais de 65 anos (Portada, 2014). A mulher, que até aqui era a tradicional cuidadora dos idosos na família começou a inserir-se no mercado de trabalho (Barros et al., 2016), o que levou uma readaptação social, cultural, económica e, na estrutura familiar.

Assim, à medida que assistimos ao aumento da esperança média de vida, também assistimos ao aumento do número de Estruturas Residenciais para Idosos, estas que assumem a responsabilidade de cuidar de pessoas idosas, dependentes ou independentes, que não têm condições de permanecer com a família ou em suas próprias casas, proporcionando-lhes uma habitação, alimentação, cuidados de saúde e convívio social (Carneiro et al., 2012).

De uma forma geral, nas Estruturas Residenciais para Idosos, as atividades desenvolvidas pelos idosos que lá habitam são geralmente poucas, o que associado ao processo de envelhecimento vai condicionar a sua capacidade funcional. Neste sentido, o sedentarismo comum a todos os idosos leva-os à perda da autonomia e, a uma dificuldade cada vez maior em realizar as tarefas do dia-a-dia (Barros et al., 2016). A fragilidade do idoso é uma situação que se caracteriza pelo declínio dos vários sistemas do corpo e, por conseguinte, as suas capacidades físicas e mentais diminuem, aumentando a vulnerabilidade destas pessoas a problemas de saúde (Fairhall et al., 2013). O declínio funcional é assim um fator a combater nesta faixa etária como meio de promoção da saúde, aumento da sensação de bem-estar, assim como redução dos custos em saúde.

Neste contexto, foi aplicado um programa de exercicio físico a um grupo de idosos residentes numa Estrutura Residencial para Idosos na região centro de Portugal como meio de promover a sua independência funcional. Assim, este estudo visa analisar a independência funcional dos idosos, sedentários, antes e após ocorrer a implementação e a participação num programa de exercicio físico e, posteriormente compará-lo com um outro grupo com características semelhantes mas, que não intregou o programa.

1. ACTIVIDADE FÍSICA E INDEPENDÊNCIA FUNCIONAL NA TERCEIRA IDADE

No último século o aumento da população idosa a nível mundial tem preocupado a sociedade em geral e, os profissionais de saúde em particular, o que tem levado ao desenvolvimento de programas de promoção da saúde e bem-estar para este grupo etário (Crocker et al., 2013; Chow et al., 2014).

Nesta dinâmica, pelos estudos analisados, verificou-se que a participação dos idosos num P.E.F. poderá ser um meio que permite combater o sedentarismo e, deste modo manter a independência do idoso o mais tempo possível. O treino de componentes da aptidão física, como meio de manutenção da autonomia é um fator que exerce um papel positivo relevante ao longo da vida, isto é, conduz a uma redução de doenças não transmissíveis, menor número de quedas, menos custos em saúde, assim como melhoria do autoconceito e da autoestima (Park et al., 2014). A prática de exercício físico é vista como um meio que conduz os idosos á manutenção, ou mesmo melhoria da sua autonomia, assim como uma forma de prevenir doenças físicas e mentais, facilitando a sua adaptação ao meio ambiente (Crocker et al., 2013).

Park et al. (2014) após analisarem vários estudos sobre os efeitos de um P.E.F. sobre os sintomas depressivos, a qualidade de vida e, a autoestima, concluiram que a prática de exercício físico melhorou todas as variáveis estudadas, deste modo os autores recomendam a prática de exercício físico por idosos.

Também Crocker et al. (2013) realizaram um estudo cujo objetivo foi analisar os efeitos de P.E.F. sobre as atividades de vida diária em idosos residentes em Estruturas Residenciais para Idosos. Os autores chegaram à conclusão que a independência dos idosos foi melhorada através dos P.E.F., embora não saibam quais as intervenções mais adequadas a aplicar a este grupo etário.

O estudo de Gonçalves et al. (2010) sobre a relação entre a aptidão física e a capacidade funcional de idosos residentes em Estruturas Residenciais para Idosos, verificando que a I.F. e a aptidão física se encontram relacionadas, pois quanto melhor for a aptidão física (coordenação, força muscular, capacidade aeróbia, agilidade e coordenação) melhor será o desempenho das atividades de vida diária.

O exercício físico consiste no planeamento, estruturação e sistematização dos movimentos corporais executados com o objetivo de manter ou melhorar a capacidade física do idoso (Borges, 2015; Breet et al., 2015).

Um dos fatores importantes para os idosos é a manutenção da sua autonomia (Chow et al., 2014), isto é, a capacidade de se mobilizarem no meio ambiente e de executarem de forma independente as suas atividades básicas de vida diária (Rocha et al., 2017). Neste sentido a procura pela sua saúde, interação social e pelo prazer leva os idosos a participarem em P.E.F. (Castro et al., 2016).

Os programas de exercicio físico podem ser executados treinando uma ou mais componentes da aptidão física, embora o treino

Mendes, C. (2019). Promoção da independência funcional em idosos institucionalizados.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

que envolva mais do que uma componente da aptidão física na mesma sessão seja um método ainda recente quando utilizado para induzir efeitos nas variáveis relacionadas com a saúde e manutenção da independência na população idosa (Rocha et al., 2017).

2. MÉTODOS

Este estudo caracteriza-se como uma pesquisa de natureza quantitativa, de desenho quase-experimental, na medida em que temos dois grupos: um experimental e outro de controlo, constituído cada grupo por 10 elementos, onde o grupo experimental foi submetido a um programa de exercício físico.

2.1 Participantes

Para selecionar os participantes no estudo foi aplicada a MIF a todos os idosos residentes numa Estrutura Residencial para Idosos, inserida no meio rural na região centro de Portugal, os quais deveriam apresentar uma independência completa, independência modificada ou supervisão em cada subnível dos seis níveis representativos – autocuidados; controlo de esfíncteres; mobilidade; locomoção; comunicação; cognição social. Para além disso, deviam ser sedentários (isto é, que não pratiquem exercício físico á pelo menos dois anos) e, que não apresentem doenças, tais como: acidente vascular cerebral, parkinson, distúrbios do equilíbrio corporal, déficit visual que lhe impossibilite de ver, ou condições clínicas não controladas como hipertensão arterial ou diabetes mellitus. Face ao exposto foram selecionados 20 idosos, estes que posteriormente foram repartidos em dois grupos de 10 elementos cada, experimental e controlo, de forma aleatória, por forma a comparar dois grupos inicialmente semelhantes, mas que após a participação de um num P.E.F., no final do estudo podem apresentar características diferentes.
Relativamente à idade, no grupo experimental 40% tinham entre 75 e 80 anos, 30% tinham menos de 75 anos e, 30% mais de 80 anos. A média de idades situou-se nos 77,40 anos. No grupo controlo, 50% tinham entre 75 e 80 anos, 20% menos de 75% e, 30% mais de 80 anos. A média de idades deste grupo situou-se nos 78 anos. A maioria dos elementos são do sexo feminino, 70% no grupo experimental e 60% no grupo controlo. Quanto á escolaridade, 60% no grupo experimental e 50% no grupo controlo não frequentaram a escola, enquanto os restantes elementos possuíam o 1º ciclo do Ensino Básico. Comparando os grupos, não se verificaram diferenças estatisticamente significativas nas características avaliadas - idade (p=0,543), sexo (p=1,000) e escolaridade (p=1,000).

2.2 Instrumentos de recolha de dados

Os participantes foram avaliados pela escala MIF em dois momentos distintos. Num primeiro momento, quando todos os idosos residentes na instituição foram avaliados e, num segundo momento, este que foi após o grupo experimental ter praticado o P.E.F.. É necessário atender que o grupo experimental antes de se dar inicio ao programa de exercício houve a necessidade de se avaliar a força muscular para adequar a carga a utilizar no mesmo, ensinar a executar os movimentos com a técnica correta e, a usar a escala de perceção subjetiva de esforço de Borg (usada para avaliar a tolerância ao esforço).
A MIF é uma escala que foi traduzida a adaptada para a população portuguesa por Lains (1991). Este instrumento tem por finalidade avaliar o que a pessoa na realidade consegue fazer, assim como determina a ajuda que uma pessoa precisa para a realização de tarefas motoras e cognitivas. A MIF permite a avaliação de seis níveis: autocuidado, controlo de esfíncteres, mobilidade, locomoção, comunicação e, cognição social, estes que se encontram subdivididos em 18 subníveis. Cada subnível recebe a pontuação de 1 a 7, que vai da dependência total á independência completa. Cada nível é analisado pela soma dos subníveis que o constituem. O valor total da MIF corresponde á soma do valor dos subníveis e, vai de 18 a 126 pontos.
Procedeu-se à determinação do coeficiente alpha de Cronbach para avaliar a homogeneidade dos itens que constituem cada uma das dimensões da MIF e, de todos os itens no seu conjunto, obtendo-se os valores apresentados na tabela 1. Segundo Maroco e Garcia-Marques (2006) são aceitáveis valores iguais ou superiores a 0,60, principalmente quando o número de itens é reduzido. Assim sendo, pode dizer-se que a consistência interna é aceitável.

Tabela 1 – Coeficientes *alpha de Cronbach* obtidos para a MIF

Níveis	Nº de itens	1ª avaliação	2ª avaliação
Autocuidados	6	0,445	0,528
Controlo de esfíncteres	2	0,176	0,089
Mobilidade	3	0,000	0,000
Locomoção	2	0,375	0,480
Comunicação	2	0,000	0,000
Cognição social	3	0,850	0,828
MIF total	18	0,732	0,782

2.3 Variável dependente e variável independente

A variável dependente é a independência funcional dos idosos, operacionalizada e mensurada pela MIF, nos seguintes itens: Autocuidados (A- alimentação, B-higiene pessoal, C-banho – lavar o corpo, D-vestir metade superior, E-vestir metade inferior, F-utilização da sanita); Controlo de esfíncteres (G-bexiga, H- intestino); Mobilidade (I-leito, cadeira, cadeira de rodas, J-sanita, K-banheiro/ duche); Locomoção (L- marcha/ cadeira de rodas; M- escadas); Comunicação (N- compreensão, O- expressão); Cognição social (P- interação social, Q-resolução dos prolemas, R- memória); MIF total.

A variável independente é o P.E.F. instituído aos idosos. O P.E.F. teve a duração de 12 semanas, tendo sido praticado 5 dias por semana (de segunda-feira a sexta-feira), começando sempre com um aquecimento, passando pela parte fundamental e, terminando com o retorno á calma.

O período de aquecimento teve a duração de 10 minutos, sem intensidade, sendo executadas duas séries de 10 repetições de cada exercício. Este exercício consistiu no treino de movimentos para os membros superiores (circundação; movimentos oscilatórios no sentido ântero-posterior e, cruzar e afastar estes ao nível do peito, na posição ortostática e, a deambular), membros inferiores (marcha, alternar tipo de marcha – rápida e lenta, esta última que por períodos foi acompanhada por movimentos dos membros superiores) e, exercícios de alongamento – alongamento da região lombar e dos isquiotibiais.

A parte fundamental praticada á 2ªf, 4ªf e 6ªf consistiu em treino de força muscular e aeróbio durante 40 minutos. O treino da força muscular foi efetuado com base na avaliação do teste de peso máximo (Kim et al., 2002) de uma repetição máxima (1RM), ou seja, 100% de 1 RM corresponde ao peso máximo que a pessoa consegue manusear ao executar determinado movimento. Com base nesta primeira avaliação foi adequado o peso usado para os idosos executarem os exercícios planeados – inicialmente 60% de 1RM e, posteriormente, a partir da nona semana 70% de 1RM.

Assim, os idosos executaram exercícios de força muscular que foram: "levantar e sentar numa cadeira sem braços, sem utilização das mãos", "flexão da coxa", "elevação lateral dos membros superiores" e, "extensão da coxa", com uma intensidade de 60% a 70% de 1 RM, duas séries de cada exercício, começando com 8 repetições, passando a 10 repetições a partir da nona semana, com um intervalo de repouso de 1 minuto entre as séries, a uma velocidade moderada, o que na escala de perceção subjetiva de esforço de Borg corresponde de 12 a 13 e, posteriormente andaram, adicionando gradualmente movimentos aos membros superiores e, subiram e desceram escadas, com uma intensidade de 12 a 15, segundo a escala de perceção subjetiva de esforço de Borg.

Ás terças e quintas os idosos andaram, inicialmente em terrenos regulares e, posteriormente irregulares (calçada e terreno inclinado), a diferentes velocidades e, subiram e desceram escadas com a duração máxima de 30 minutos.

Por MIF, realizou-se o "retorno á calma", com uma duração de 10 minutos, praticado de segunda-feira a sexta-feira, sem intensidade, esta que começou com uma breve caminhada com a duração de 1 minuto a um ritmo lento e, posteriormente foram efetuados alongamento dos membros superiores, alongamento do tríceps sural e, alongamento do quadricípede, duas séries de 10 repetições, com a duração de 10 a 30 segundos, com um intervalo de 20 segundos entre cada exercício.

2.4 Procedimento ético

O estudo foi aprovado pela instituição onde foi implementado a 5 de Setembro de 2011 e, teve em conta cinco princípios/direitos que permitem proteger os direitos e liberdades das pessoas que nele participaram, que são: direito á autodeterminação, direito á intimidade, direito ao anonimato e á confidencialidade, direito á proteção contra o desconforto e o prejuízo e, direito a um tratamento justo e equitativo. Para além disso, os participantes assinaram o termo de consentimento livre e esclarecido, que comprova o seu conhecimento e autorização para participar no estudo.

Quanto á obtenção dos dados, estes foram colhidos num primeiro momento em Março de 2012 e, posteriormente num segundo momento em Junho de 2012, após o grupo experimental ter praticado o P.E.F..

2.5 Tecnicas de análise de dados e grau de confiança

Recorreu-se ao Statistical Package for the Social Sciences (SPSS) na versão 20,0 para organizar e sistematizar a informação contida nos dados e obter resultados descritivos e inferenciais. Ao longo do estudo foram usadas técnicas de estatística descritiva e estatística inferencial, especificamente medidas de tendência central (média aritmética e mediana) e, testes estatísticos (exato de Fisher, Mann-Whitney e Wilcoxon). Em todos os testes fixou-se o valor 0,050 como limite de significância.

3. RESULTADOS E DISCUSSÃO

Manter a independência funcional, mesmo em idade avançada, é uma condição essencial para o idoso. Neste sentido, na tabela 2 pode-se analisar os valores apresentados pelos elementos do grupo experimental e do grupo controlo nos dois momentos de avaliação.

Mendes, C. (2019). Promoção da independência funcional em idosos institucionalizados.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

Tabela 2 – Valores obtidos no grupo experimental e o no grupo controlo nos dois momentos de avaliação.

		Primeira avaliação		Segunda avaliação		Teste de Wilcoxon
		Média	Mediana	Média	Mediana	
Grupo experimental	Autocuidados	6,63	6,67	6,73	6,83	P=0,014
	Esfíncteres	6,55	7,00	6,55	7,00	P=1,000
	Mobilidade	6,63	6,67	6,87	7,00	P=0,008
	Locomoção	6,25	6,00	6,65	6,50	P=0,011
	Comunicação	6,65	6,75	6,80	7,00	P=0,083
	Cognição social	6,40	6,33	6,53	6,50	P=0,102
	MIF total	6,54	6,53	6,70	6,69	P=0,007
Grupo controlo	Autocuidados	6,58	6,50	6,58	6,50	P=1,000
	Esfíncteres	6,75	7,00	6,60	6,75	P=0,083
	Mobilidade	6,43	6,33	6,40	6,33	P=0,317
	Locomoção	6,35	6,50	6,35	6,50	P=1,000
	Comunicação	6,80	7,00	6,75	7,00	P=0,317
	Cognição social	6,37	6,33	6,37	6,33	P=1,000
	MIF total	6,54	6,53	6,51	6,50	P=0,059

A análise comparativa dos valores obtidos evidencia que, após a participação num P.E.F., os idosos tenderam a revelar maior capacidade para o autocuidado. A comparação entre os dois momentos de avaliação revelou a existência de diferenças estatisticamente significativas entre estes dois momentos (p= 0,014) no grupo experimental. Fazendo o estudo ao nível de cada um dos subníveis que constituem a escala, verificou-se que a diferença estatisticamente significativa ocorreu, unicamente, no nível de "banho" (p= 0,025), tendo os idosos melhorado o seu nível de independência. No entanto, os idosos também melhoraram outro subnível, mas este não se revelou estatisticamente significativo, que foi o autocuidado "vestir a metade inferior do corpo".

Quanto ao autocuidado "banho" os idosos passaram na sua maioria de uma necessidade de supervisão, onde precisavam que lhe preparassem os utensílios de banho, assim como de serem estimulados para lavarem na integra o corpo, do pescoço até aos pés com exclusão das costas, no chuveiro, para uma

situação em que a maioria já não precisava de ser estimulada a executar este autocuidado, mas demorava mais tempo a fazê-lo e, faziam-no numa cadeira com braços. Os idosos passaram assim de uma necessidade de "supervisão ou preparação" para uma necessidade de "independência modificada".

A análise referente ao nível mobilidade, a diferença estatisticamente significativa situa-se no subnível utilização da "banheira, duche" (p=0,001). Nesta capacidade os idosos passaram na sua maioria de uma necessidade de "independência modificada" em que para entrarem e saírem do duche demoravam mais tempo que o normal e não o faziam em total segurança, para uma situação de "independência completa", em que entravam e saiam do duche em segurança. Apenas uma idosa passou de uma situação de "supervisão ou preparação" em que precisava de ser estimulada a entrar e sair do duche e, requeria a presença de outra pessoa para tal, para uma situação de "independência modificada", em que já não precisava de outra pessoa que a incentiva-se a entrar e sair do duche para uma situação em que o fazia mas demorava algum tempo.

Quanto á locomoção verificou-se que a diferença estatisticamente significativa, se situou em "escadas". Aqui os idosos melhoraram a capacidade de subir e descer escadas (p=0,014). Neste subnível "escadas", os idosos passaram de uma condição de "supervisão ou preparação" em que apenas subiam um lance de escadas na presença de outra pessoa, pois só assim se sentiam seguras, para uma condição de "independência modificada", ou seja, já sobem o lance de escadas sem a presença de outra pessoa e, usam na sua maioria o corrimão ou, passaram de uma situação de "independência modificada" para uma condição de "independência completa", isto é, sobem o lance de escadas sem ser necessário usar o corrimão.

Analisando os resultados obtidos, verificou-se que no grupo controlo não se verificaram diferenças estatisticamente significativas entre os momentos de avaliação, ou seja, este grupo de idosos manteve a independência funcional durante o período em que ocorreu o estudo, enquanto o grupo experimental melhorou em alguns subníveis a sua independência funcional e manteve os outros. Este estudo permitiu verificar que a participação num programa de exercício físico permite manter e/ou melhorar componentes essenciais á independência funcional da pessoa idosa institucionalizada.

À semelhança do nosso estudo, também Rocha et al. (2017) demonstraram no seu estudo que as idosas do grupo experimental apresentam melhorias significativas na capacidade funcional, comparativamente ás idosas do grupo controlo. Estes autores realizaram um estudo de natureza experimental com mulheres idosas, divididas em dois grupos – grupo experimental e grupo controle. As idosas do grupo experimental foram submetidas a um programa de treino de força muscular e resistência cardiovascular durante 20 semanas. O treino de força muscular foi efetuado 3 vezes por semana, 3 séries de 8 a 10 repetições,

com intervalo de 1 a 2 minutos entre as séries e os exercícios, com duração de 30 minutos, usando uma carga de 70% a 85% de 1 RM. Para o treino cardiovascular usaram o step, com uma frequência de 3 vezes por semana, durante 30 minutos, a uma intensidade entre 70% a 89% da frequência cardíaca de reserva. Comparando os grupos verificou-se que as idosas do grupo experimental apresentam uma melhoria significativa nas atividades instrumentais de vida diária, segundo a escala de lawton (p=0,07), enquanto as idosas do grupo controlo apresentam uma redução significativa na capacidade funcional segundo a escala de Katz (p<0,01).

Também Nogueira (2018) realizou um estudo com 23 idosos com idades compreendidas entre os 65 e os 94 anos, onde estes participaram num programa de exercício físico durante 12 semanas, efetuado 3 vezes por semana, tendo cada sessão a duração de 60 minutos. Os idosos praticaram treino de força muscular

– 3 séries de 8 repetições, com intensidade de 50% de 1 RM nas primeiras 6 semanas e de 70% de 1 RM nas semanas seguintes, com 1 minuto de intervalo entre as séries e os exercícios. No final do programa não se verificaram diferenças estatisticamente significativas na capacidade dos mesmos para a realização dos autocuidados – andar, transferir-se, virar-se, elevar-se, uso do sanitário, alimentar-se, arranjar-se, vestir-se e despir-se, no entanto os valores obtidos mostraram uma ligeira tendência de melhoria.

Também Tiggemann et al. (2016) aplicaram um programa de treino de força muscular a mulheres com idades entre os 60 e os 75 anos, que incluiu 6 exercícios, durante 12 semanas, a uma intensidade de 13 a 18, segundo a escala de perceção subjetiva de esforço de Borg. No final, os autores verificaram que as participantes tinham melhorado o seu desempenho funcional apenas no teste "subir degrau".

Relativamente ao controlo de esfíncteres, à comunicação e, à cognição social, não se verificaram diferenças estatisticamente significativas entre os dois momentos de avaliação.

Quanto ao controlo de esfíncteres, o estudo vai de encontro á literatura consultada. Segundo Cândido et al. (2017) para melhorar a continência urinária através do exercício devem ser efetuados exercícios específicos para os músculos do pavimento pélvico, assim como, para melhorar a continência intestinal segundo Ferreira et al (2012) através do exercício físico também é necessário treinar os músculos do pavimento pélvico. No entanto o nosso estudo não incluiu o treino destes músculos.

Quanto á comunicação, o nosso estudo não trouxe benefícios aos participantes, apenas permitiu mantê- las. Treinar exercício em grupo, segundo Bentancourt (2008), vai promover os contactos sociais e, consequentemente poderá melhorar a capacidade de comunicar, nomeadamente da pessoa ser capaz de compreender e de se expressar melhor. No entanto, no nosso estudo, apesar dos idosos realizarem os exercícios em grupo cada elemento executou-os por si só, não havendo interação mútua entre os elementos, podendo esta ser a razão da não obtenção de ganhos neste nível.

Na componente cognição social também não se verificaram melhorias, o programa instituído apenas permitiu mantê-las (p=0,012). Com o envelhecimento ocorrem alterações da função cerebral. A memória a curto prazo (processo utilizado para adquirir e reter novas informações) diminui com o avançar da idade, no entanto, a memória a longo prazo (conhecimento adquirido á anos) pouco se altera. Segundo Santos et al (2006) os idosos que participam em P.E.F., ou outros grupos que requeiram interação entre os mesmos, o declínio cognitivo tende a ser menor.

Fernandez-Mayoralas et al (2015) realizaram um estudo em instituições de longa permanência para idosos no qual subdividiram os idosos residentes nestas instituições em 3 grupos: ativos, moderadamente ativos e inativos. Os autores chegaram á conclusão que os idosos moderadamente ativos, onde se encontram idosos com um certo grau de deterioração da saúde física e/ou mental são os que mais beneficiam com programas de atividade física que promovam a saúde física e mental, atrasando assim o declínio físico, mas também o comprometimento cognitivo. No nosso estudo os idosos não apresentavam déficit cognitivo, podendo esta ser a razão de não se verificarem melhorias neste nível.

CONCLUSÕES

O presente estudo mostra que o programa de exercício físico praticado por idosos residentes numa Estrutura Residencial para Idosos foi uma mais-valia para os mesmos, uma vez que se verificaram alterações positivas para alguns níveis avaliados. Comparando este grupo a um outro grupo com características semelhantes, verificou-se que este último não sofreu alterações nas componentes avaliadas.

Envelhecer junto dos familiares é cada vez uma realidade menos comum, passando as Estruturas Residenciais para Idosos a desempenhar um papel cada vez mais relevante nesta fase da vida das pessoas. No entanto, à que combater o sedentarismo da população idosa, característica muito observada nestas instituições. Nesta dinâmica, a prática de exercício físico constituiu não apenas uma maneira de combater o sedentarismo, mas, um meio que permitiu melhorar a independência funcional.

Manter a funcionalidade física e cognitiva é essencial ao idoso, de forma que o mesmo consiga autocuidar- se, movimentar-se no meio ambiente e, relacionar-se com as outras pessoas, pois todas estas atitudes são essenciais para a obtenção de uma boa qualidade de vida.

No entanto, o tamanho reduzido da amostra não permite generalizar os resultados para populações com características idênticas e, além disso, poderá sobrevalorizar os resultados. Estudos futuros deverão incluir uma amostra maior.

Mendes, C. (2019). Promoção da independência funcional em idosos institucionalizados.
Millenium, 2(8), 103-111.
DOI: https://doi.org/10.29352/mill0208.09.00224

Outra limitação foi o facto de o programa não trazer benefícios ás dimensões comunicação e cognição social. Nesta dinâmica, estudos futuros deverão incluir a interação entre os elementos do grupo de forma a promover o lazer e a socialização.
Por MIF, poucos foram os estudos realizados com idosos institucionalizados que participaram num programa de exercício físico treinando mais do que uma componente da aptidão física, assim como nenhum usou um programa semelhante ao por nós instituído, o que dificultou a análise comparativa dos resultados.

CONFLITO DE INTERESSES

Declaro não existirem conflitos de interesse.

AGRADECIMENTOS

Agradeço à instituição onde foi efetuado o estudo – Fundação D. Fernanda Marques, assim como a toda a equipa que colaborou comigo na elaboração deste trabalho.

REFERÊNCIAS BIBLIOGRÁFICAS

Barros, T., Santos, A., Gonzaga, J., Lisboa, M., & Brand, C. (2016). Capacidade funcional de idosos institucionalizados: revisão integrativa. *ABCS HEALTH SCIENCES,* 41(3), 176-180. doi:10.7322/abcshs.v41i3.908

Betancourt, C. (2008). Evaluación funcional y anciano frágil. *Revista Cubana de Enfermería*, *24*(2), Retrieved from http://scielo.sld.cu/scielo.php?script=sci_arttext&pid=S0864- 03192008000200005&lng=es&tlng=en.

Borges, L., Meurer, S., Borges, R., Gerage, A., & Benedetti, T. (2015). What is the intensity of exercise sessions of a physical exercise program for older adults? *Revista Brasileira de Cineantropometris e Desempenho Humano*, 17(5), 527-538. doi:10.5007/1980-0037.2015v17n5p527

Breet, L., Traynor, V., & Stapley, P. (2015). Effects of physical exercise on health and well-being of individuals living with a demencia in nursing homes: a systematic review. *Journal of the American Medical Directors Associaion*, 17(2), 104-116.

Cândido, F., Matnei, T., Galvão, L., Santos, V., Santos, M., Sarris, A., & Sobreiro, B. (2017). Incontinência urinária em mulheres: breve revisão de fisiopatologia, avaliação e tratamento. Visão Acadêmica, 18(3), 67-80.

Carneiro, F., Brasileiro, I., Vasconcelos, T., Arruda, V., Florêncio, R., & Moreira, T. (2012). Independência funcional de idosas residentes em instituições de longa permanência. *Acta Fisiat,* 19(3), 156-160.

Castro, M., Lima, L., & Duarte, E. (2016). Jogos recreativos para a terceira idade: uma análise a partir da percepção dos idosos. *Revista Brasleira de Ciências do Esporte,* 38(3), 283-289.

Chow, H., Chen, H., & Lin, L. (2014). Association between out-of-homes trips and older adults functional fitness. *Geriatrics Gerontology Internacional*, 14, 596-604.

Crocker, T., Young, J., Forster, A., Brown, L., Ozer, S., & Greenwood, D. (2013). The effects of physical rehabilitation on activities of daily living in older residents of long-term care facilities: systematic review with meta-analysis. *Age and Ageing,* 42, 682-688.

Fairhall, N.J., Sherrington, C., & Cameron, I. (2013). Mobility training for increasing and functioning in older people with frailty. *Cochrane Database of Systematic Reviews,* 5, 1-13. doi:10.1002/14651858.CD010494

Fernández-Mayoralas, G., Pérez, F., Martínez-Martín, P., Prieto-Flores, M., Rodríguez-Blázquez, C., Martín- García, S., Rojo-Abuín, J., & Forjaz, M. (2015). Active ageing and quality of life: factors associated with participation in leisure activities among institutionalized older adults, with and without dementia. Aging & Mental Health, 19(11), 1031-1041. doi: 10.1080/13607863.2014.996734

Ferreira, L., Marino, L. & Cavenaghi, S. (2012). Intervenção fisioterapêutica na incontinência fecal no idoso. *Arquivos Brasileiros de Ciências da Saúde*, 37(3), 168-172.

Gonçalves, L.H., Silva, A.H., Mazo, G.Z., Benedetti, T.R., Santos, S.M., Marques, S., … Rezende, T. (2010). O idoso institucionalizado: avaliação da capacidade funcional e aptidão física. *Cad. Saúde Pública*, 26(9), 1738-1746.

Kim, P.S., Mayhem, J., & Peterson, D. (2002). A modified YMCA bench press test as a predictor of 1 repetition maximum bench press strength. *Journal of strength and conditioning*, 16(3), 440-445.

Kolbe-Alexander, T., Pacheco, K., Tomaz, S., Karpul, D., & Lambert, E. (2015). The relationship between the built environment and habitual levels of physical activity in South African older adults: a pilot study. *BMC Public Health*, 15, 518-527. doi.10.1186/s12889-015-1853-8

Laíns, J. (1991). *Guia para o uso do Banco Uniformizado de Dados para Reabilitação Médica – MIF. Versão Portuguesa de Guide for the Uniform Data System for Medical Rehabilitation, Adult MIF (1984).* Produção Sandoz.

Maroco, J., & Garcia-Marques, T. (2006). Qual a fiabilidade do alfa de Cronbach? Questões antigas e soluções modernas? *Laboratório de Psicologia,* 4(1), 65-90.

Martins, E., Fernandes, R., & Mendes, F. (2017). Bem-estar subjetivo e atividade física em pessoas idosas.

Millenium, 2(ed espec nº2), 65-72.

Nogueira, I. (2017). *Impacte de um programa de exercício físico na capacidade funcional de idosos institucionalizados.* (Dissertação de Mestrado, Escola Superior de Enfermagem do Porto). Retrieved from https://comum.rcaap.pt/bitstream/10400.26/21630

Park, S.H., Han, K., & Kang, C., (2014). Effects of exercise programs on depressive symptoms, quality of life, and self-esteem in older people: A systematic review of randomized controlled trials. *Applied Nursing Research*, 27(4), 219-226.

Pordata (2014). *Indicadores de envelhecimento segundo os Censos em Portugal.* Retrieved from http://www.pordata.pt/Portugal/Indicadores+de+envelhecimento+segundo+os+censos++-525

Rocha, C., Guimarães, A., Borba-Pinheiro, C., Santos, C., Moreira, M., Mello, D., & Dantas, E. (2017). Efeito de 20 semanas de treinamento combinado na capacidade funcional de idosas. *Revista Brasileira de Ciências do Esporte,* 39(4), 442-449.

Santos, C., Rossetti, C., & Ortega, A. (2006). O funcionamento cognitvo de idosos e de adolescentes num contexto de jogos e regras. *Estudos Interdisciplinares sobre envelhecimento*, 9(3),53-74.

Tiggemann, C., Dias, C., Radaelli, R., Massa, J., Bortoluzzi, R., Schoenell, M., Noll, M., Alberson, C., & Kruel,

L. (2016). Effect of traditional resistance and power training using rated perceived exertion for enhancement of muscle strenght, power, and functional performance. *Official Jornal of the American Aging Association*, 38(42), 1-12.

millenium
Journal of Education, Technologies, and Health

Política de submissão de artigos à Revista Millenium
A revista Millenium está aberta à colaboração de todos os interessados e aceita continuamente a submissão de artigos.
Os autores devem submeter os manuscritos para publicação no site da Millenium, devendo, contudo, observar as indicações para colaboration, designadamente: Condições de submissão; Instruções de preparação dos manuscritos; Licença Creative Commons.
Documentos necessários à submissão, disponíveis no site da Revista: http://revistas.rcaap.pt/millenium/about/submissions

Article submission policy to Millenium Journal
Millenium Journal is open to the collaboration of all interested parties and continually accepts the submission of articles.
Authors must submit manuscripts for publication on Millenium's website, however, they should observe the collaboration indications, namely: Conditions of submission; Instructions for preparing the manuscripts; License Creative Commons.
Documents required for submission, available on the website of the journal: http://revistas.rcaap.pt/millenium/about/submissions

Politica de sumisión de artículos a la Revista Millenium
La revista Millenium está abierta a la colaboración de todos los interesados y acepta continuamente la sumisión de artículos.
Los autores deben someter los manuscritos para su publicación en el sitio web de Millenium, pero deben observar las indicaciones para colaboración, en particular: Condiciones de envío; Instrucciones de preparación de los manuscritos; Licencia Creative Commons.
Los documentos necesarios para la presentación, disponibles en el sitio de la Revista: http://revistas.rcaap.es/millenium/about/submissions

millenium
Journal of Education, Technologies, and Health

millenium

Journal of Education, Technologies, and Health

Revisores Nacionais Externos| External National Reviewers | Revisores Nacionales Externos

Adalberto Dias de Carvalho – FLUP (PT)
Aires Pereira do Couto – Universidade Católica Portuguesa, Viseu (PT)
Alexandra Maria Dantas de Castro Araújo – U. Portucalense Inf. D. Henrique - Porto (PT)
Ana Maria Frias - Universidade de Évora (PT)
Ana Maria Mouraz Lopes – Universidade do Porto (PT)
Ana Sofia Carvalho – Universidade Católica, Porto (PT)
Anabela Antunes - Centro Hospitalar Tondela Viseu (PT)
Ândrea Marques - Centro Hospitalar da Universidade de Coimbra (PT)
António Boleto Rosado – Universidade Técnica de Lisboa (PT)
António Gomes Ferreira – Universidade de Coimbra (PT)
Cândida Koch - Escola Superior de Enfermagem do Porto (PT)
Carlinda Leite – Universidade do Porto (PT)
Carlos Fernandes Silva – Universidade de Aveiro (PT)
Carlos Duarte Peixeira Marques - Universidade de Trás-os-Montes e Alto Douro (PT)
Célia dos Prazeres Ribeiro – Universidade Católica Portuguesa, Viseu (PT)
Cristina Paula Albuquerque - Centro Hospitalar Tondela Viseu, Viseu (PT)
Eduardo José Ferreira dos Santos - Centro Hospitalar da Universidade de Coimbra (PT)
Elisabete Esteves - Centro Hospitalar da Universidade de Coimbra (PT)
Fernando Pina - Centro Hospitalar Tondela Viseu (PT)
Flávio Nelson Fernandes Reis - Universidade de Coimbra (PT)
Francisco Rui Cádima – Universidade Nova de Lisboa (PT)
Goreti Maria dos Anjos Botelho - Instituto Politécnico de Coimbra (PT)
Gustavo Pires – Universidade Técnica de Lisboa (PT)
Isa Margarida Vitória Severino – Instituto Politécnico da Guarda (PT)
Isabel Cabrita – Universidade de Aveiro (PT)
Isabel Maria Marques Alberto – Universidade de Coimbra (PT)
Isabel Mesquita – Universidade do Porto (PT)
Isabel Vieira - Universidade de Aveiro (PT)
João Carlos Matias Celestino Gomes da Rocha - Universidade de Aveiro (PT)
João Eduardo Quintela Varajão – Universidade de Trás-os-Montes e Alto Douro UTAD (PT)
Jorge Adelino Rodrigues da Costa – Universidade de Aveiro (PT)
Jorge Manuel Rodrigues Bonito - Universidade de Évora (PT)
Jorge Trinidad Ferraz de Abreu – Universidade de Aveiro (PT)
José Carlos Rodrigues Gomes - Instituto Politécnico de Leiria (PT)
José Roquette – Universidade Técnica de Lisboa (PT)
Luís Amaral – Universidade do Minho (PT)
Luís Lopes - INEM (PT)
Manuel António Brites Salgado – Instituto Politécnico da Guarda (PT)
Manuel Celestino Vara Pires - Instituto Politécnico de Bragança (PT)
Manuel Vicente de Freitas Martins – Instituto Politécnico de Castelo Branco (PT)
Margarida Gomes Moldão Martins (PT)
Margarida Isabel dos Santos Amaral – Universidade de Aveiro (PT)
Margarida Vieira - Universidade Católica Portuguesa (PT)
Maria dos Anjos Coelho Rodrigues - Instituto Politécnico de Leiria. Escola Superior de Saúde (PT)
Maria dos Anjos Pires – Universidade de Trás-os-Montes e Alto Douro UTAD (PT)
Maria Elisabete da Silva Tomé Mendes – Instituto Politécnico de Portalegre (PT)
Maria João Barroca – Instituto Politécnico de Coimbra (PT)
Maria Margarida Silva Reis Santos Ferreira - Escola Superior de Enfermagem, Porto (PT)
Maria Neto da Cruz Leitão – Escola Superior de Enfermagem de Coimbra (PT)
Maria Raquel Freire - Universidade de Coimbra (PT)
Maria Teresa Pires de Medeiros - Universidade dos Açores – (PT)
Maria Teresa Rijo da Fonseca Lino – Universidade Nova de Lisboa (PT)
Mauro Lopes Mota - ULS, Guarda, Hospital de Seia (PT)
Nádia Paiva - Sonae Arauco. R&D Manager - Chemicals & Impregnation (PT)
Nuno Marques - INEM (PT)
Paulo Joaquim Pina Queirós – Escola Superior de Enfermagem de Coimbra (PT)
Paulo Jorge Almeida Pereira - Universidade Católica Portuguesa, Viseu (PT)
Paula Prata - Escola Superior de Enfermagem, Porto (PT)
Paulo Providência – Universidade de Coimbra (PT)
Pedro Sousa - Instituto Politécnico de Leiria (PT)
Preciosa Teixeira Fernandes – Universidade do Porto (PT)

millenium

Journal of Education, Technologies, and Health

Revisores Nacionais Externos ad hoc | External National Reviewers ad hoc | Revisores Nacionales Externos ad hoc

Millenium, 2(8) - 2019

Ana Figueira, Universidade do Algarve, Faro, Portugal
António Pirra, Universidade de Trás-os-Montes e Alto Douro, Vila Real, Portugal
Armando Ramalho, Instituto Politécnico de Castelo Branco, Castelo Branco, Portugal
Cristovão Silva, Universidade de Coimbra, Coimbra, Portugal
Elsa Ramalhosa, Instituto Politécnico Bragança, Bragança, Portugal
Fernando Gonçalves, ESAV-IPV, Viseu, Portugal
Inga Ciprovica, Faculty of Food Technology of Latvia, University of Agriculture, Jelgava, Latvia, Letónia
Isabel Brás, ESTGV-IPV, Viseu Portugal
Ivone Ferreira, Faculdade de Ciências Sociais e Humanas - UNL, Lisboa, Portugal
Jaime Anibal, Universidade do Algarve, Faro, Portugal
Joana Martins, ESEV-IPV, Viseu, Portugal
José Pereira, ESAV-IPV, Viseu, Portugal
Maria Manuela Guerra, Escola Superior de Hotelaria e Turismo do Estoril, Estoril, Portugal
Nuno Alvarenga, Instituto Nacional de Investigação Agrária e Veterinária, Oeiras, Portugal
Ofélia Anjos, Instituto Politécnico de Castelo Branco, Castelo Branco, Portugal
Olivério Ribeiro, ESSV-IPV, Viseu, Portugal
Paulo Vaz, ESTGV-IPV, Viseu Portugal
Serafim Oliveira, ESTGV-IPV, Viseu Portugal
Vitor Manuel Rodrigues, Universidade de Trás-os-Montes e Alto Douro, Vila Real, Portugal

millenium
Journal of Education, Technologies, and Health

Revisores Nacionais - Instituto Politécnico de Viseu (IPV) | National Reviewers (IPV) | Revisores Nacionales (IPV)

Escola Superior Agrária
António Manuel Santos Tomas Jordão
Dulcineia Ferreira Wessel
Edite Maria Relvas das Neves Teixeira de Lemos
Helder Filipe dos Santos Viana
Helena Maria Vala Correia
Maria João Cunha Silva Reis Lima
Pedro Rodrigues
Raquel de Pinho Ferreira Guiné
Vitor João Pereira Domingues Martinho

Escola Superior de Educação
Abel Aurélio Abreu de Figueiredo
Ana Isabel Pereira Pinheiro da Silva
Ana Maria Marques Costa Pereira Lopes
Ana Paula Pereira Oliveira Cardoso
Anabela Clara Barreto Marques Novais
Antonino Manuel de Almeida Pereira
António Augusto Gaspar Ribeiro
António Manuel Tavares Azevedo
Belmiro Tavares da Silva Rego
Cátia Clara Ávila Magalhães
Cristina Azevedo Gomes
Dulce Helena Melão
Emília da Conceição Figueiredo Martins
Esperança do Rosário Jales Ribeiro
Filomena Antunes Sobral
Francisco Emiliano Dias Mendes
Henrique Manuel Pereira Ramalho
Isabel Aires de Matos
Ivone Ferreira
Joana Martins
João Paulo Rodrigues Balula
José Luís Menezes Correia
Lia João de Pinho Araújo
Maria Isabel Rola Rodrigues Abrantes
Maria Pacheco Figueiredo
Maria Paula Martins de Oliveira Carvalho
Paula Maria de Azevedo Ferreira Rodrigues
Rosina Inês Ribeiro de Sá Fernandes
Sara Maria Alexandre e Silva Felizardo
Susana Barros Fonseca
Susana Cristina Santos Fidalgo Fonseca Moura Lopes
Véronique Delplancq

Escola Superior de Saúde
Amadeu Matos Gonçalves
Amarílis Pereira Rocha
Ana Isabel Andrade
António Madureira Dias
Carla Maria Viegas e Melo Cruz
Carlos Manuel Figueiredo Pereira
Carlos Manuel de Sousa Albuquerque
Cláudia Margarida C. Balula Chaves
Daniel Marques da Silva
Emília de Carvalho Coutinho
Ernestina Maria Veríssimo Batoca Silva
João Carvalho Duarte
José dos Santos Costa
Lídia do Rosário Cabral
Manuela Maria Conceição Ferreira
Maria Conceição Almeida Martins
Maria da Graça F. Aparício Costa
Maria Isabel Bica de Carvalho
Maria Odete Pereira Amaral
Olivério de Paiva Ribeiro
Paula Alexandra de Andrade B. Nelas
Rosa Maria Lopes Martins
Susana Maria Fernandes S. André
Sofia Campos Pires

Escola Superior de Tecnologia e Gestão de Lamego
Ana Teresa Guia
Isabel Maria Soares Pinto de Oliveira
José Paulo Ferreira Lousado
Paula Alexandra Marques dos Santos

Escola Superior de Tecnologia e Gestão de Viseu
Alexandre David Aibeo Fernandes
Ana Cristina Bico Rodrigues de Matos
António Manuel Pereira Ferrolho
António Pedro Martins Soares Pinto
Bruno Emanuel Morgado Ferreira
Bruno Miguel Morais Lemos Esteves
Carla Manuela Ribeiro Henriques
Carla Maria Alves da Silva
Cláudia Patrícia de Almeida Seabra Moreira
Cristina Maria do Amaral Pereira de Lima Coelho
Daniel Filipe Albuquerque
Gilberto Antunes Ferreira Rouxinol
Henrique Almeida
Idalina de Jesus Domingos
Isabel Maria Loureiro Pais Esteves Martins
Isabel Maria Soares Pinto de Oliveira
Isabel Paula Lopes Brás
Joaquim Goncalves Antunes
José Francisco Monteiro Morgado
José Vicente Rodrigues Ferreira
Luísa Maria Hora de Carvalho
Luísa Paula Goncalves Oliveira Valente da Cruz Lopes
Manuel António Pinto da Silva Amaral
Maria de Lurdes Costa e Sousa
Maria Madalena de Freitas Malva
Nuno Melão
Odete Paiva
Paulo Alexandre da Silveira Costeira Marques da Silva
Paulo Moisés Almeida da Costa
Paulo Rogério Perfeito Tome
Pedro Manuel Nogueira Reis
Suzanne Amaro
Sérgio Miguel Gomes Lopes

millenium
Journal of Education, Technologies, and Health

UNIDADES TÉCNICAS | TECHNICAL UNITS | UNIDADES TÉCNICAS

Unidade Técnica de Supervisão Estatística e Línguas | Statistics and Languages Technical Unit Supervision | Unidad Técnica de Supervisión de Estadísticas y Lenguas

Revisores da Estatística | Statistical Reviewers | Revisores de Estadísticas

Ana Cristina Matos – ESTGV, IPV
António Madureira Dias – ESSV, IPV
Carla Henriques – ESTGV, IPV
Carlota Lemos – ESAV, IPV
Emília Martins – ESEV, IPV
Francisco Emiliano – ESEV, IPV
João Carlos Gonçalves – ESAV, IPV
João Carvalho Duarte – ESSV, IPV
Madalena Malva – ESTGV, IPV
Ricardo Gama – ESTGL, IPV

Revisores dos Idiomas | Languages Reviewers | Revisores de Idiomas

Português | Portuguese | Portugués

Ana Isabel Silva – IPV, ESEV
Dulce Melão – IPV, ESEV
Isabel Aires de Matos – IPV, ESEV
João Paulo Balula – IPV, ESEV

Inglês | English | Inglés

Ana Maria Costa – IPV, ESEV
Susana Fidalgo – IPV, ESEV
Susana Amante – IPV, ESEV

Francês | French | Francés

Ana Maria Oliveira – IPV
Véronique Delplancq – ESEV, IPV

Castelhano | Spanish | Español

António Ferreira – IPV, ESTGV
Paula Fonseca – IPV, ESTGV

Unidade Técnica de Redação, Edição e Documentação | Redaction Technical Unit, Publishing and Documentation | Unidad Técnica de Redacción, Publicaciones y Documentación

Edição e Gestão da Revista Millenium no SARC/RECAAP | Millenium Magazine Edition and Management in SARC/ RECAAP | Edition y Gestión de la Magazine Millenium en SARC / RCAAP

Ângelo Fonseca – ESEV

Apoio Documental e Bibliográfico | Documental and Bibliographical Support | Soporte Documental y Bibliográfico

Ascensão Abrantes – ESEV, IPV
Damiana Guedes – ESTGL, IPV
Fátima Jorge – ESSV, IPV
Luís Carneiro – ESAV, IPV
Rosa Silva – ESTGV , IPV

Edição Internet - Desenvolvimento e manutenção da plataforma da Revista | Internet Edition - Development and magazine platform maintenance | Edición Internet - Desarrollo y mantenimiento de la plataforma de la revista

Ângelo Fonseca – ESEV

Apoio Técnico, Redação e Edição de Texto | Technical Support, Redaction and Text Edition | Soporte Técnico, Redacción y Edición de Texto

Joel Marques – IPV

Composição e Conceção Gráfica | Composition and Graphic Design | Composición y Diseño Gráfico

Paulo Medeiros – IPV
Joel Marques – IPV

fevereiro • february 2019
série | serie 2 • ano | year 4 • quadrimestral | quarterly