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By-products valorisation: Pasta enrichment with olive pomace and coffee silverskin

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Resumo

O crescimento populacional esperado para os próximos anos aliado às alterações climáticas, torna urgente a procura de alternativas alimentares mais sustentáveis que possam garantir alimento para as gerações futuras. Além disso, os consumidores são cada vez mais exigentes e preocupados com a alimentação, levando as indústrias a desenvolver novos produtos alternativos/enriquecidos com ingredientes diferentes. Os subprodutos da agroindústria são ricos em macronutrientes e outras substâncias de interesse como os antioxidantes, podendo ser utilizados como ingredientes no desenvolvimento desses novos produtos.

O principal objetivo deste trabalho foi valorizar dois subprodutos, a pele de prata do café e o bagaço da azeitona, utilizando-os para desenvolver massas alimentícias enriquecidas. Foi realizada a análise nutricional e química das massas produzidas, assim como dos respetivos subprodutos. Foram avaliados os seguintes parâmetros: humidade, gordura, proteína, cinzas, fibra total, solúvel e insolúvel, hidratos de carbono disponíveis, vitamina E, ácidos gordos, fenólicos totais, flavonoides totais e atividade antioxidante. Foi ainda realizada uma análise sensorial relativa à aceitabilidade por parte do consumidor e um teste de preferência às massas alimentícias.

A pele de prata é uma fina camada que se desprende do grão de café durante a torrefação. Os resultados da análise nutricional revelaram que é rica em fibra (≈ 60% fibra total e ≈ 55% fibra insolúvel), proteína (≈ 18%) e minerais totais (≈ 9%). O vitâmero da vitamina E presente em maior quantidade é o α-tocoferol (≈ 25 mg/kg). Os ácidos gordos presentes em maior quantidade são o ácido palmítico (≈ 25%) e o acido beénico (≈ 21%). O bagaço da azeitona, é o resíduo que fica após a produção de azeite. Os resultados da análise nutricional revelaram ser rico em fibra (≈ 55% fibra total e ≈ 51% de fibra insolúvel) e também em gordura (≈ 9%). O principal vitâmero é também o α-tocoferol (≈ 43 mg/kg). O ácido gordo presente em maior quantidade é o ácido oleico (≈ 76%). Ambos os subprodutos, apresentaram atividade antioxidante, mas a pele de prata revelou valores inferiores comparativamente ao bagaço da azeitona.

No entanto, o bagaço da azeitona apresenta um problema físico, a presença do caroço. Neste estudo foi realizado um procedimento teste para a remoção do caroço, que consistiu na secagem do bagaço da azeitona a duas temperaturas diferentes (40ºC por 48h e 70ºC por 24h) com posterior trituração e peneiração. A secagem a 70ºC revelou-se como o melhor processo uma vez que se trata de um processo mais rápido do que a secagem a 40ºC e não apresenta diferenças a nível nutricional e químico que

justifiquem optar por um processo mais demorado. Foi por isso escolhido o bagaço de azeitona seco a 70ºC para ser incorporado na massa.

As massas alimentícias enriquecidas foram desenvolvidas com 7,5% de pele de prata do café e 7,5% de bagaço da azeitona, tendo sido avaliadas cruas e após cozedura. Os resultados revelaram que a adição de pele de prata do café levou ao aumento dos teores de proteína, minerais, fibra total e insolúvel, vitamina E e compostos antioxidantes, nomeadamente fenólicos e flavonoides. A adição de bagaço de azeitona levou ao aumento dos teores de gordura, cinzas, fibra total, solúvel e insolúvel, vitamina E e compostos antioxidantes, nomeadamente fenólicos e flavonoides. Contudo, as propriedades antioxidantes não são um benefício do enriquecimento das massas já que os resultados obtidos demostraram que, após o processo de cozedura, estes compostos sofrem perdas. A incorporação destes subprodutos causou ainda mudanças no perfil e teores de ácidos gordos. Apesar disso, na análise sensorial realizada aos consumidores verificou-se que estes preferiram a massa que não foi enriquecida com nenhum dos subprodutos. Como segunda opção, os consumidores preferiram a massa enriquecida com pele de prata à massa enriquecida com bagaço de azeitona.

Palavras-chave: Subprodutos; Pele de prata do café; Bagaço de azeitona; Massa.

Abstract

The expected population growth for the coming years, combined with climate change, makes it urgent to search for more sustainable food alternatives that can guarantee food security for future generations. In addition, consumers are more demanding and concerned about their food, which leads industries to develop new alternative products or products enriched with different ingredients. Agro-industrial by-products are rich in macronutrients and other substances of interest like antioxidants and can be used as ingredients in the development of these new products.

The main objective of this work was to use two by-products, the coffee silverskin and olive pomace, in the development of enriched pastas. Nutritional and chemical analysis of pasta samples and by-products were carried out. The following parameters were accessed: moisture, fat, protein, ash, total soluble and insoluble fiber, available carbohydrates, vitamin E, fatty acids profile, total phenolics, total flavonoids and antioxidant activity. A sensory analysis on consumer acceptability and a preference test were also carried out on pasta samples.

Coffee silverskin is a thin layer that comes off the grain during roasting. It is rich in fiber (≈ 60% total fiber and ≈ 55% insoluble fiber), protein (≈ 18%) and minerals (≈ 9%). The vitamin E vitamer present in highest concentration is α-tocopherol (≈ 25 mg/kg) and the major fatty acids present in coffee silverskin are palmitic (≈ 25%) and behenic (≈ 21%). Olive pomace is the residue that remains after the olive oil production. It has a high content in fiber (≈ 55% total fiber and ≈ 51% Insoluble fiber) and fat (≈ 9%). The main vitamin E vitamer present is α-tocopherol (≈ 43 mg/kg) and the major fatty acid is oleic acid (≈ 76%). Both by-products showed antioxidant activity but coffee silverskin showed lower results than olive pomace samples for all antioxidant parameters accessed.

However, olive pomace presents a physical problem, the presence of the stone. In this study, a procedure was tested to remove the stone, which consisted of drying the olive pomace at two different temperatures (40ºC for 48h and 70ºC for 24h) with subsequent milling and sieving. The drying procedure at 70ºC turned out to be the best process since it is a faster process and does not present nutritional and chemical differences. Olive pomace dried at 70ºC was chosen to be incorporated into pasta.

Enriched pastas were developed with 7.5% of coffee silverskin or 7.5% of olive pomace. Raw and cooked pastas were analysed. Results revealed that coffee silverskin enrichment led to the increase in protein, ash, total and insoluble fiber, vitamin E and antioxidant compounds as phenolics and flavonoids. Olive pomace enrichment led to the

increase in fat, ash, soluble and insoluble fiber, vitamin E and antioxidant compounds as phenolics and flavonoids. However, antioxidant properties are not a benefit of pasta enrichment with these by-products as they are lost during the cooking procedure. It also led to changes in fatty acids profile and amounts. Despite this, as the sensory analysis results showed, consumers preferred the pasta that was not enriched with any of the by-products. As a second choice, consumers preferred pasta enriched with silverskin to the pasta enriched with olive pomace.

Keywords: By-products; Coffee silverskin; Olive pomace; Pasta.

Publications and communications

Parts of this dissertation were presented in national and international scientific congresses and meetings:

1. Poster presentation in VI Jornadas da Bioquímica, 7-9 of May 2021, online, entitled “By-products valorisation: Processing of olive pomace for incorporation into foodstuffs” - B.C.C. Oliveira, J. Lobo, M.A. Nunes, D. Melo, A.S.G. Costa, R.C. Alves, M.B.P.P. Oliveira.
2. Poster presentation in XX Congress of Food and Nutrition, 23-24 of September 2021, online, entitled “Pasta enrichement with olive pomace: Nutritional advantages” - B.C.C. Oliveira, J. Lobo, M.A. Nunes, A.S.G. Costa, R.C. Alves, M.B.P.P. Oliveira.

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Lista de Abreviaturas e Símbolos

ROS- Reactive oxygen species

DNA- Deoxyribonucleic acid

OTA- Ochratoxin A

GI- Glycaemic index

GF- Gluten-free

TPC-Total phenolics content

TFC- Total flavonoids content

FRAP- Ferric reducing antioxidant power

DPPH- 2,2-diphenyl-1-picrylhydrazyl

OP- Fresh olive pomace

OP40ºC- Olive pomace powder dried at 40ºC

OP70ºC- Olive pomace powder dried at 70ºC

CS- Coffee silverskin

RP- Raw control pasta

CP- Cooked control pasta

RPP- Raw olive pomace (7.5%) pasta

AOAC- Association of Official Analytical Chemists

HPLC- High-performance liquid chromatography

DAD- Diode array detector

FLD- Fluorescence detector

GC- Gas chromatography

FID- Flame ionization detector

UV- Ultraviolet

FC- Folin-Ciocalteu

GAE- Gallic acid equivalents

CE- Catechin equivalents

FSE- Ferrous sulphate equivalents

PUFA- Polyunsaturated fatty acids

MUFA- Monounsaturated fatty acids

SFA- Saturated fatty acids

dw- Dry weight

fw- Fresh weight

TPTZ- 2,4,6-Tris(2-pyridyl)-s-triazine

CHD- Coronary heart disease

1. Introduction

Throughout this introduction, the topics that motivated and sustain this work will be addressed. First, are presented the reasons for the importance of using by-products, which will be described in the chapters 1.1 (Food security and sustainable development), 1.2 (Consumers trends and food product development) and 1.3 (Agro-industrial by-products). After, the by-products used in this work and their obtainment are presented in chapters 1.4 (Olive oil production and olive pomace) and 1.5 (Coffee production and coffee silverskin), as well as the matrix used in this work for their incorporation in chapter 1.6 (Pasta products).

1.1. Food security and sustainable development

Nowadays, we are almost 8 billion people around the world and one in eight of us does not have enough food to eat (Dimick, 2014). According to the United Nations, in 2018, it was estimated that about 821 million people in the world were undernourished, which makes hunger and malnutrition the biggest risks to health worldwide (United Nations). World population is increasing which will lead to a shortage of resources. The United Nations predicts that by the end of the century world's population will be about 11.2 billion, compared to the 7.7 billion on 2019. We will not have enough food, water, energy, and employment for everyone if we do not start to look for alternatives now. The least developed countries will be responsible for the most rapid population growth and this mean, they will struggle to keep up with the rapid changes. As profits in developing countries are continuously rising and living conditions improving, demand for dairy products, fruit, meat, vegetables, and especially innovative food products are growing (Combinator, 2017). In the future, we will have to face many social, economic, and environmental challenges as a consequence of the growth of urban areas (Garcia et al., 2020).

Meanwhile, we are facing significant threats to food security, such as soil erosion, melting of ice caps, loss of biodiversity, and extreme weather like heat waves and rainstorms (FAO, 2017). Food industry has an enormous challenge to provide healthy food for the growing human population while preventing environmental exhaustion and guarantee natural resources for future generations. It is mandatory that we act now, in order to assure the food security in the future. The concept of food security is that it "exists when all people, at all times, have physical and economic access to sufficient, safe and nutritious food that meets their dietary needs and food preferences for an active and healthy life" (World Food Summit, 1996). In order to feed the entire world in 2050 and guarantee food security, food production will have to increase by more than 70% (McKenzie and Williams, 2015). Food alternatives must be found, and a sustainable development must be practiced (Nunes et al., 2018).

According to the United Nations a sustainable development is “development that meets the needs of the present without compromising the ability of future generations to meet their own needs”. There are three crucial elements to achieve a sustainable development: economic growth, social inclusion, and environmental protection. In 2015, the United Nations comes up with a new program called “Transforming our world: the 2030 Agenda for Sustainable Development”. The aim is to achieve a sustainable consumption and production until 2030 (United Nations). The 2030 Agenda for Sustainable Development sets out a 15-year plan to achieve 17 goals which were adopted by all United Nations Member States. Reaching these goals requires action on all fronts: governments, businesses, civil society, scientists, industry, and people everywhere.

Agricultural and food sustainable development has the same goal, respond to the present needs without compromising the future (Sustainable Agriculture Research and Education Program). Everyone which is involved in the food system (growers, food processors, distributors, retailers, consumers, and waste managers) play an important role to ensure a sustainable agricultural system. There are many ways to contribute to a sustainable agriculture and food system. It is important that growers use methods that promote soil health, minimize water use, and lower pollution levels (Costa et al., 2014). Food industry has a big challenge to face, as it is necessary to improve the environmental impact of products, but also have opportunity to innovate the production technologies. In recent years, consumers are more aware and are looking for a sustainable food manufacturing and processing, that reduces raw materials and minimizes waste. As consumers, looking for foods that are grown using green or environmentally friendly methods, that also help local economy, it is an easy way that we can play a part on this change.

The idea whether future generations needs will be met is uncertain due to a potential lack of resources. In order to achieve sustainable development, it is therefore necessary to choose materials that are more sustainable, to rationalize the exploitation of resources, reduce the production of greenhouses gases, increase the use of renewable energy sources and it is also important to study ways of using the waste resulting from industrial activities. Increasing the value of these products, may result in the transformation of waste into industrial by-products and new raw materials (Valente, 2015).

1.2. Consumer trends and food product development

The food industry is one of the sectors with more impact on the European Union economy. In recent years, it has undergone many transformations, mainly due to the changes in consumers behaviour (Román et al., 2017). The development of new products

is necessary to create and support competitiveness in the food industry, and it is essential when the industry wants to stand out in the market (Costa and Jongen, 2006). It also helps to maintain growth and creates financial support to industries.

Consumer acceptance depends on the social and political background, as religion and education (Stübler et al., 2020). However, in developed societies, food consumption is also decided by health concerns, sustainability, and the convenience of the food product (Asioli et al., 2017). To keep up with consumers' expectations, it is necessary to create products classified as organic, natural, sustainable, and that contribute to well-being. These new food products should be developed in a short period, be more convenient and practical, without compromise the flavour and quality. People want variety and different nutritional sources with health benefits but also products that will save their time, as ready-to-eat meals, frozen food, fast food, or fast cooking (Asioli et al., 2017).

Today's consumers are more aware about the processes utilized in food production considering the levels of safety, hygiene, and environmental effects that could be associated. They also are more concerned about the presence or absence of chemicals and the potential adverse effects of these substances on human health, which led to an increase in the production of foods free from additives or that utilize natural additives. Furthermore, food is also consumed to enhance health and wellbeing which prevent diseases (Bigliardi and Galati, 2013). This interest in healthy and natural food has brought up an increase in the production of functional foods (Bigliardi and Galati, 2013; Granato et al., 2020). Functional food are food products that besides providing nutrients they also have the potential to prevent diseases and improving life quality (Henry, 2010). The development of functional foods involves the presence of compounds with beneficial properties to health, as antioxidant compounds.

The development of a product includes chemical tests, microbiological procedures, sensory analysis, and the use of physical equipment. Even with all the previous mentioned procedures, a lot of new food products fail. There are two major categories of new products: products containing new or alternative ingredients and products, or ingredients obtained from by-products of agricultural and food production. The aim is to improve the quality of the existing products, with health, environmental, economic, and quality benefits (Stübler et al., 2020).

The process of a food product development is a five to eight-step process. It includes the generation of idea/concept, research, development, product testing, and marketing launch (Stewart-Knox and Mitchell, 2003). First, analysis and market study are performed to find something new and different from the competition. A new product concept is defined,

and marketing gives all the information of the product to be launched. Then, different formulas are created and tested in a laboratory to verify costs, nutritional information, and ingredients. The packaging and its composition are also studied, considering the current legislation. After the product is defined, pilot tests are performed to define the production process and sensory and stability characteristics. The product is tested at an industrial level, aligning it with the production and quality necessities. Finally, if a product is adequate to the objectives set, it is validated and is ready for the market launch. All these steps can influence if a product is going to fail or succeed.

A way of predicting consumers' behaviour regarding a food product is through sensory analysis (Yang and Lee, 2019). Sensory evaluation is a scientific method that uses human senses to evolve, measure, analyse, and interpret the consumers' responses to products. There are different sensory analysis methods (discrimination tests, descriptive tests, and consumer acceptance/ preference tests) (Reddy and Love, 1999). Discrimination tests are used to determine differences or similarities between products, while descriptive tests are used to identify specific sensory attributes present in a sample. These tests should use 20 to 50 tasters (Pozo et al., 2006). Consumer acceptance/ preference tests are used to determine consumer acceptance of a product. These tests should use 70 to 150 consumers and ask questions related to the frequency of consumption of that food product (Pozo et al., 2006).

Without sensory evaluation, product development would reflect the product developer's personal feelings, views, and choices, resulting in few product development successes (Pozo et al., 2006). Sensory analyses make people’s perceptions of sensory quality a priority and integrate them with the market. To have a market success is necessary to ensure that food is appealing and appetizing. Eating quality attributes as aroma, taste, tactual properties, and appearance should be acceptable to the consumer so they decide to buy and consume that product (Singh and Maharaj, 2014).

1.3. Agro-industrial by-products

During food production and processing, several agro-industrial by-products are generated. Generally, the food industry converts raw material into an original product using a transformation process (Stübler et al., 2020). As a result, one or more by-products can be obtained. These by-products are not the main purpose of a company's production but can represent a significant percentage (Elmadfa and Meyer, 2010). To be considered a by-product it must show posterior utility in other processes (Mensink et al., 2012). Even though most of them are edible, the majority are discarded, lost, or degraded. Some of them are utilized for animal consumption but not included in the human consumption chain (Garcia

et al., 2020). Food industries produce large amount of by-products and waste, which constitute a serious problem from environmental and economic points of view (Difonzo et al., 2020; Stübler et al., 2020).

The food industry needs to adopt more sustainable measures to preserve natural resources and guarantee food to all people. Therefore, food waste management is of great importance to combat hunger, raise income, maximize the benefit for the environment, society, and economy, and improve food security in the world's poorest countries. Using these by-products is a way to contribute to sustainable food system development, as it will lead to a low amount of waste discarded in nature. Many by-products have toxic compounds with a high impact on the environment. However, when it is reused, the number of residues discarded decreases and it results in less pollution and a low negative impact on the environment. The reuse can lead to tradable items and consequently to more incomes for industries. By-products can be used in other processes of the same industry or sold to other industries (FAO, 2017).

Researchers are paying more attention to waste and by-products because they are not only a potential source of energy but also a source of bioactive molecules (Difonzo et al., 2020). The valuation of by-products represents a trend of increasing interest in many industrial areas, including pharmaceuticals and cosmetics, since these by-products can be purchased at a reduced price but can be rich in important compounds (Gemechu, 2020). They are also a potential source of nutrients and non-nutrient health-promoting compounds. High-value components such as proteins, polysaccharides, fibers, flavour compounds, and phytochemicals can be extracted and reused as novel food ingredients. The food industry has an interest in replacing synthetic with natural additives, derived from plants and agroindustry by-products (Gemechu, 2020). This situation is of crucial importance, especially when it comes to a sector with a high weight in the economy, such as the food industry.

1.4. Olive oil production and olive pomace

The olive tree or *Olea europaea* L. is an evergreen tree, which culture is known for more than 7000 years (Ghanbari et al., 2012; Nunes et al., 2016). Olives grow in the olive tree branches (**Figure 1**). Countries in the Mediterranean basin are the major olive growers, as they are responsible for 97-98% of the world's total olive production (Esteve et al., 2015; Ghanbari et al., 2012). This agro-industrial activity is one of the most important ones for these countries (Nunes et al., 2016). However, it is possible to find this tree all around the world. Over 10 million ha area are covered by about 900 million olive trees. Each olive tree produces 15-40 kg of olives/year, depending on the climate conditions (Bhatnagar et al.,

2014). Global olive oil production is increasing. In the early 90s, were produced 1.8 million tons per year compared to the 3 million tons produced in 2017 (Dumana et al., 2020).

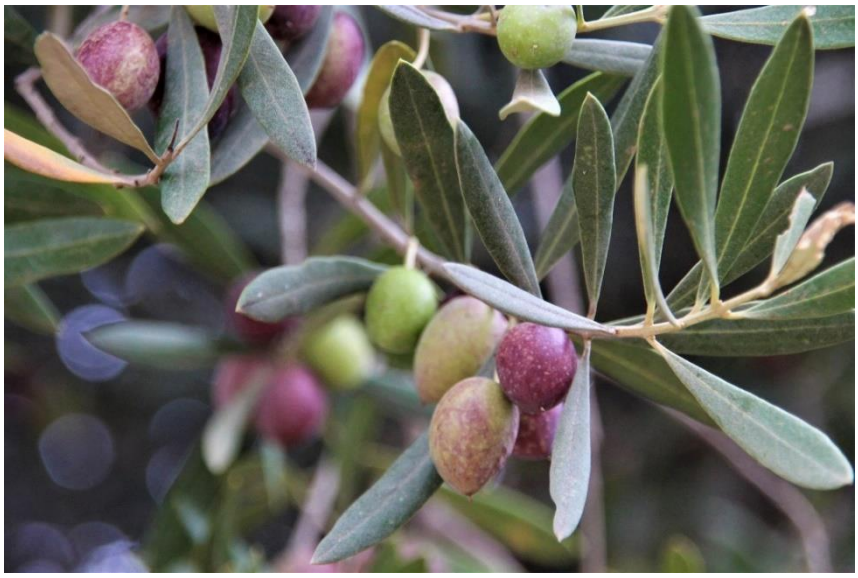

Figure 1. Olives on an olive tree branch
Source: https://pixabay.com/pt/

Olives chemical composition is very variable, depending on the olive variety, soil type, and climatic conditions. In general, it consists of 18–28% oil, 30-35% olive pulp, and 40–50 % vegetation water and stone (Bhatnagar et al., 2014). Olive fruit consists essentially of the epicarp (or peel), mesocarp (or pulp), and the endocarp (or stone), which includes the seed (Bianchi, 2003). The mesocarp is the principal reserve supply of all constituents, including water and oil. The endocarp is also composed of grain, which contains a relevant amount of oil (22-27%). This stone fruit is the raw material for the production of olive oil (Bianchi, 2003).

Different procedures are used to extract olive oil. The most common techniques are the traditional pressing mills (mainly used by small producers), the two-phase system, and the three-phase system (Nunes et al., 2016). Mechanical techniques are used to extract the virgin oil from the olives to guarantee maximum quality. The main objective of the process is to separate the oil drops contained in the fruit's mesocarp cells. The used procedures essentially involve the crush of the fruit to break the plant's tissues (allows the release of the oil), the mixing of the olive paste (induces the coalescence of the oil drops), and the recovery of the oil by centrifugation (continuous mode) or pressing (batch mode) (Nunes et al., 2016; Puértolas and Marañón, 2015). Finally, olive oil is usually filtered or decanted to remove solid waste before bottling.

One of the principal industrial disadvantages of olive oil production is the low efficiency of current extraction techniques in which just 80% of the oil present in the fruit is released (Puértolas and Marañón, 2015). Olive oil production has many health and economic benefits but, it also has a negative environmental impact (Dumana et al., 2020). As a result of seasonable production, large quantities of solid and liquid wastes are generated in a short period (Bhatnagar et al., 2014; Nunes et al., 2016).

These residues are pollutants and cause serious problems as they are toxic, hardly biodegradable, and harmful to the environment (Esteve et al., 2015). They contain phytotoxins, phenolics, and high organic content (Nunes et al., 2016). They are also a threat to aquatic life, as they can create an impenetrable film that difficult oxygen transference and discoloration of natural water (Dumana et al., 2020). Moreover, due to volatile fatty acids, particularly butyric, caproic, valeric, and isobutyric acids, these wastes are a source of odours, especially during warm and dry weather (Bhatnagar et al., 2014). These wastes need to be treated as quickly as possible. However, the management and treatment of these wastes create an economic burden to olive oil producers.

Besides olive leaves and wood, these wastes can be of two different types: solid residues, such as olive pomace or olive cake (characterized by a combination of olive pulp and stone) and the mill's effluent (an aqueous liquor made up of vegetation water, olives tissues, and of water added to the process) (Esteve et al., 2015; Difonzo et al., 2020). Olive mill wastewaters are obtained as a result of the three-phase extraction system. The two-phase system, which is at present the most implemented in this industry, is more eco-friendly as it does not need water. In addition to the olive oil, a by-product is obtained (wet olive pomace) (Nunes et al., 2016; Medina et al., 2012). The global production of olive pomace is approximately 2 million tonnes per year (Dumana et al., 2020).

Olive pomace (**Figure 2**), the main by-product of olive oil production, is a heterogeneous semi-solid biomass with a moisture content of 50–65%, depending on the type of decanter used and residual olive oil (Difonzo et al., 2020; Nunes et al., 2016). After the separation of the oil, this residue consists of fragments of epicarp, mesocarp, and endocarp. Since olive pomace is composed of the remaining constituents of olives, the main chemical compounds are sugars (oligosaccharides and polysaccharides), proteins, monounsaturated and polyunsaturated fatty acids, pigments, and phenolics (Nunes et al., 2016). Olive pomace could also be a good source of dietary fiber and minerals. The unsaponifiable fraction is composed of a great variety of compounds such as aliphatic and triterpenic alcohols, sterols, hydrocarbons, volatile compounds, and antioxidants, as carotenes and phenolics (Medina et al., 2012).

Figure 2. Wet olive pomace
Source: By the author

Reactive oxygen species (ROS) cause the oxidation of lipids and proteins, resulting in DNA molecules breakdown, bases modification, and influencing gene expression modulation (Esteve et al., 2015). Antioxidants can act against aging, cell death, diabetes, and cancer. The peroxidation of fatty acids can cause deleterious effects on food, the development of undesirable flavours such as odours, dark colours, and potentially toxic reaction products. Antioxidant compounds control the oxidation of lipids in food products. Natural antioxidants are safer than synthetic ones (Esteve et al., 2015). Antioxidants are present in olive pomace in concentrations up to 100 times higher than in olive oil (Medina et al., 2012).

The presence of bioactive compounds such as phenolics, phytosterols, tocopherols, tocotrienols, squalene, and carotenoids confers antioxidant properties to OP, making this a low-cost, valuable, and promising by-product (Nunes et al., 2016). The olive pomace bioactive compounds also exert anti-inflammatory, anticancer, and antihypertensive effects. Olive pomace is rich in hydroxytyrosol, an oleuropein derivative and a powerful antioxidant with health benefits (improves cardiac and tumoral diseases) (Nunes et al., 2016). It also has significant content in other phenolics as tyrosol, caffeic acid, p-coumaric acid, vanillic acid, verbascoside, elenolic acid, apigenin-7- glucoside, luteolin-7-glucoside, catechol, and rutin (Nunes et al., 2016; Medina et al., 2012). Among the phenolic acids, cinnamic and ferulic were also detected. These bioactive compounds can be recovered from olive pomace and utilized in the cosmetic, pharmaceutical, and food industries (Nunes et al., 2018).

Recovering and valorisation of this by-product is crucial to the economic return of producers, for the environment and the olive-oil chain sustainability (Esteve et al., 2015). Currently, olive pomace is used to extract the residual oil by solvent extraction and to recover the stone fragments, which can be used as fuel or to produce activated carbon. However, olive pomace is hard to manage for the oil extraction as it is necessary time and energy for pomace dehydrating (Difonzo et al., 2020). Olive pomace is also used as an energy source (biomass) for heaters and fireplaces as a waste reduction strategy. However, through this method, there is no recovery of the valuable components of the olive pomace. Researchers are focusing their efforts on finding alternative and sustainable uses of olive pomace, which involve the extraction of molecules of interest or their direct use as it is.

In the food industry, olive pomace constitutes a great source of functional compounds, and it also has been exploited by several authors to formulate functional foods. It is possible to obtain useful products from olive pomace by different methods. These methods differ in the way physical and chemical treatments are carried out, in the raw materials used, and in the disposal techniques (Dumana et al., 2020). It has been possible to recover phenolic terpenes and polar lipids from solid waste, while from the mill's wastewater, phenolic compounds and polar lipids have been extracted.

Phenolics extracted from olive pomace are being considered in the development of new food formulations with high antioxidant content. Olive pomace phenolic extracts can be used for the enrichment of refined oils such as olive pomace oil, corn, soybeans, high oleic sunflower, sunflower, olive oil, rapeseed oils, and virgin olive oil (Medina et al., 2011). The enrichment of food products with phenolic compounds of olives can have a bitter and spicy taste, being a challenge (Nunes et al., 2016). Despite the health benefits of phenolic compounds, these sensory attributes can lead to rejection by consumers.

Olive pomace is also a promising matrix for extracting and purifying dietary fiber. The cell walls of the olive pulp are rich in pectic polysaccharides with high levels of arabinose that have a high gelation potential. Thus, olive pomace has the potential to be used as a source of pectin for new food applications (Cardoso et al., 2003). The polysaccharides of the olive cell wall contribute to the total dietary fiber content (Nunes et al., 2016). Also, insoluble fiber components can be used as a source of fermentable sugars and specific saccharides. Soluble fiber, such as pectin, can be used as a stabilizing agent or fat substitute in the confectionery industry (Galanakis, 2011).

1.5. Coffee production and coffee silverskin

Coffee is one of the most consumed products in the world (Angeloni et al., 2020). The coffee tree or *Coffea sp.* belongs to the family *Rubiaceae* (Gemechu, 2020). Coffee is

produced from the seeds present in cherries of this small tree (**Figure 3**), largely cultivated in tropical countries (Pozo et al., 2021). The two most popular species of coffee are *Coffea arabica* (known as "Arabica") and *Coffea canephora* (known as "Robusta") (Gemechu, 2020). The world's coffee production is of 75% Arabica and 24% Robusta (Narita and Inouye, 2014). Arabica coffee has a sweeter taste, while Robusta coffee has a higher caffeine content. Coffee industry is the second largest industry in the world, only after oil industry. Brazil is the biggest coffee producer country in the world (Narita and Inouye, 2014).

Figure 3. Coffee tree with cherries
Source: https://pixabay.com/pt/

There are many steps in coffee production: cultivation, harvest, postharvest processing, roasting and brewing (Gemechu, 2020). During the cultivation, the seeds grow and become plants that can reach ten meters high. The harvest is done once a year when the coffee cherries are mature. Two procedures can be utilized: manual or mechanical harvest. The manual harvest gives origin to a better-quality coffee because only the mature cherries are harvested. The mechanical harvest needs a posterior selection as all the cherries are harvested at the same time. In the processing phase, the cherries go through a drying procedure utilizing a dry or wet method (Gemechu, 2020). These methods influence the coffee aroma and acidity. After the drying, cherries go through a cure process in which cascara is removed, and coffee beans are classified according to quality, form, and size. In the roasting phase, coffee is subjected to high temperatures that reach 200°C causing it to increase in size by 80-100% (Narita and Inouye, 2014). It is during this phase that coffee acquires the characteristic colour and reaches the characteristic flavours and aromas (Gemechu, 2020).

Coffee companies generate large amounts of liquid and solid residues which, represent a serious environmental problem. The coffee cherries have an outer skin that covers the pulp and gives origin to cascara, followed by a layer of mucilage and then by the parchment (Iriondo-DeHond et al., 2020). Finally, each hemisphere of the green coffee beans is covered by the silverskin (**Figure 4**). Approximately 90% of the edible parts of the coffee cherries are discarded to obtain the coffee beverages resulting in agricultural waste or by-products (Iriondo-DeHond et al., 2020).

Figure 4. Coffee silverskin
Source: https://overdosecoffee.com/chaff-kepek/

The main by-products originated are cascara or husk, mucilage, parchment, silverskin, and spent coffee grounds. Before they become waste, the coffee industry should exploit and valorise these by-products to increase the sustainability of the process, increase economical income and create new jobs (Iriondo-DeHond et al., 2020). Coffee is one of the most important agricultural products in international trade. Therefore, coffee by-products are amply available (Borrelli et al., 2004). To reduce the disposal of coffee by-products, several authors have previously proposed different approaches to reuse them. Importing countries only perform the roast of green beans so, the wastes are mainly associated with roasting and then consumption, resulting in coffee silverskin and spent grounds, respectively (Iriondo-DeHond et al., 2020). Spent grounds are the remains of coffee after beverage preparation. Researchers have been studying the possibility of utilizing it as fertilizer after composting (Costa et al., 2014).

Coffee silverskin is a strongly adherent layer that covers the bean (Machado et al., 2020). During the roasting procedure, due to the physical expansion of the beans, coffee silverskin detaches from coffee beans and represents the only by-product of the coffee roasting industry (Machado et al., 2020). After that, this by-product is separated from the beans by

airflow (Costa et al., 2014). Coffee silverskin is the principal residue generated in coffee roasting industries, although it only accounts for 1-2% of the whole coffee cherry. Each eight tons of coffee roasted produce 60 kg of coffee silverskin (Nzekoue et al., 2020). It is currently being underutilized and not being industrially exploited by the food sector.

Usually, coffee silverskin is utilized as combustible, firelighters, fertilizer, or dispatched for landfills (Pozo et al., 2021; Costa et al., 2014). The potential use as cellulose for paper production is also under study (Angeloni et al., 2020). There are no commercially available products containing this potentially novel ingredient. However, many recent approaches are suggested to its valorisation to answer the circular economy and sustainability issues (Machado et al., 2020).

Different authors highlighted the potential of this by-product as a raw material for the recovery of functional compounds of potential interest or as an alternative ingredient for the development of functional food (fiber-rich) and food enrichment or even to be consumed as a food supplement (Machado et al., 2020; Nzekoue et al., 2020). It also can be used in body weight control products or as a natural preservative for the food industry. The silverskin incorporation in flakes, bread, biscuits, cakes, and snacks and a new coffee blend containing coffee silverskin has been studied (Machado et al., 2020). Some studies suggest that the incorporation of coffee silverskin can reduce caloric density, increase dietary fiber content, improve moisture content, thickness, colour, textural and sensory attributes, and create healthier, nutritious, and high-sensorial-quality products (Martinez-Saez and Castillo, 2019).

The chemical composition of the raw seeds varies according to their origin and maturation degree. These factors and roasting conditions have an impact on coffee silverskin chemical composition. Silverskin is a rich source of dietary fiber (≈ 60%, mainly insoluble but with a significant amount soluble (14%), protein (≈ 20%), and several bioactive compounds with antioxidant activity (Borrelli et al., 2004; Machado et al., 2020). These characteristics are recognized factors in contributing to consumer's well-being and in the prevention of chronic diseases (Costa et al., 2014). The dietary fiber and protein present in this by-product could easily be extracted to use as an ingredient within the concept of a healthy diet. It contains high concentrations of polysaccharides (60−70%), and the total sugars content varies from 2% to 12%. It has a low amount of carbohydrates, a low-fat content (≈ 3%), and it is also a source of micronutrients, vitamins (such as ascorbic acid) and minerals (≈ 8%), mainly potassium, magnesium, and calcium (Machado et al., 2020).

The main fatty acid found in coffee silverskin is palmitic acid (C16:0), followed by linoleic (C18:2), behenic (C22:0), and arachidic (C20:0) acids (Angeloni et al., 2020). Silverskin is

an interesting by-product to be used for food applications due to its low moisture content (5–10%) (Machado et al., 2020). This makes coffee silverskin a stable by-product. The odour-active compound fraction of coffee silverskin is very interesting as it shows a high similarity to coffee beans but a less complex and intense aroma. A recent study identified a total of 40 odorants in coffee silverskin, including organic acids (especially short-chain fatty acids), furans, furfurals, ketones, aldehydes, alcohols, pyridines, phenolics, and lactones (Angeloni et al., 2020).

A recent study found a total of 17 bioactive compounds in coffee silverskin. Caffeine and chlorogenic acids usually are the analytes present in the highest concentrations. Bioactive compounds present in coffee silverskin include melanoidins, caffeine, alkaloids, chlorogenic acids, phenolic acids, flavonoids, and secoiridoids. Caffeine levels vary depending on the geographical origin but usually range from 0.8% to 1.4%. The main phenolic compounds found are chlorogenic acids (Angeloni et al., 2020). The compounds have health-promoting properties, such as antioxidant, anti-diabetic, and anti-obesity. The reported high antioxidant activity of coffee silverskin is due to the presence of phenolic compounds mentioned before but mainly to melanoidins, compounds formed during the roasting procedure through the substantial amount of Maillard reactions (Borrelli et al., 2004).

Coffee shows health properties as antioxidant capacity, antibacterial activity, anti-obesity, anticarcinogenic, anti-inflammatory, antihypertensive, antiglycative activities, and prebiotic effects (Iriondo-DeHond et al., 2020; Borrelli et al., 2004). As coffee silverskin is the outer layer of the roasted coffee beans, it also shows some of the properties described for coffee (Borrelli et al., 2004). Several biological activities have been attributed to coffee melanoidins.

This by-product has an inhibitory effect on *in vivo* fat accumulation, reducing body fat by 21-24%, possibly due to the presence of caffeine and chlorogenic acids at physiologically active doses. Coffee silverskin may decrease total cholesterol and triglycerides plasma levels, and pancreatic lipase activity, acting as a preventive and therapeutic effect in obesity. The components present in coffee silverskin have a positive effect on pancreas health and can reduce the risk of diabetes. Due to the high dietary fiber content (especially insoluble fiber), coffee silverskin can potentially benefit the intestine and gut microbiota, as it preferentially supports the growth of bifidobacteria *in vitro*, suggesting that its consumption may have some prebiotic effects (Borrelli et al., 2004; Machado et al., 2020).

Some apparent problems with the use of coffee silverskin need to be taken into consideration. One of them is the caffeine content. The European Food Safety Authority (EFSA) considered 400 mg for the general population and 200 mg for lactating women as

the safety level for daily caffeine consumption. In all coffee by-products, the caffeine content is lower than that found in green and roasted coffee beans commercially available (Iriondo-DeHond et al., 2020).

Another problem is acrylamide, a processing contaminant formed during the roasting process through Maillard reactions. Acrylamide is formed when there is a combination of precursors like reducing sugars (glucose and fructose) and asparagine, with high temperature, and long heating time (Pedreschi et al., 2014). The limit levels of acrylamide established by EFSA for coffee was 450 µg/kg (Iriondo-DeHond et al., 2020). Some previous studies found levels of 346-370 µg/kg of acrylamide in coffee silverskin, so acrylamide levels for coffee silverskin are also under the limit levels (Garcia-Serna et al., 2014) .

Ochratoxin A (OTA) is another problem that needs to be considered. *Aspergillus ochraceus* and *Penicillium verrucosum* produce the mycotoxin that can induce immunosuppression, nephropathy, and renal toxicity (Toschi et al., 2014). OTA is accumulated in the harvest and postharvest handling of coffee cherries, so OTA is already present in coffee before storage (Iriondo-DeHond et al., 2020). However, a study demonstrated that even if the green beans are highly contaminated, OTA is destroyed during roasting (Ferraz et al., 2010). The coffee-transforming process can reduce the OTA amount to values below the limit established by the European Commission (5 µg/kg) and that, does not represent a risk to human health.

Other studies also confirmed the absence of pesticides and mycotoxins (aflatoxin B1 and enniatin B) (Iriondo-DeHond et al., 2019). Therefore, there are no limitations to the use of this by-product as a food ingredient for human nutrition, as coffee silverskin safety is already confirmed.

1.6. Pasta products

Among all products available on the market, pasta products are one of the most popular, simpler, and convenient food products (Gupta et al., 2021). Pasta is a traditional cereal-based and staple food product. Originally it was a part of Italian and European cuisine. With an increase in popularity, pasta has crossed international borders and now, is accepted by many cultures and available as a major dietary source of energy around the world (Huang et al., 2017; Kadam and Prabhasankar, 2010).

Pasta can be divided into two categories: dried and fresh pasta. The convenience, nutritional quality, high versatility, and palatability are reasons that can justify this success (Littardi et al., 2020). Pasta has a low cost, long shelf life, good nutritional composition and

preparation and transportation are easy (Gupta et al., 2021; Marti and Pagani, 2013). In 2018, world production of pasta reached 14.3 million tonnes with Italy being responsible for 3.24 million tonnes of the worldwide production (Gupta et al., 2021). It is estimated that in Italy every person consumes about 28.5 kg of pasta per year (Kadam and Prabhasankar, 2010). Pasta can be formed into many shapes, and have different tastes, colours, and textures (**Figure 5**).

Figure 5. Pasta products made from different ingredients with various shapes and colours Source: https://pixabay.com/pt/

The consumer's acceptance and the cooking characteristics of pasta are dependent upon the raw material composition (Xie et al., 2020). Pasta is a type of food typically made from an unleavened dough of white flour or semolina, from durum wheat (*Triticum durum*), mixed with water or eggs (Saget et al., 2020; Xie et al., 2020). To produce high-quality pasta, the most suitable raw material is durum wheat semolina (Petitot et al., 2009). This is due to its quality properties, as cooking properties, unique colour, flavour, and aroma, that influence consumer's choices (Littardi et al., 2020).

Through a process called kernel milling, it is removed the germ and external layers of the caryopses from durum wheat (Hidalgo et al., 2020). As a result, white flour and semolina are the most common refined products. Most lipids, fiber, minerals, and antioxidants from wheat are located on the parts that are removed during this refining procedure. As consequence, products prepared from white flour and semolina have an inferior nutritional value than those manufactured from whole grain (Hidalgo et al., 2020).

Nutritionally, pasta is a complex multi component system (Kadam and Prabhasankar, 2010). It is a good source of biomacromolecules such as carbohydrates, proteins, and lipids.

The interest of pasta is increasing due to its low glycaemic index (GI) (Huang et al., 2017). The GI measures the relative glucose-raising property of carbohydrates-containing foods compared to glucose or white bread with equivalent amount of carbohydrates. For pasta, GI was found to be low to moderate (GI < 55 on the glucose scale) (Huang et al., 2017). The specific structure of pasta obtained after successive structural changes of starch and proteins, is responsible for this interesting nutritional property (Petitot et al., 2009).

Perhaps, it is not considered a nutritionally well-balanced product as it shows low protein and fibre contents (Littardi et al., 2020). Usually, raw past consists in 74-77% of carbohydrates while cooked pasta consists in 62% of humidity, 31% of carbohydrates and 6% of protein. It has a very low content in fat. In fact, semolina contains starch as a principal constituent but also protein, fat, vitamins, minerals, and bioactive compounds. The protein content of semolina ranged between 9.0 and 14.0 g/100 g. Semolina has lysine as a limiting amino acid (Gupta et al., 2021).

Pasta products have excellent nutritional and safety quality. The most important factors in assessing quality in pasta products are the cooking quality and appearance (Feillet et al., 2000). Three groups of parameters determine the appearance of pasta: colour, specks (brown and black specks) and texture (Feillet et al., 2000). The quality of cooked pasta implies low amounts of solids lost during cooking, low stickiness, and firm structure (Ungureanu-Iuga et al., 2020).

Traditional pasta is characterised by the presence of gluten proteins (Littardi et al., 2020). A few decades ago, appeared the gluten-free (GF) pasta products. Gluten-free products are recommended for people suffering from gluten related disorders as celiac disease, non-celiac gluten sensitivity and gluten allergy sufferers (Ungureanu-Iuga et al., 2020). Around 1.4% individuals of the world suffer from celiac disease, and it can occur at any age (Marti and Pagani, 2013; Saget et al., 2020). Gluten intolerance is caused by the main protein from wheat. In addition, chronic diseases (obesity, cardiovascular diseases, diabetes, constipation) are associated with diets rich in processed foods that have high amounts of saturated fat, refined sugar, and low content of dietary fiber (Ungureanu-Iuga et al., 2020).

Nowadays, consumer's health awareness is leading to an increase of gluten-free products demands (Ungureanu-Iuga et al., 2020). These products are consumed not only by celiac but also by people that wish to exclude from their diet gluten-based products for health reasons. Gluten is responsible for the unique rheology attributes of the dough and influence the quality of the cooked product, as it is responsible for the elasticity and chewability (Littardi et al., 2020; Ungureanu-Iuga et al., 2020).

Gluten absence from pasta can cause technological quality problems to the final product. However, some studies report that these problems can be minimized using an appropriate ratio of proteins, hydrocolloids, and water. To meet those people needs there is a broad variety of pasta GF products, some use rice, maize, vegetable powders, corn, quinoa, amaranth, oats, as wheat flour substitutes (Marti & Pagani, 2013). Rice flour, or legumes such as beans or lentils, are sometimes a good alternative to wheat flour to give a different taste and texture to pasta. Unfortunately, and most of GF products exhibit poor cooking quality, particularly when compared to the wheat-based foods (Marti & Pagani, 2013). Therefore, the production of good quality GF products is a challenge.

Despite all the characteristics that make pasta a success, pasta is a low source of protein, minerals, essential fatty acids, vitamins, and bioactive compounds. There is a demand for value-added products that meet specific dietary needs, namely organic, wholemeal, and gluten-free pasta. There is also a great demand for new formats that make a dish more appealing due to the image and texture of the pasta.

In addition to the conventional pasta, pasta may also be enriched or fortified with algae, carob, spinach, oats, vegetables, or bamboo fiber. It is common to enrich pasta to enhance nutritional properties. Pasta is usually enriched with protein or protein-rich products as legume flour, shrimp, meat, fish and whey proteins, and some cereals or pseudo-cereals to provide sources of fibre, minerals, antioxidants, and phenolics (Marti and Pagani, 2013; Xie et al., 2020). However, different protein sources may significantly affect the cooking quality. For example, whey protein increases the cooking loss and decreases pasta quality (Xie et al., 2020). This means it is necessary to find out a protein source that with an appropriate incorporation percentage, can lead to a nutritional increase and maintain the textural properties of pasta.

Dry pasta is an excellent matrix to stabilize phytochemicals that in fresh vegetables are easily degraded (Oliviero and Fogliano, 2016). The use of pasta as a carrier of specific compounds is not new as the relatively simple formulation of pasta makes it perfect to incorporate specific nutrients as protein, different dietary fiber, omega-3 fatty acids, or antioxidants, in order to become a functional product (Oliviero and Fogliano, 2016). The most common is the enrichment with dietary fibre as it reduces the GI of pasta. A low GI may prevent constipation and stimulate the formation of short-chain fatty acids (Oliviero and Fogliano, 2016).

Several studies have been conducted to improve the nutritional quality of pasta (**Table 1**). The addition of eggs can be an improvement to the nutritional composition but also to the technological characteristics (Hidalgo et al., 2020). In order to achieve better nutritional

values, organoleptic properties, and structure formation, eggs are usually incorporated into pasta with a range of 17% to 30% (Xie et al., 2020). Traditionally it is utilized whole eggs or egg yolk. The egg white powder can also be incorporated to improve the texture while chewing and elasticity of pasta (Xie et al., 2020).

Table 1. Research and development of alternative pastas

Article title	Pasta	Reference
Changes in the physical, textural and chemical properties of the enriched pasta elaborated with cassava starch	Pasta enriched with cassava starch	Milde et al., 2021
Effects of mixing, sheeting, and cooking on the starch, protein, and water structures of durum wheat semolina and chickpea flour pasta	Pasta made of or enriched with chickpea flour	Garcia-Valle et al., 2021
Pasta from yellow lentils: How process affects starch features and pasta quality	Pasta made from 100% yellow lentils	Bresciani et al., 2021
Pasta products enriched with moringa sprout powder as nutritive dense foods with bioactive potential	Pasta enrichment with 5%, 10%, 15%, 20%, and 30% of moringa sprout powder	Coello et al., 2021
Simultaneous optimization of wheat heat moisture treatment and grape peels addition for pasta making	Pasta enriched with grape peels	Iuga and Mironeasa, 2021
Use of Moldavian dragonhead seeds residue for pasta production	Pasta enriched with Moldavian dragonhead seeds	Zarzycki et al., 2021
Common wheat pasta enriched with cereal coffee: Quality and physical and functional properties	Pasta enriched with cereal coffee	Biernacka et al., 2021
Rejuvenated Brewer's Spent Grain: The impact of two BSG-derived ingredients on techno-functional and nutritional characteristics of fibre-enriched pasta	Pasta enriched with brewer's spent grains	Sahin et al., 2021

Food by-products valorisation: Grape pomace and olive pomace (pâté) as sources of phenolic compounds and fiber for enrichment of tagliatelle pasta	Pasta fortified with grape pomace or olive pomace	Balli et al., 2021
Pasta fortification with olive pomace: Effects on the technological characteristics and nutritional properties	Pasta fortified with olive pomace	Simonato et al., 2019
Nutrient composition and in vitro digestibility of fresh pasta enriched with *Vicia faba*	Pasta enriched with 10, 30 and 50% of *Vicia faba*	Tazrart et al., 2016
Buckwheat-pasta enriched with silkworm powder: Technological analysis and sensory evaluation	Buckwheat pasta enriched with silkworm	Biró et al., 2019
Physicochemical, texture and sensorial evaluation of pasta enriched with chickpea flour and protein isolate	Pasta enriched with 2.5, 5, 7.5 and 10% of chickpea flour or chickpea protein isolate	El-Sohaimy et al., 2020

Many different strategies are followed by researchers to improve the nutritional quality of pasta. The direct use or the use of bioactive compound-rich extracts from waste and food industry by-products represents an objective to pursue for their valorisation and for economic and environmental sustainability. However, it is important to choose the optimal quantity to incorporate to avoid a negative influence on sensory attributes and the overall quality of pasta.

Pasta is even starting to appear on the market in which wheat is not the main ingredient (Saget et al., 2020). There are already on the market pastas made from pea flour, oat flour, quinoa, teff, wholemeal, rice, maize, or multicereal. Consumer preference has been changing from animal-based foods to plant-based foods. The nutritional potential of plant-based food applications could lead to functional food ingredients (Gupta et al., 2021).

2. Main and specific objectives

Considering the aforementioned, it is important to add value to agro-food industry by-products. Incorporating these by-products into food, was what motivated this work.

The main objective of this dissertation was the development of pasta enriched with two completely different by-products, coffee silverskin and olive pomace. To a complete assessment of the developed formulations, the nutritional and antioxidant properties were analysed and compared. Pasta consumers' acceptability was also evaluated. A group of tasters performed a sensory analysis filling up a 9-parameter questionnaire based on a hedonic scale for each question. It was also performed a preference test.

In order to accomplish the main objective, there was several intermediate goals to consider:

- Olive pomace treatment to enable its incorporation into food;
- Nutritional (moisture, ash, fat, protein, and carbohydrates) and antioxidant (TPC, TFC, FRAP assay and DPPH scavenging activity) characterization of fresh and treated olive pomace;
- Fatty acids and vitamin E profiles of fresh and treated olive pomace;
- Nutritional (moisture, ash, fat, protein, and carbohydrates) and antioxidant (TPC, TFC, FRAP assay and DPPH scavenging activity) characterization of coffee silverskin;
- Fatty acids and vitamin E profiles of coffee silverskin;
- Definition of a control pasta and enriched recipes;
- Definition of coffee silverskin and olive pomace percentages to be incorporated into pasta;
- Nutritional (moisture, ash, fat, protein, and carbohydrates) and antioxidant characterization of control pasta, olive pomace pasta and coffee silverskin pasta (raw and cooked);
- Fatty acids and vitamin E profiles of control pasta, olive pomace pasta and coffee silverskin pasta (raw and cooked);
- Consumer acceptability (sensory analysis) of control pasta, olive pomace pasta and coffee silverskin pasta;

3. Material and Methods

3.1. Reagents and Standards

Sodium hydroxide pellets were acquired from Fisher Scientific (UK) and boric acid was from Labkem (Barcelo, Spain). Kjeldahl tablets, sodium sulphate, anhydrous sodium sulphate and Folin-Ciocalteau reagent were acquired from Merck (Darmstadt, Germany). Sulfuric acid and methanol were from Honeywell Fluka (Düsseldorf, Germany). Petroleum ether and hydrochloric acid 1 mol/l were provided by CARLO ERBA REAGENTS (Val de Reuil Cedex, France). Sand was from VWR CHEMICALS (Belgium). Sodium hydroxide 1mol/l from and sodium dihydrogen phosphate were provided by VWR Chemicals (France). Total Dietary Fiber Assay Kit, celite, sodium carbonate, gallic acid, sodium nitrate, aluminium chloride, sodium acetate, ferric chloride, TPTZ 10 mM, ferrous sulphate, and catechin were acquired were from SIGMA-ALDRICH (St. Louis, U.S.A). Ethanol 96% was from AGA (Prior Velho, Portugal). Acetone was from Fischer Chemical (UK). Acetic acid (99-100%) were provided from Chem-Lab (Belgium). A standard mixture (FAME 37) was obtained from Supelco, Bellefonte, PA, USA. HPLC-grade solvents were obtained from Sigma–Aldrich (St. Louis, USA) or Merck (Darmstadt, Germany). All other reagents were of analytical grade. Ultrapure water was obtained in a Milli-Q water purification system (Millipore, Bedford, MA, USA).

3.2. Equipment

Different apparatus were utilized for the research, being the case of: a Bimby (TM5; VORWERK) for prepare, cooking and triturate samples; a freeze dryer (Telstar Cryodos-80 Terrassa; Barcelona) for sample preservation; an analytical balance KERN AES, with a range between 0.0001 and 220 g, for weight samples; an infrared balance (Scaltec model SMO01; Scaltec Instruments, Heiligenstadt, Germany) for the moisture content; a Buchi Digestion Unit K-424, Scrubber B-414 and a KjelFlex K-360 for total protein; and a huber minichiller and a heating mantle P Selecta for lipid fraction; a multistirrer 6, a heating magnetic stirrer, a heated circulating bath, and a filtration system CSF 6, all from VELP Scientific, and an 827-pH lab (Metrohm) were used for fiber analysis.

For vitamin E profile determination was utilized an HPLC system equipped with a multiwavelength diode array detector (MD-2015), coupled to a fluorescence detector (FP-2020; Jasco, Tokyo, Japan). A normal phase column, 75 mm x 3.0 mm inner diameter and 3.0 µm film thickness (SupelcosilTM LC-SI; Supelco, Bellefonte, USA), was utilized.

For fatty acids profile determination were utilized a GC-2010 Plus gas chromatograph, a split/splitless auto injector (AOC-20i), an automatic sampler and a Flame Ionization Detector

(Shimadzu, Tokyo, Japan) A silica capillary column, 50.0 m x 0.25 mm inner diameter and 0.20 µm film thickness (CP-Sil 88; Varian, Middelburg, Netherlands), was utilized.

A furnace (48000; ThermoLyne), an oven, two centrifuge types (Sepatech and Biofuge pico; Heraeus), a microplate reader (Synergy HT; BioTek) an orbital vortex mixer (VV3; VWR Int.) and a heating block (SBH130D/3; Stuart, Stafford, UK) were also used during the experimental procedures.

3.3. Sampling and samples preparation

Different samples were prepared and analysed in this work: the by-products (coffee silverskin and olive pomace) and pasta prepared with the by-products.

3.3.1. By-product's preparation

The coffee silverskin sample was used directly as it is obtained from producer and milled at the laboratory in order to obtain a powder. It was also analysed an olive pomace sample which was divided into three parts: one part was freeze-dried and milled; other was dried at 40˚C for 48h, milled and then sieved to remove the stone; the last part was dried at 70˚C for 24h (**Figure 6**), milled and then it was also sieved in various stainless-steel sieves with a particle size of 1.4, 1.25, 1 and 0.5 mm to remove the stone. In the two last, it was possible to obtain an olive pomace powder without the olive stone (**Figure 7**).

Figure 6. Olive pomace dried at 70ºC
Source: By the author

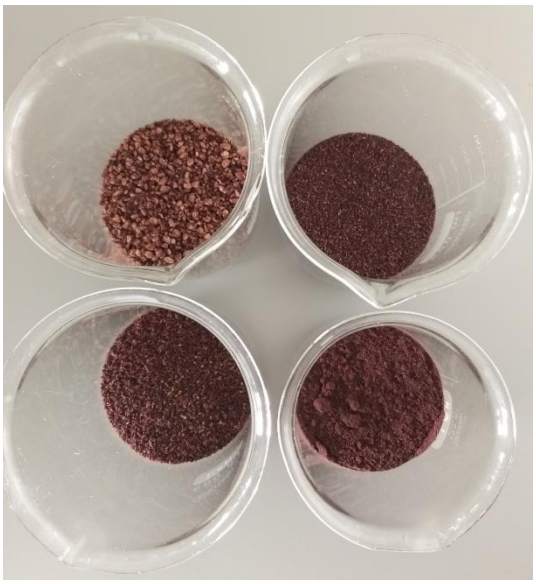

Figure 7. Sieved olive pomace
Source: By the author

3.3.2. Pasta samples' preparation

Pasta samples were manufactured at the laboratory. The control sample was made with wheat flour or semolina and water, the enriched samples were obtained replacing a percentage of wheat flour or semolina for coffee silverskin powder or for olive pomace

powder. Pasta was structured into laces and dried at 50ºC for 5 hours according to Gupta et al. (2021) to increase shelf time.

For control sample were tested 2 different formulations:

1- 200 g of wheat semolina + 110 mL of water
2- 200 g of wheat flour + 110 mL of water (**Figure 8**)

Figure 8. Selected control pasta
Source: By the author

For coffee silverskin were tested 2 different percentages to incorporate (10% and 7.5%) with a total of 4 different formulations:

1- 180 g of wheat semolina + 20 g of coffee silverskin + 110 mL of water
2- 180 g of wheat flour + 20 g of coffee silverskin + 110 mL of water
3- 185 g of wheat semolina+ 15 g of coffee silverskin + 110 mL of water
4- 185 g of wheat flour + 15 g of coffee silverskin + 110 mL of water (**Figure 9**)

Figure 9. Selected coffee silverskin (7.5%) pasta
Source: By the author

For olive pomace were tested 2 different percentages to incorporate (10% and 7.5%) with a total of 4 different recipes:

1- 180 g of wheat semolina + 20 g of olive pomace + 110 mL of water
2- 180 g of wheat flour + 20 g of olive pomace + 110 mL of water
3- 185 g of wheat semolina+ 15 g of olive pomace + 110 mL of water
4- 185 g of wheat flour + 15 g of olive pomace + 110 mL of water (**Figure 10**)

Figure 10. Selected olive pomace (7.5%) pasta
Source: By the author

These pastas were cooked for 10 min at 100˚C using a Bimby TM5 and then, raw, and cooked samples were freeze-dried and milled to further analysis. Sample codes are presented in **Table 2**.

Table 2. Samples codes

Code	Sample
OP	Fresh olive pomace
OP40ºC	Olive pomace powder dried at 40ºC
OP70ºC	Olive pomace powder dried at 70ºC
CS	Coffee silverskin
RP	Raw control pasta
CP	Cooked control pasta
RPP	Raw olive pomace (7.5%) pasta
CPP	Cooked olive pomace (7.5%) pasta
RSP	Raw coffee silverskin (7.5%) pasta
CSP	Cooked coffee silverskin (7.5%) pasta

3.4. Extracts preparation

Olive pomace extracts were obtained using 100 mg of sample and 40 mL of solvent (50% ethanol/ 50% water solution; v/v), for 1 h, on vortex (**Figure 11**). Coffee silverskin extracts were performed using 400 mg of sample and 40 mL of solvent (50% ethanol/ 50% water solution; v/v), for 1 h, on vortex (**Figure 11**). For pasta extracts it was performed an extract optimization. Pasta extracts were obtained by testing 250 mg, 500 mg, and 1 g of sample in 40 mL of solvent (50% ethanol/ 50% water solution; v/v), for 1 h, on vortex. All extracts were made in triplicate, centrifuged for 5 min at 5000 rpm and stored at -20˚C.

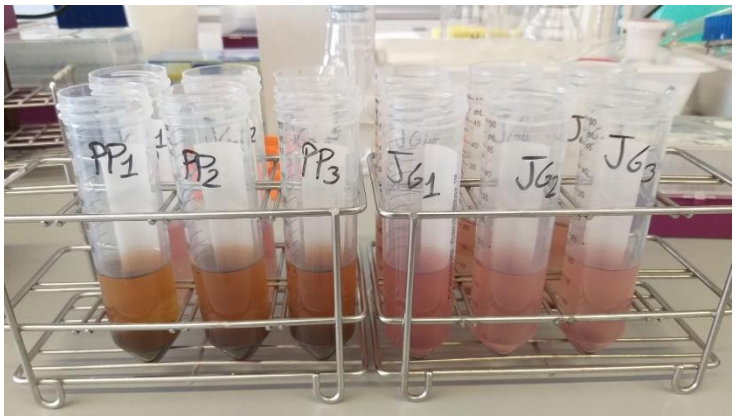

Figure 11. Olive pomace and coffee silverskin extracts
Source: By the author

3.5. Nutritional analysis

For the nutritional analysis, it was assessed the samples moisture, the protein, fat and ash content, the available carbohydrates and fiber (total, insoluble and soluble fiber), according to AOAC INTERNATIONAL procedures (AOAC, 2019). All samples were analysed at least in triplicate.

3.5.1. Moisture

The moisture was determined directly utilizing an infrared balance (**Figure 12**).

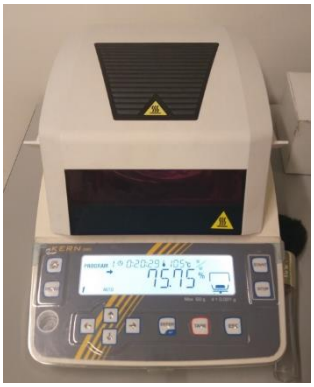

Figure 12. Moisture infrared balance
Source: By the author

3.5.2. Protein content

Protein content was determined by Kjeldahl method (AOAC 920.153). This method assesses the total nitrogen content based on two assumptions: dietary carbohydrates and fats do not contain nitrogen, and almost all the nitrogen in the diet is present as amino acids. 1 g of sample wrapped in parchment paper was introduced in Kjeldahl tube and added to two catalyst pellets and sulphuric acid. The digestion occurred for 1 hour. After, it was distilled (**Figure 13**) and then It was performed an acid-base titration between sodium hydroxide and sulfuric acid using methyl red as indicator. The used factor was 6.25.

Figure 6. Protein samples distillation
Source: By the author

3.5.3. Fat

Total fat was determined in continuous extraction with a single solvent, by Soxhlet method (AOAC 991.36). This method is recommended by AOAC and to low-moisture foods (AOAC, 2019). It was utilized 5 g of sample with sand and anhydrous sodium sulphate. This mix was transferred to cellulose cartridges and soaked in petroleum ether which was the solvent used for extraction performed for 12 hours (**Figure 14**).

Figure 7. Procedure of fat extraction
Source: By the author

3.5.4. Ash

The mineral percentage was determined by sample (5 g) incineration in crucibles at 500ºC (**Figure 15**), until samples became white (AOAC 920.153).

Figure 85. Samples for mufla incineration
Source: By the author

3.5.5. Available Carbohydrates

Available carbohydrates were obtained by difference, according to Tontisirin (2003). Results are presented in dry weight and were obtained using the following equation:

% Available carbohydrates = 100 – (% moisture + % protein + % fat + % ash + % total fiber)

3.5.6. Total, soluble, and insoluble fiber

Total, soluble, and insoluble fiber were analysed through the enzymatic-gravimetric method (AOAC 985.29) using the Total Dietary Fiber Assay Kit (TDF 100A- 1KT).

For the total fiber results obtention, 3 days were need. In the first day, 0.5 g of sample was introduced in a beaker with 50 mL of phosphate buffer (pH = 6) and 100 µL of α-amylase and introduced in a bath at 95ºC for 20 minutes, with stirring. After the sample cooled, it was added 10 mL of sodium hydroxide 0.275M and 100 µL of protease. Another bath at 60ºC for 35 minutes with stirring was used. Once again, after the sample cooled, it was added 10 mL of hydrochloric acid 0.325M and 100 µL of amyloglucosidase and introduced in the last bath at 60ºC for 35 minutes with stirring. After the sample cooled, it was added 200 mL of ethanol (95%). The solution was left at room temperature overnight. On the second day, the samples were filtrated in a filtration system and washed with 60 mL of ethanol (78%), 20 mL of ethanol (96%) and 20 mL of acetone. After that, crucibles were left overnight in oven at 105ºC. On the last day, half content of the crucibles was incinerated at 525ºC for 5 hours. In the other half of crucibles, the protein content was determined, as previously described.

The procedure for the insoluble fiber is almost the same that for total fiber. The difference is that in the first day we did not add the last 200 mL of ethanol and in the second day samples were washed with 20 mL of distilled water, 20 mL of ethanol (96%) and 20 mL of acetone.

The results for soluble fiber were obtained by difference, according to the following equation:

% Soluble fiber = % total fiber – % insoluble fiber

3.6. Lipidic fraction analysis

3.6.1. Lipidic fraction extraction

Samples' lipid fraction was extracted according to Alves et al. (2009), with some modifications.

Briefly, to an appropriate amount of sample with a 20% total fat content were added 75 µL of BHT 0.1% (m/V), 50 µL of tocol (internal standard, 0.1 mg/mL) and 1 mL of absolute ethanol. The solution was homogenized for 30 min in a vortex. After, 2 mL of n-hexane (HPLC grade) were added and the solution was homogenized for another 30 min. 1 mL of NaCl 1% (m/V) was added, the solution was vortexed and centrifuged at 3000 rpm for 5 min. The supernatant was collected, and the residue was re-extracted with 2 mL of n-hexane, homogenized for 30 min, and centrifuged at 3000 rpm for 5 min. The supernatant was collected and mixed with the previous one. Anhydrous sodium sulphate was added and the solution then vortexed and centrifuged at 3000 rpm for 5 min. Then, the supernatant was collected and evaporated in nitrogen stream until 1 mL. Another centrifugation was performed at 13 000 rpm for 5 min and after, 500 µL were injected immediately in a HPLC-DAD-FLD (high-performance liquid chromatography coupled to diode array detector and fluorescence detector) system for determination of the vitamin E profile. 500 µL were used for determination of the fatty acids profile by GC-FID (gas chromatography coupled with flame ionization detector).

3.6.2. Vitamin E

The vitamin E profile and quantification by HPLC-DAD-FLD of the lipidic fraction of the samples were determined according to Alves et al. (2009), with slight modifications. The chromatographic analysis was carried out in a HPLC system equipped with a multiwavelength diode array detector coupled to a fluorescence detector, programmed for excitation at 290 nm and emission at 330 nm. The chromatographic separation of the compounds was achieved using a normal phase Supelcosil™ LC-SI column (75 mm × 3.0 mm, 3.0 µm), the eluent used was 1.2% 1.4-dioxane in n-hexane (HPLC

grade), at 0.6 mL/min. Vitamin E vitamers were identified based on their UV spectra and by comparing their retention times with those of standards (α-tocopherol, β-tocopherol, γ-tocopherol, δ-tocopherol, α-tocotrienol, β-tocotrienol, γ-tocotrienol, δ-tocotrienol). Quantification was performed based on the fluorescence signals.

3.6.3. Fatty acids

The fatty acids profiles were obtained using a gas chromatograph coupled with flame ionization detector (GC-FID), after derivatization to fatty acids methyl esters, according to ISO 12966-2017. Briefly, the lipid fraction extract obtained before (section 3.6) was resuspended in 1 mL dichloromethane. 1.5 mL of KOH (0.5 M) were added and mixed. Solutions were placed in a heating block at 100ºC, for 10 min. After, they were left at room temperature for 1 min and then placed in ice for 5 min. Then, 2 mL of BF_3 in methanol solution (14%) was added and mixed. Solutions were placed in the heating block at 100ºC, for 30 min. After, they were cooled at room temperature for 1 min and then placed in ice for 5 min. 2 mL of deionized water and 3 mL of n-hexane (HPLC grade) were added and mixed. The tubes were centrifuged at 3000 rpm, for 5 min. The superior phase was collected, and it was added anhydrous sodium sulphate. The mixture was centrifuged at 3000 rpm for 5 min and the supernatant was transferred to an injection vial for GC analysis.

The compounds separation was performed by a GC, with an automatic sampler and a split/splitless auto injector operating with a 50:1 split ratio at 250 ºC (injection), CP-Sil 88 silica capillary column (50 m × 0.25 mm i.d., 0.20 µm film thickness) and a Flame Ionization Detector at 270 ºC. The analyses were performed using helium (3.0 mL/min) as the carrier gas and applying the following temperature programme: 120 ºC held for 5 min, 2 ºC/min to 160 ºC held for 15 min and 2 ºC/min to 220 ºC held for 10 min. The injection volume was 1.0 µL. The fatty acids methyl esters were identified by comparison with a standard mixture (FAME 37). Data were analysed based on relative peak areas. Results of fatty acids were expressed in relative percentage of total fatty acids.

3.7. Total phenolics content (TPC)

The amount of total phenolics was determined using the Folin-Ciocalteu (FC) method, according to Costa et al. (2014) with modifications. TPC of extracts and a blank test of deionised water were determined using a calibration curve with increasing concentrations of a standard solution of gallic acid (5-100 mg/mL; R^2= 0.999), by spectrophotometric measurement. In a microplate, 30 µL of each extract/ deionised water were mixed with 150 µL of FC reagent and 120 µL of sodium carbonate (7.5%; m/v). The microplate was incubated at 45 ºC for 15 minutes, after it was cooled down at room temperature for 30

minutes, protected from light (**Figure 16**). The absorbance was measured at 765 nm and the results expressed as gallic acid equivalents (GAE).

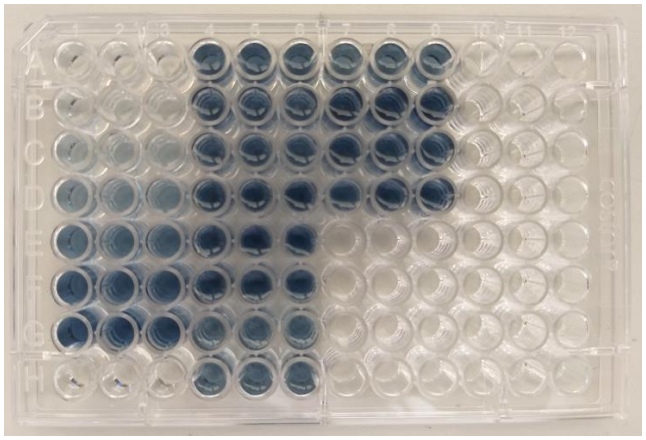

Figure 16. Microplate after reading TPC
Source: By the author

3.8. Total flavonoids content (TFC)

TFC were determined according to Costa et al. (2014). A colorimetric assay based on the formation of flavonoid-aluminium complex with a rosaceous colour in alkaline medium, as consequence of sample reaction with a reagent containing aluminium chloride and sodium nitrite. The aluminium cation forms stable complexes with flavonoids, causing a shift to higher wavelengths (bathochromic effect). A calibration curve, with increasing concentrations of standard solution catechin (0-400 µL/mL; R^2=0.999) was used in the determination. 1 mL of each extract/ standard/ blank test of ethanol were mixed with 4 mL of distilled water and 300 µL of sodium nitrate (1%). After 5 minutes, it was added 300 µL of aluminium chloride (5%). 1 minute after that, 2 mL of sodium hydroxide 1M and 2.5 µL of distilled water were also added. The absorbances were measured at 510 nm in a microplate (**Figure 17**) and the results expressed as catechin equivalents (CE).

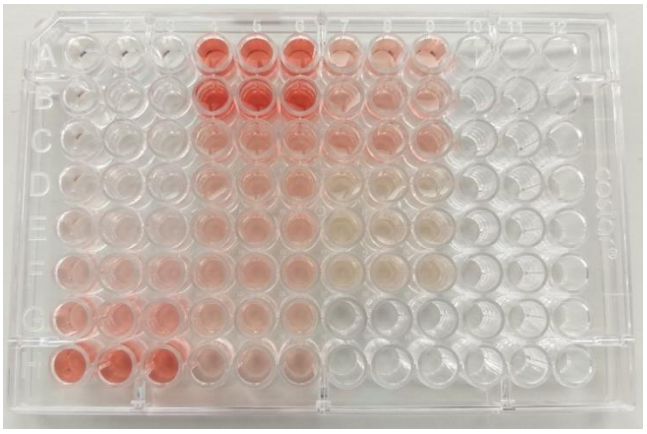

Figure 17. Microplate after reading TFC
Source: By the author

3.9. Ferric reducing antioxidant power (FRAP) assay:

The FRAP assay was carried out according to Costa et al. (2017) with modifications.

A calibration curve of a standard solution of ferrous sulphate with increasing concentrations (25–500 µmol/L; R^2=0.999) was used. Each extract and a blank test of deionised water were applied in the microplate, in triplicate. 35 µL of extract/deionised water was mixed with 265 µL of FRAP reagent (0.3 M acetate buffer, 10 mM TPTZ solution, and 20 mM of ferric chloride) and incubated at 37 ºC protected from light for 30 minutes. Then, absorbance was measured at 595 nm. The results were expressed as ferrous sulphate equivalents (FSE).

3.10. DPPH• scavenging activity

2,2-diphenyl-1-picrylhydrazyl (DPPH•) method is the most rapid, simple, and inexpensive of all free radical scavenging methods. This method is based on the principle that a hydrogen donor is an antioxidant. This colorimetric assay uses the DPPH• stable free radical, with a strong purple colour that changes to yellow when reduced in the presence of antioxidants. DPPH• is soluble in organic solvents and presents a typical absorption band at 525 nm. The antioxidant activity is measured by the decrease in absorption after reduction by the antioxidant. A large decrease in the absorbance at the endpoint of the reaction indicates strong free radical scavenging activity of the tested compound/extract.

DPPH• inhibition of the extracts was evaluated according to Costa et al. (2014). In a microplate, 30 µL of each extract were mixed with 270 µL of an ethanolic solution of DPPH•. The absorbance was measured at 525 nm for 40 minutes with intervals of 2 minutes in order to observe the kinetics reaction (**Figure 18**). A calibration curve of a standard solution of Trolox with increasing concentrations (5.62– 175.34 mg/L; R^2=0.998) was used.

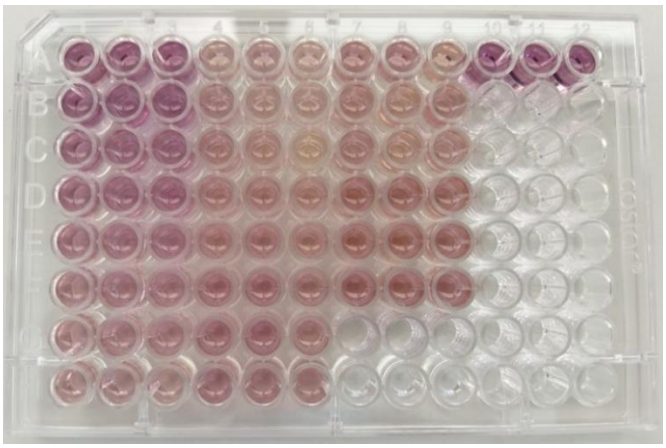

Figure 18. Microplate after reading DPPH scavenging activity
Source: By the author

3.11. Statistical analysis

Experiments were performed in triplicate. Statistical analysis was performed using IBM SPSS v. 25 (IBM Corp., USA). Data are expressed as mean ± standard deviation. One-way ANOVA was used to assess significant differences between samples, followed by Tukey's post hoc test to make pairwise comparisons between means. Statistical significance of the difference between two groups was evaluated by Student's t-test. The level of significance for all hypothesis tests (p) was 0.05.

3.12. Sensory analysis

The sensory evaluation of pasta samples was carried out in the place of usual consumption (at home). All the participants accepted voluntarily to do the evaluation and to be involved in the study. Each participant was given a bag containing the three pasta samples separated and properly identified with a three-digit number randomly generated by a computer program (**Figure 19**).

Figure 19. Bag containing the three pasta samples for sensory analysis
Source: By the author

Each participant was asked to cook the pasta at home, following the procedure:

- Use the labels provided to identify dishes according to the numbers on the pasta packages.
- Place the pan with water (one cup of tea or the equivalent of 250 mL) and put on fire.

- When the water is boiling, place pasta sample in the pan and add the salt (half a coffee spoon).
- Let it cook for 10 minutes.
- After cooking, drain the water, place the pasta sample on the corresponding plate and wrap the plate in aluminium foil to keep the pasta warm.
- Repeat the procedure for the remaining pastas.
- Start the test following the indications on the sensory analysis sheet.

A total of 71 participants evaluated the sensory properties of the three pasta samples, using a hedonic scale according to the intensity of preference. Participants were asked to evaluate product attributes as aspect, colour, shine, odour, taste, texture, flavour persistence and overall analysis. For each attribute, a 9-point hedonic scale, ranging from 1 (“extremely unpleasant”) to 9 (“extremely pleasant”) was used. The buying probability and preference order were also evaluated. For the buying probability, the scale ranged from 1 (“certainly not buying”) to 5 (“certainly buying”). The document used for sensory analysis is presented in Annex 1.

4. Results and Discussion

4.1. Nutritional profile

Table 3 presents the results concerning the nutritional value of the by-products samples.

Table 3. Nutritional analysis of OP (Fresh olive pomace), OP40ºC (Olive pomace powder dried at 40ºC), OP70ºC (Olive pomace powder dried at 70ºC) and CS (Coffee silverskin), in g/100g dw.

	OP	OP40˚C	OP70˚C	CS
Protein	4.25 ± 0.09 [b]	6.61 ± 0.09 [a]	6.59 ± 0.39 [a]	17.73 ± 0.09*
Fat	9.23 ± 0.26 [b]	16.69 ± 0.14 [a]	16.58 ± 0.19 [a]	3.10 ± 0.07*
Ash	4.03 ± 0.07 [c]	6.46 ± 0.07 [a]	5.65 ± 0.10 [b]	8.82 ± 0.02*
Total fiber	54.68 ± 0.54 [a]	47.31 ± 0.27 [b]	47.23 ± 0.05 [b]	59.95 ± 0.38*
Insoluble fiber	51.43 ± 0.31 [a]	42.66 ± 0.33 [b]	42.42 ± 0.53 [b]	54.64 ± 0.42*
Soluble fiber	3.25 ± 0.23 [a]	4.65 ± 0.06 [a]	4.80 ± 0.48 [a]	5.32 ± 0.03
Available Carbohydrates	27.81 ± 0.22 [a]	22.93 ± 0.16 [c]	25.09 ± 0.26 [b]	10.39 ± 0.10*

In each line, different letters represent significant differences (p<0.05) for the OP, OP40˚C and OP70˚C samples.
* represents significant differences (p<0.05) between OP70˚C (selected to prepare the pasta) and CS samples.

OP showed a high amount of total fiber (≈ 55 g/100g), specially of insoluble fiber (≈ 51 g/100g), and a considerable amount of fat (≈ 9 g/100g). When the drying process was applied, the obtained results were similar. The main component was total fiber and a considerable amount of fat for both heating treatments. There are significant differences between the OP40ºC and OP70ºC for ash and available carbohydrates. OP40ºC showed a significant higher result for ash while OP70ºC showed a significant higher result for available carbohydrates. Moreover, in dried samples there were significant increases in protein, fat, ash and soluble fiber compared to OP. There were also significant decreases in total fiber, insoluble fiber and available carbohydrates.

The results obtained for OP are different from the obtained results reported by Nunes et al. (2018). In that study it was obtained a higher result for protein (7.4 compared to 4.25 g/100g dw), and lower results for fat (5.8 compared to 9.23 g/100g dw) and ash (1.9 compared to 4.03 g/100g dw). On the other hand, more similar results were obtained by Nunes et al., 2020, especially in the olive pomace from Koroneiki cultivars. It was also obtained a higher result for protein (9.7 compared to 4.25 g/100g dw). Similar results were obtained for fat (9.7 compared to 9.23 g/100g dw) and ash (4.5 compared to 4.03 g/100g

dw). Several reasons can justify these differences between olive pomace results, for example: variations in climatic conditions, different olive varieties, different mills, and therefore different oil extraction methods.

Pasten et al. (2019) also tested a procedure to dry olive pomace at 40ºC and 70ºC. Their results reveal a significant increase in ash, and a significant decrease in fat and protein, with the increase of temperature. These results differs from those obtained in this study but can be explained due to the different times of exposure to that temperature and also to different types of olive pomace.

CS also showed a great amount of total fiber (≈ 60 g/100g), mostly due to the insoluble fiber (≈ 55 g/100g). The protein and ash content of CS is also considerable with approximately 18 g/100g and 9 g/100g, respectively. Comparing this by-product with the other that was chosen to incorporate into pasta, OP70ºC, it is possible to see that almost all parameters have significant differences. CS showed significant higher results for protein, ash, total fiber and insoluble fiber. On the other hand, OP70ºC showed significant higher results for fat and available carbohydrates.

Borrelli et al. (2004) found similar results for protein (18.6 compared to 17.73 g/100g dw), fat (2.2 compared to 3.10 g/100g dw), ash (7.0 compared to 8.82 g/100g dw) and insoluble fiber (53.7 compared to 51.43 g/100g dw). More different results were obtained for total fiber (62.4 compared to 54.68 g/100g dw) and soluble fiber (8.8 compared to 3.25 g/100 g dw).

The OP70ºC was selected to incorporate into pasta as it was a fast-drying procedure and the nutritional differences between it and OP40ºC were not critical. Moreover, it showed higher values for vitamin E, total phenolics, total flavonoids and higher antioxidant activity, as it will be discussed ahead.

Ten different formulations of pastas were tested but only the 3 best ones were analysed: one control pasta, one pasta enriched with olive pomace and one pasta enriched with coffee silverskin. Considering the control pasta formulations, both recipes resulted in a texture and aspect uniform, as well as an elastic and easy to handle dough. After cooking, both samples had a good aspect and were accepted at a preliminary tasting made by 4-5 people at the lab.

Pasta samples enriched with 10% of olive pomace showed an unpleasant taste and a granular aspect in the case of pasta sample made from wheat semolina. Pasta sample made from wheat semolina and enriched with 7.5% of olive pomace also showed a granular appearance and an acceptable flavour. However, pasta sample made from wheat flour and

enriched with 7.5% of olive pomace showed a uniform aspect as well as, an acceptable taste.

Pasta samples enriched with 10% of coffee silverskin showed an irregular and granular appearance, as well as an unpleasant taste and texture, with ingredients separation after cooking procedure. Pasta sample made from wheat semolina and enriched with 7.5% of coffee silverskin also showed a granular appearance and an unpleasant flavour. Pasta sample made from wheat flour and enriched with 7.5% of coffee silverskin showed a uniform aspect as well as, acceptable texture and flavour.

Considering these preliminary tasting results, pastas that were chosen to be analysed were: the pasta made from wheat flour enriched with 7.5% of coffee silverskin, the pasta made from wheat flour enriched with 7.5% of olive pomace and the wheat flour control pasta.

The pasta enrichment with olive pomace and coffee silverskin contributed to the increase of macronutrients. **Table 4** presents the results concerning the nutritional value of all pasta samples in g/100g dw and g/100g fw.

The RP showed a high result for available carbohydrates and a considerable amount of protein (g/100g dw). It showed low results for the other macronutrients as fat, ash and fiber (g/100g dw). After cooking, CP showed some significant differences compared to RP (g/100g dw). CP presented a significant higher value for protein while RP showed a significant higher value for ash. For the other parameters there were not significant differences between RP and CP.

Comparing RP with RPP in dw, there was a significant increase in fat (0.79 compared to 2.10 g/100g), ash (0.58 compared to 0.96 g/100g), total fiber (2.52 compared to 5.93 g/100g), soluble fiber (1.58 compared to 3.05 g/100g) and insoluble fiber (0.95 compared to 2.88 g/100g). There was also a significant decrease in protein and available carbohydrates. These results were expected as OP70ºC have a higher percentage of total fiber, insoluble fiber, soluble fiber, ash and fat compared to control pasta. As so, it was expected that 7.5% of this by-product would increase these parameters. On the other hand, as the value of protein and available carbohydrates is lower for OP70ºC compared to CP, it was expectable to obtain a decrease in these parameters. Comparing RPP with CPP in g/100g dw, there was a significant decrease of ash after cooking. On the other hand, there was also an increase in protein.

Table 4. Nutritional analysis results of RP (Raw control pasta), CP (Cooked control pasta), RPP (Raw olive pomace (7.5%) pasta), CPP (Cooked olive pomace (7.5%) pasta), RSP (Raw coffee silverskin (7.5%) pasta) and CSP (Cooked coffee silverskin (7.5%) pasta), in g/100g dw or g/100g fw.

	RP	CP	RPP	CPP	RSP	CSP		CP	CPP	CSP
	g/100g dw							g/100g fw		
Moisture	-	-	-	-	-	-		62.1 ± 0.8 ^{A}	42.5 ± 0.8 ^{C}	50.6 ± 0.8 ^{B}
Protein	9.64 ± 0.02 ^{c}	10.48 ± 0.15 ^{b}	9.11 ± 0.19 ^{d}	10.14 ± 0.13 ^{bc}	11.06 ± 0.20 ^{a}	11.29 ± 0.16 ^{a}		3.97 ± 0.06 ^{C}	5.84 ± 0.07 ^{A}	5.57 ± 0.08 ^{B}
Fat	0.79 ± 0.13 ^{b}	0.70± 0.02 ^{b}	2.10 ± 0.05 ^{a}	2.10 ± 0.05 ^{a}	0.69 ± 0.07 ^{b}	0.78 ± 0.01 ^{b}		0.27 ± 0.01 ^{C}	1.21 ± 0.03 ^{A}	0.39 ± 0.01 ^{B}
Ash	0.58 ± 0.01 ^{d}	0.44 ± 0.02 ^{e}	0.96 ± 0.01 ^{b}	0.75 ± 0.01 ^{c}	1.21 ± 0.01 ^{a}	0.99 ± 0.01 ^{b}		0.17 ± 0.01 ^{C}	0.43 ± 0.01 ^{B}	0.49 ± 0.01 ^{A}
Total fiber	2.52 ± 0.12 ^{b}	2.48 ± 0.18 ^{b}	5.93 ± 0.04 ^{a}	5.45 ± 0.43 ^{a}	7.13 ± 0.32 ^{a}	6.42 ± 0.91 ^{a}		0.94 ± 0.07 ^{B}	3.13 ± 0.25 ^{A}	3.52 ± 0.16 ^{A}
Insoluble fiber	0.95 ± 0.03 ^{c}	1.00 ± 0.02 ^{c}	2.88 ± 0.10 ^{b}	2.55 ± 0.36 ^{b}	5.24 ± 0.54 ^{a}	4.53 ± 0.22 ^{a}		0.38 ± 0.01 ^{C}	1.47 ± 0.20 ^{B}	2.24 ± 0.11 ^{A}
Soluble fiber	1.58 ± 0.15 ^{c}	1.48 ± 0.20 ^{c}	3.05 ± 0.05 ^{a}	2.90 ± 0.09 ^{a}	1.90 ± 0.22 ^{c}	1.89 ± 0.69 ^{c}		0.56 ± 0.08 ^{C}	1.67 ± 0.05 ^{A}	1.29 ± 0.05 ^{B}
Available Carbohydrates	86.46 ± 0.14 ^{a}	85.89 ± 0.18 ^{a}	81.90 ± 0.22 ^{b}	81.56 ± 0.18 ^{b}	80.62 ± 0.25 ^{c}	79.81 ± 0.16 ^{d}		32.56 ± 0.07 ^{C}	46.93 ± 0.10 ^{A}	39.40 ± 0.08 ^{B}

In each line, different lowercase letters represent significant differences (p<0.05) between samples with results expressed in dry weight.
In each line, different capital letters represent significant differences (p<0.05) between samples with results expressed in fresh weight.

Comparing RP with RSP, in dw, there was a significant increase in protein (9.64 compared to 11.06 g/100g), ash (0.58 compared to 1.21 g/100g), total fiber (2.52 compared to 7.13 g/100g) and insoluble fiber (0.95 compared to 5.24 g/100g). There were not significant differences on the other parameters. CS have a higher percentage of protein, ash, total fiber, insoluble fiber and soluble fiber compared to control pasta. As expected, the coffee silverskin incorporation increased protein, ash, total fiber and insoluble fiber. The decreases in fat and available carbohydrates were also expected due to the lower percentage of these macronutrients in CS compared to CP. After cooking, CSP showed significant decreases in ash and available carbohydrates compared to RSP.

Comparing RPP with RSP, the first have significant higher results in fat (2.10 compared to 0.69 g/100g), soluble fiber (3.05 compared to 1.90 g/100g) and available carbohydrates (81.90 compared to 80.62 g/100g) than RSP. On the other hand, RSP have better results in protein (11.06 compared to 9.11 g/100g), ash (1.21 compared to 0.96 g/100g) and insoluble fiber (5.24 compared to 2.88 g/100g). This means that RPP is richer in fat and soluble fiber than RSP. Differently, RSP present a better content in protein, ash and insoluble fiber.

These results were expected with the exception of total and soluble fiber. As previous mentioned, CS had significant higher results for protein, ash, total fiber and insoluble fiber while OP70ºC showed significant higher results for fat and available carbohydrates. It would be expected that RSP showed a higher result for total fiber compared to RPP. On contrast, it was not expected that soluble fiber would show a higher result in RSP, as there were not significative differences between OP70ºC and CS.

The results are also presented in fresh weight for the cooked pasta since it is the form that the food product is ingested. Compared to the control pasta, the cooked pasta results (g/100g fw), show that the incorporation of by-products lead to a significant decrease of the water absorbed during the cooking procedure. CP showed a moisture content of approximately 62 g/100g while CSP showed 51 g/100g and CPP showed 43 g/100g. The ready to eat CPP and CSP showed significant higher results for all the nutritional parameters compared to CP. CPP showed significant higher values of protein, fat, soluble fiber and available carbohydrates while CSP showed higher results of ash and insoluble fiber. It is possible to conclude that the incorporation of by-products improves the nutritional composition of pasta.

4.2. Vitamin E and fatty acids results

Vitamin E is a fat-soluble vitamin which is essential for the body function due to its antioxidant and anti-inflammatory properties. It is constituted by a group of lipophilic molecules, tocopherols and tocotrienols. This vitamin helps to improve the immune system and prevent diseases such as cardiovascular diseases and Alzheimer (Vardi et al., 2013). **Table 5** presents the results concerning the total vitamin E and vitamers of all by-products samples.

Table 5. Total vitamin E and vitamers results of OP (Fresh olive pomace), OP40ºC (Olive pomace powder dried at 40ºC), OP70ºC (Olive pomace powder dried at 70ºC) and CS (Coffee silverskin), in mg/kg.

	OP	OP40˚C	OP70˚C	CS
α-Tocopherol	42.46 ± 2.94 ^{c}	58.87 ± 1.04 ^{b}	70.83 ± 1.07 ^{a}	24.65 ± 0.42*
β-Tocopherol	1.36 ± 0.03 ^{c}	2.19 ± 0.02 ^{b}	2.48 ± 0.08 ^{a}	12.01 ± 0.42*
γ-Tocopherol	2.48 ± 0.09 ^{b}	3.90 ± 0.02 ^{a}	4.06 ± 0.05 ^{a}	1.81 ± 0.04*
γ-Tocotrienol	3.71 ± 0.21 ^{c}	6.12 ± 0.08 ^{b}	6.63 ± 0.08 ^{a}	ND
δ-Tocopherol	1.29 ± 0.01 ^{b}	2.25 ± 0.04 ^{a}	2.34 ± 0.04 ^{a}	ND
δ-Tocotrienol	ND	ND	ND	0.94 ± 0.05
Total Vit E	51.30 ± 3.26 ^{c}	73.33 ± 1.18 ^{b}	86.34 ± 1.21 ^{a}	39.40 ± 0.93*

In each line, different letters represent significant differences (p<0.05) for the OP, OP40ºC and OP70ºC samples. * represents significant differences (p<0.05) between OP70ºC and CS samples (selected to prepare the doughs).

The vitamin E profile of olive pomace samples is very similar. It was possible to identify 5 vitamers, α-tocopherol, β-tocopherol, γ-tocopherol, γ-tocotrienol and δ-tocopherol. Olive pomace showed an increase in total vitamin E and vitamers because of temperature. The highest result for total vitamin E was obtained for OP70ºC with approximately 86 mg/kg. This result is significantly higher compared to OP40ºC and OP results. The main vitamer present in olive pomace samples was α-tocopherol with approximately 43 mg/kg in OP, 59 mg/kg in OP40ºC and 71 mg/kg in OP70˚C. α-Tocopherol is very important because is the one demonstrating the highest biological activity (Cilla et al., 2014). For all vitamers, OP70ºC showed significantly higher results than OP. The results of α-tocopherol, β-tocopherol and γ-tocotrienol were also significantly higher in OP70ºC than in OP40ºC. These results show that OP70ºC was the best option of olive pomace samples, to introduce

into pasta. δ-tocotrienol was not identified in olive pomace samples but it was present in CS (**Table 5**).

The results for OP vitamers reported by Nunes et al. (2018) are different from the obtained results in this study. It was obtained higher results for α-tocopherol (76.4 mg/kg compared to 42.46 mg /kg dw, obtained in this study), and lower results for β-tocopherol (0.9 mg/kg compared to 1.36 mg/kg dw) and γ-tocopherol (1.3 mg/kg compared to 2.48 mg/kg dw). It was identified a vitamer that was not possible to identify in this study, α-tocotrienol. On the other hand, they did not identify γ-tocotrienol, δ-tocopherol and δ-tocotrienol. Nunes et al. (2020), also identified α-tocotrienol but did not identify γ-tocotrienol, δ-tocopherol and δ-tocotrienol. Their results are expressed in mg/g oil, so it is not possible to compare.

CS also showed lower amounts of total vitamin E compared to olive pomace samples, with only approximately 39 mg/kg. However, the vitamin E profile is different. It was possible to identify 4 vitamers, α-tocopherol, β-tocopherol, γ-tocopherol and δ-tocotrienol. The vitamer present in highest concentration was also α-tocopherol (≈ 25 mg/kg) followed by β-tocopherol (≈ 12 mg/kg). Comparing OP70ºC and CS, the results of α-tocopherol and γ-tocopherol were significantly higher in OP70ºC. On contrast, β-tocopherol showed a significant higher result in CS. Vitamers like γ-tocotrienol, and δ-tocopherol were only identified in OP samples while δ-tocotrienol was only identified in CS.

Table 6 presents the results concerning the total vitamin E and vitamers of all pasta samples, in dw and fw.

Considering the vitamin E profile of control sample, it was possible to identify 5 vitamers, α-tocopherol, β-tocopherol, γ-tocopherol, α-tocotrienol and β-tocotrienol. γ-tocotrienol was not present in these samples. The presence of these vitamers and vitamin E in control pasta is due to ingredient utilized, wheat flour. The vitamer present in highest concentration for the control samples was β-tocotrienol (≈ 5 mg/kg dw for RP and ≈ 8 mg/kg dw for CP). β-tocopherol and γ-tocopherol also presented significant higher results in CP compared to RP (≈ 0.7 mg/kg dw compared to ≈ 1.0 mg/kg dw and ≈ 0.5 mg/kg and 0.6 mg/kg, respectively). A higher result of total vitamin E was obtained for cooked pasta (CP) (≈ 10 mg/kg compared to ≈ 7 mg/kg).

Table 6. Total vitamin E and vitamers results of RP (Raw control pasta), CP (Cooked control pasta), RPP (Raw olive pomace (7.5%) pasta), CPP (Cooked olive pomace (7.5%) pasta), RSP (Raw coffee silverskin (7.5%) pasta) and CSP (Cooked coffee silverskin (7.5%) pasta), in mg/kg dw or fw.

	RP	CP	RPP	CPP	RSP	CSP		CP	CPP	CSP
	dw							fw		
α-Tocopherol	0.58 ± 0.02 ^{d}	0.69 ± 0.01 ^{d}	7.38 ± 0.11 ^{a}	7.32 ± 0.07 ^{a}	3.38 ± 0.11 ^{c}	4.19 ± 0.06 ^{b}		0.26 ± 0.00 ^{C}	4.21 ± 0.04 ^{A}	2.07 ± 0.03 ^{B}
α-Tocotrienol	0.28 ± 0.00 ^{d}	0.30 ± 0.00 ^{d}	1.49 ± 0.08 ^{a}	1.49 ± 0.02 ^{a}	0.77 ± 0.02 ^{c}	1.11 ± 0.08 ^{b}		0.11 ± 0.00 ^{C}	0.85 ± 0.01 ^{A}	0.55 ± 0.04 ^{B}
β-Tocopherol	0.68 ± 0.02 ^{e}	1.01 ± 0.01 ^{d}	2.10 ± 0.04 ^{b}	2.09 ± 0.02 ^{b}	1.95 ± 0.05 ^{c}	2.37 ± 0.01 ^{a}		0.38 ± 0.00 ^{C}	1.20 ± 0.01 ^{A}	1.17 ± 0.01 ^{B}
γ-Tocopherol	0.47 ± 0.01 ^{d}	0.60 ± 0.00 ^{b}	0.91 ± 0.01 ^{a}	0.91 ± 0.01 ^{a}	0.54 ± 0.01 ^{c}	0.56 ± 0.01 ^{c}		0.23 ± 0.00 ^{C}	0.52 ± 0.01 ^{A}	0.27 ± 0.00 ^{B}
β-Tocotrienol	4.92 ± 0.17 ^{e}	7.61 ± 0.04 ^{d}	14.14 ± 0.33 ^{a}	14.14 ± 0.19 ^{a}	9.51 ± 0.26 ^{c}	11.89 ± 0.07 ^{b}		2.89 ± 0.01 ^{C}	8.13 ± 0.11 ^{A}	5.87 ± 0.03 ^{B}
γ-Tocotrienol	ND	ND	1.01 ± 0.09 ^{a}	1.06 ± 0.09 ^{a}	ND	ND		ND	0.61 ± 0.05	ND
Total Vit E	6.93 ± 0.21 ^{e}	10.21 ± 0.04 ^{d}	27.03 ± 0.58 ^{a}	27.00 ± 0.35 ^{a}	16.15 ± 0.45 ^{c}	20.11 ± 0.07 ^{b}		3.87 ± 0.01 ^{C}	15.54 ± 0.20 ^{A}	9.93 ± 0.04 ^{B}

In each line, different lowercase letters represent significant differences (p<0.05) between samples with results expressed in dry weight.
In each line, different capital letters represent significant differences (p<0.05) between samples with results expressed in fresh weight.

Vitamin E profile of olive pomace pasta is different from control pasta. The γ-tocotrienol was identified in these samples due to the olive pomace incorporation. This vitamer was the second present in higher quantity for OP70ºC. The incorporation of olive pomace (OP70ºC) in pasta also resulted in a significant increase for all vitamin E vitamers, even for those that were not identified in OP70ºC like α-tocotrienol and β-tocotrienol. This is probably due to the temperature that pasta was subjected during the drying procedure. The bigger increases were in β-tocotrienol (14.14 mg/kg for RPP compared to 4.92 mg/kg for RP) and α-tocopherol (7.38 mg/kg for RPP compared to 0.58 mg/kg for RP). α-tocopherol was the vitamer present in higher concentration in OP70ºC, so this increase was expected. On contrast, β-tocotrienol was not even present in OP70ºC. These unexpected results are probably due to the matrix and the processing of pasta making. Olive pomace pasta showed a significantly higher result of total vitamin E compared to control pasta (27.03 mg/kg for RPP compared to 6.93 mg/kg for RP). There were not significant differences between RPP and CPP as consequence of the cooking procedure.

CS vitamin E profile is similar to the control pasta, it was also not possible to identify γ-tocotrienol in these samples. Even though, CS presented δ-tocotrienol while OP70ºC did not, the percentage incorporated into pasta was not enough to make it possible to identify this vitamer in pasta. The incorporation of coffee silverskin (CS) in pasta resulted in a significant increase for all vitamin E vitamers. The bigger increases were also in β-tocotrienol (9.51 mg/kg for RSP compared to 4.92 mg/kg for RP) and α-tocopherol (3.38 mg/kg for RSP compared to 0.58 mg/kg for RP). Once again, the α-tocopherol increase was expected as it is the major vitamer present in CS. However, β-tocotrienol was also not present in CS. The cooking procedure resulted in significant increases between RSP and CSP for almost all vitamin E vitamers except for γ-tocopherol. A significantly higher result of total vitamin E was obtained for coffee silverskin pasta compared to control pasta (16.15 mg/kg for RSP compared to 6.93 mg/kg for RP) in dry weight.

Comparing RPP with RSP, the vitamin E result was higher for RPP (27.03 mg/kg for RPP compared to 16.15 mg/kg for RSP). RPP showed higher results for all vitamers, and it was also identified one more vitamer in RPP compared to RSP, γ-tocotrienol.

Comparing cooked pasta samples in fw, the results show that the incorporation of by-products lead to a significant increase of vitamin E and vitamers. CPP showed higher values compared to CSP and was the one capable of introducing a vitamer that was not present in control sample, the γ-tocotrienol.

Fatty acids are SFA (saturated fatty acids), MUFA (monounsaturated fatty acids) or PUFA (polyunsaturated fatty acids) based on their double bonds between carbon atoms. SFA does not contain double bonds, MUFA contains one double bond and PUFA contains

two or more double bonds (Nutrition Information Centre University of Stellenbosch). A high consumption of foods rich in saturated fatty acids increase cholesterol levels and consequently the risk factors of CHD. Replacing SFA by PUFA or MUFA could help to reduce the risk (Nutrition Information Centre University of Stellenbosch).

The fatty acids profiles of the by-products and pastas were analysed. **Table 7** presents the results concerning the fatty acids of all by-products samples.

Table 7. Fatty acids results of OP (Fresh olive pomace), OP40ºC (Olive pomace powder dried at 40ºC), OP70ºC (Olive pomace powder dried at 70ºC) and CS (Coffee silverskin), in relative %.

	OP	OP40˚C	OP70˚C	CS
C14:0	ND	ND	ND	1.61 ± 0.10
C16:0	10.40 ± 0.06 [a]	10.59 ± 0.11 [a]	10.47 ± 0.15 [a]	25.25 ± 0.05*
C16:1	0.48 ± 0.04 [a]	0.54 ± 0.05 [a]	0.46 ± 0.06 [a]	ND
C18:0	3.38 ± 0.19 [a]	3.36 ± 0.24 [a]	3.65 ± 0.08 [a]	6.58 ± 0.15*
C18:1n9c	76.19 ± 0.24 [a]	76.28 ± 0.89 [a]	75.04 ± 0.09 [a]	10.02 ± 0.82*
C18:2n6c	8.70 ± 0.49 [a]	8.38 ± 0.69 [a]	9.44 ± 0.12 [a]	16.09 ± 0.71*
C20:0	0.31 ± 0.02 [a]	0.27 ± 0.04 [a]	0.32 ± 0.03 [a]	13.41 ± 0.25*
C18:3n3	0.45 ± 0.07 [a]	0.49 ± 0.07 [a]	0.53 ± 0.02 [a]	0.71 ± 0.04*
C20:1n9	ND	ND	ND	0.18 ± 0.01
C21:0	ND	ND	ND	0.51 ± 0.10
C22:0	ND	ND	ND	20.59 ± 0.50
C23:0	ND	ND	ND	0.56 ± 0.03
C24:0	0.07 ± 0.02 [a]	0.08 ± 0.01 [a]	0.09 ± 0.01 [a]	4.50 ± 0.17*
SFA	14.17 ± 0.19 [a]	14.30 ± 0.14 [a]	14.30 ± 0.14 [a]	73.01 ± 0.27*
MUFA	76.67 ± 0.27 [a]	76.82 ± 0.85 [a]	76.82 ± 0.85 [a]	10.20 ± 0.81*
PUFA	9.16 ± 0.46 [a]	8.88 ± 0.75 [a]	8.88 ± 0.75 [a]	16.79 ± 0.72*

In each line, different letters represent significant differences (p<0.05) for the OP, OP40ºC and OP70ºC samples. * represents significant differences (p<0.05) between OP70ºC and CS samples (selected to prepare the doughs).

The fatty acids profile is similar between olive pomace samples. It was possible to identify palmitic acid (C16:0), palmitoleic acid (C16:1), stearic acid (C18:0), oleic acid (C18:1n9c), linoleic acid (C18:2n6c), arachidic acid (C20:0), α-linolenic acid (C18:3n3) and lignoceric acid (C24:0). There were not significantly differences between olive pomace samples. The major fatty acid present is oleic acid (C18:1n9c) with a % relative of proximately 76% for OP and OP40ºC, and 75% for OP70ºC. As consequence, olive pomace samples have a great MUFA content. Oleic acid is a monounsaturated omega-9 fatty acid (Choulis, 2011). This fatty acid is important because it is considered one of the healthier sources of fat and can enhance the activity of antioxidants. Actually, in combination with antioxidants like tocopherols, it can prevent oxidation (Hernandez, 2016). It also can prevent heart disease and reduce cholesterol.

Nunes et al. (2018) obtained similar results for the same fatty acids identified, C16:0 (10.40 compared to 10.40%), C16:1 (0.48 compared to 0.68%), C18:0 (3.38 compared to 3.27%), C18:1n9 (76.19 compared to 75.25%), C18:2n6c (8.70 compared to 8.46%), C20:0 (0.31 compared to 0.42%), C18:3n3 (0.45 compared to 0.80%) and C24:0 (0.07 compared to 0.07%). However, they identify C14:0, C17:0 (Heptadecanoic acid), C22:0, C17:1 (cis-10-heptadecenoic acid) and C20:1n9 in OP, that were not possible to identify in this present study. In Nunes et al., 2020 study, they also identified the C14:0, C17:0, C22:0, and C20:1n9 in OP.

The fatty acids profile of CS is different than olive pomace samples. It was possible to identify myristic acid (C14:0), palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1n9c), linoleic acid (C18:2n6c), arachidic acid (C20:0), α-linolenic acid (C18:3n3), cis-11-eicosenoic acid (C20:1n9), henicosanoic acid (C21:0), behenic acid (C22:0), tricosanoic acid (C23:0) and lignoceric acid (C24:0). For CS, the major fatty acid present was palmitic acid (C16:0) with ≈ 25%, followed by behenic acid (C22:0) with ≈ 21%. Palmitic acid (16:0) is a saturated fatty acid present in the diet and synthesized endogenously (Innis, 2016). For a long time, it has been negatively considered for its adverse effects on chronic disease in adults (Carta et al., 2017). However, this fatty acid is an essential component of membrane with crucial roles in lipids transport and signal molecules, participating in many physiological activities (Innis, 2016). Behenic acid is a saturated fatty acid which does not significantly affect cholesterol concentrations (Cater and Garg, 1997). It is poorly absorbed due to its very long chain length and its low bioavailability (Cater and Denke, 2001). This fatty acid is important to maintain a good skin as it provides a protective barrier against the environment (Raghallaigh et al., 2012). CS is rich in SFA.

Borrelli et al. (2004) did not identified C21:0 and C23:0. On the other hand, they identified C15:0, C17:0, C18:1c-11 and C18:3n6. In that study it was obtained similar results for C14:0 (1.28 compared to 1.61%), C18:0 (6.38 compared to 6.58%) and C18:3n3 (0.88 compared to 0.71%). They obtained higher results for C16:0 (35.64 compared to 25.25%) and C18:2n6c (27.62 compared to 16.09%). Lower results were obtained in that study for C18:1n9 (5.77 compared to 10.02%), C20:0 (8.62 compared to 13.41%), C22:0 (10.70 compared to 20.59%) and C24:0 (1.03 compared to 4.50%).

Comparing the two samples incorporated into pasta, OP70ºC and CS, there are some differences in the fatty acids profile. In all olive pomace samples it was possible to identify palmitoleic acid (C16:1) that was not present in CS. On contrary, in CS it was possible to identify myristic acid (C14:0), cis-11-eicosenoic acid (C20:1n9), henicosanoic acid (C21:0), behenic acid (C22:0) and tricosanoic acid (C23:0), that were not present in olive pomace samples. In quantitative terms, there are also some significant differences. OP70º showed a significant higher result for oleic acid (C18:1n9c) while CS showed significantly higher results for palmitic acid (C16:0), stearic acid (C18:0), linoleic acid (C18:2n6c), arachidic acid (C20:0), α-linolenic acid (C18:3n3) and lignoceric acid (C24:0).

Table 8 presents the results concerning the fatty acids of all pasta samples.

The fatty acids profiles vary between samples. In RP and CP, it was possible to identify palmitic (C16:0), stearic (C18:0), oleic (C18:1n9c), linoleic (C18:2n6c), linolenic (C18:3n3) and cis-11-eicosenoic acids (C20:1n9). Palmitoleic acid (C16:1), arachidic acid (C20:0), behenic acid (C22:0) and lignoceric acid (C24:0) were not present in these samples. The fatty acid present in highest concentration for the control samples was linoleic acid (C18:2n6c) with approximately 60% for RP and 63% for CP. Control sample is richer in PUFA, representing more than 63%.

The olive pomace incorporation resulted in a modification on the fatty acids profile. The fatty acids profiles of RPP and CPP are different. In RPP, it was possible to identify palmitic acid (C16:0), palmitoleic acid (C16:1), stearic acid (C18:0), oleic acid (C18:1n9c), linoleic acid (C18:2n6c), arachidic acid (C20:0), α-linolenic acid (C18:3n3), cis-11-eicosenoic acid (C20:1n9) and behenic acid (C22:0). The incorporation of olive pomace in pasta, resulted in the presence of palmitoleic acid (C16:1), arachidic acid (C20:0) and behenic acid (C22:0) that were not present initially in RP. The latest fatty acid was not identified in OP70ºC, but the other results were expected. The enrichment also caused a quantitative increase in some fatty acids (C18:0 and C18:1n9c), while it caused a decrease in others (C16:0, C18:2n6c, C18:3n3 and C20:1n9).

Table 8. Fatty acids results of RP (Raw control pasta), CP (Cooked control pasta), RPP (Raw olive pomace (7.5%) pasta), CPP (Cooked olive pomace (7.5%) pasta), RSP (Raw coffee silverskin (7.5%) pasta) and CSP (Cooked coffee silverskin (7.5%) pasta), in relative %.

	RP	CP	RPP	CPP	RSP	CSP
	Relative %					
C16:0	19.93 ± 0.40 ^{b}	19.39 ± 0.09 ^{b}	13.63 ± 0.22 ^{d}	14.85 ± 0.35 ^{c}	20.85 ± 0.24 ^{a}	19.55 ± 0.26 ^{b}
C16:1	ND	ND	0.40 ± 0.01	ND	ND	ND
C18:0	1.54 ± 0.10 ^{c}	1.16 ± 0.07 ^{d}	2.78 ± 0.03 ^{a}	2.70 ± 0.26 ^{a}	1.91 ± 0.20 ^{b}	1.58 ± 0.13 ^{c}
C18:1n9c	14.68 ± 0.49 ^{c}	12.47 ± 0.46 ^{d}	53.39 ± 0.40 ^{a}	50.52 ± 0.23 ^{b}	13.36 ± 0.63 ^{d}	12.97 ± 0.56 ^{d}
C18:2n6c	60.32 ± 0.46 ^{b}	63.12 ± 0.70 ^{a}	27.61 ± 0.22 ^{f}	30.20 ± 0.51 ^{e}	55.59 ± 0.56 ^{d}	58.51 ± 0.39 ^{c}
C20:0	ND	ND	0.21 ± 0.02 ^{c}	ND	1.57 ± 0.13 ^{a}	1.20 ± 0.06 ^{b}
C18:3n3	3.12 ± 0.20 ^{ab}	3.20 ± 0.14 ^{ab}	1.55 ± 0.01 ^{c}	1.72 ± 0.03 ^{c}	3.28 ± 0.03 ^{a}	2.88 ± 0.22 ^{b}
C20:1n9	0.41 ± 0.03 ^{b}	0.67 ± 0.06 ^{a}	0.25 ± 0.02 ^{c}	ND	ND	0.44 ± 0.04 ^{b}
C22:0	ND	ND	0.17 ± 0.01 ^{c}	ND	2.46 ± 0.16 ^{a}	2.15 ± 0.09 ^{b}
C24:0	ND	ND	ND	ND	0.97 ± 0.10 ^{a}	0.72 ± 0.03 ^{b}
SFA	21.47 ± 0.49 ^{c}	20.55 ± 0.08 ^{d}	16.79 ± 0.20 ^{e}	17.56 ± 0.42 ^{e}	27.77 ± 0.12 ^{a}	25.20 ± 0.27 ^{b}
MUFA	15.09 ± 0.45 ^{c}	13.13 ± 0.50 ^{d}	54.04 ± 0.40 ^{a}	50.52 ± 0.23 ^{b}	13.36 ± 0.63 ^{d}	13.41 ± 0.60 ^{d}
PUFA	63.44 ± 0.46 ^{b}	66.32 ± 0.56 ^{a}	29.16 ± 0.22 ^{f}	31.92 ± 0.52 ^{e}	58.87 ± 0.58 ^{d}	61.40 ± 0.38 ^{c}

In each line, different letters represent significant differences (p<0.05) between samples with results expressed in dry weight.

These results were expected as the increases were verified to the fatty acids present in higher percentage for OP70ºC than in control pasta. A decrease was observed when the opposite occurs: higher percentage for those fatty acids in control pasta than in OP70ºC. After cooking it was not possible to identify palmitoleic acid (C16:1), arachidic acid (C20:0), cis-11-eicosenoic acid (C20:1n9) and behenic acid (C22:0). These fatty acids absence were probably due to the cooking process. The fatty acid present in highest concentration for RPP and CPP was oleic acid (C18:1n9c) with approximately 53% and 51%, respectively. These samples are richer in MUFA as well as OP70ºC, representing more than 50%. The presence of high percentages of MUFA offers advantages such as greater resistance to the drying process to which pastas are submitted and greater resistance to oxidation which contributes to a longer shelf life of the product.

The results obtained by Padalino et al. (2018) were different from those obtained in this study. They identified myristic acid (C14:0) but did not identify arachidic acid (C20:0), cis-11-eicosenoic acid (C20:1n9) and behenic acid (C22:0). The percentages of those fatty acids identified in both studies are also different. The fatty acid present in highest concentration in their study was also oleic acid but only with approximately 39% compared to 53%. Miriana Durante et al. (2019) obtained results more similar to ours, however they identified heptadecanoic acid (C17:0) that it was not possible to identify in this study. The fatty acid present in highest concentration in their study was also oleic acid with approximately 67% compared to 53%. These differences can be explained by the different raw materials utilized in pasta making and also the different percentage used to incorporate into pasta.

The coffee silverskin incorporation also resulted in a change of the fatty acids profile. The fatty acids profile of RSP and CSP are also different. In RSP, it was possible to identify palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1n9c), linoleic acid (C18:2n6c), arachidic acid (C20:0), α-linolenic acid (C18:3n3), behenic acid (C22:0) and lignoceric acid (C24:0). In CSP, it was also possible to identify cis-11-eicosenoic acid (C20:1n9). The coffee silverskin enrichment is responsible for the identification of arachidic acid (C20:0), behenic acid (C22:0) and lignoceric acid (C24:0). The fatty acid present in highest concentration for RSP and CSP was linoleic acid (C18:2n6c) as for the control samples. However, the results were lower with approximately 56% for RSP and 59% for CSP. These samples are richer in PUFA (polyunsaturated fatty acids) like the control sample. The enrichment also caused a significant quantitative increase in some fatty acids (C16:0 and C18:0), while it caused a decrease in others (C18:1n9c and C18:2n6c). Once again, the increases were verified in the fatty acids present in higher percentage for CS than in control pasta and decreases occurred when the contrary was verified as expected. After cooking, all fatty acids identified in both samples, RSP and CSP, suffered a significant decrease probably due to cooking process.

Comparing RPP and RSP, there are differences in terms of profile. In RPP it was possible to identify palmitoleic acid (C16:1) and cis-11-eicosenoic acid (C20:1n9) that were not present in RSP. On the other hand, it was possible to identify lignoceric acid (C24:0) in RSP and not in RPP. Quantitatively there were also differences. RPP showed significant higher values for stearic acid (C18:0) and oleic acid (C18:1n9c) while RSP showed higher values for palmitic acid (C16:0), linoleic acid (C18:2n6c), arachidic acid (C20:0), α-linolenic acid (C18:3n3), and behenic acid (C22:0). Overall, RSP is richer in PUFA than RPP (59% *versus* 32%) whereas RPP is richer in MUFA (54% *versus* 13%).

4.3. Antioxidant activity

Considering the richness in phenolic compounds of both by-products, the phytochemicals (total phenolics and flavonoids) and antioxidant activity (FRAP and DPPH) of by-products and pastas were analysed. **Table 9** shows the antioxidant results concerning all by-products samples.

Table 9. Total phenolics content (TPC), total flavonoids content (TFC) and antioxidant activity (FRAP assay FRAP and DPPH• scavenging activity) of OP (Fresh olive pomace), OP40ºC (Olive pomace powder dried at 40ºC), OP70ºC (Olive pomace powder dried at 70ºC) and CS (Coffee silverskin), in dw.

	OP	OP40˚C	OP70˚C	CS
TPC (g GAE/100 g)	3.92 ± 0.49 ^{a}	2.73 ± 0.15 ^{c}	3.38 ± 0.15 ^{b}	0.67 ± 0.04*
TFC (g CE/100 g)	3.00 ± 0.39 ^{b}	2.26 ± 0.13 ^{c}	3.46 ± 0.11 ^{a}	0.28 ± 0.02*
FRAP (g FSE/100 g)	5.37 ± 0.95 ^{b}	4.84 ± 0.34 ^{b}	7.49 ± 0.33 ^{a}	1.45 ± 0.08*
DPPH• (g Trolox/100 g)	1.50 ± 0.23 ^{a}	1.18 ± 0.09 ^{b}	1.27 ± 0.12 ^{ab}	0.15 ± 0.02*

In each line, different letters represent significant differences (p<0.05) for the OP, OP40˚C and OP70˚C samples.
* Represents significant differences (p<0.05) between OP70˚C and CS samples (selected to prepare the doughs).

Comparing olive pomace samples, OP was the one that showed higher value of TPC with ≈ 4 g GAE/100 g. The temperature resulted in a decrease of TPC in olive pomace. However, the OP70ºC presented a higher result than OP40ºC. This probably means that is not only the temperature that can influence the TPC but also the time of exposure to that temperature and to oxygen. OP70ºC showed a significantly higher result for TFC (≈ 3.5 g CE/100g). Lower results were obtained for OP and OP40ºC. The high temperature can degrade some compounds but can also result in the formation of some compounds. This main explain these higher results for OP70ºC compared to OP and OP40ºC. For FRAP assay, OP70ºC also presented a significantly higher result than OP and OP40ºC. On contrast, for DPPH• scavenging activity better results were obtained for OP, followed by OP70ºC. The results between these two samples are not significantly different.

In CS, it was possible to obtain results for all the parameters evaluated. However, the results were low. In fact, when comparing the two by-products incorporated into pasta, all the results obtained for CS were significantly lower than the obtained for OP70ºC.

To determine the best extraction procedure total phenolic contents of all pasta extracts were accessed and the higher results for all pastas were obtained for the extracts with 250

mg of sample in 40 mL of solvent. **Table 10** shows the antioxidant results concerning all pasta samples.

Control samples showed very low results for all the parameters. Actually, it did not even show antioxidant activity in DPPH•. The enrichment with olive pomace or coffee silver skin resulted in higher results for all parameters. The biggest increase was for olive pomace pasta, both raw and cooked.

For olive pomace pastas the results of all parameters were significantly higher compared to the control pasta. These results are expected as OP70ºC is rich in phenolic compounds (**Table 9**). Despite of the low percentage of enrichment, only 7.5%, it was enough to make significative differences. Comparing RPP and CPP in dw, values are significantly lower for TPC, TFC and FRAP assay for CPP. There were no significative differences between these pastas for DPPH• results. This means that a part of these bioactive compounds is lost or degraded during the cooking procedure. In the results obtained by Simonato et al. (2019), it is also possible to observe the decrease in TPC and DPPH values after the cooking procedure.

The enrichment with coffee silverskin also resulted in significant higher results for all parameters, compared to the control. These results were also expected as CS showed higher results than control pasta. Although, the increase in these compounds and antioxidant activity was lower than the one that was verified for pasta enriched with olive pomace. Furthermore, after cooking the result of TPC was significantly lower and it was not possible to detect antioxidant activity in DPPH• assay. TFC and FRAP results did not show significant differences between RSP and CSP in dw. Part of these bioactive compounds that are a result of coffee silverskin incorporation, are also lost or degraded during the cooking procedure.

Comparing all cooked pastas in fw, which shows the real “ready-to-eat”, it is possible to see that the enrichment with by-products led to an increase of phenolic compounds and flavonoids, as well as for the antioxidant activity (FRAP assay). The results were significantly higher for CPP compared to CSP and beyond that, CSP was not capable of reducing the radical DPPH•

Table 10. Total phenolics content (TPC), total flavonoids content (TFC) and antioxidant activity (FRAP assay FRAP and DPPH• scavenging activity) of RP (Raw control pasta), CP (Cooked control pasta), RPP (Raw olive pomace (7.5%) pasta), CPP (Cooked olive pomace (7.5%) pasta), RSP (Raw coffee silverskin (7.5%) pasta) and CSP (Cooked coffee silverskin (7.5%) pasta), in dw or fw

	RP	CP	RPP	CPP	RSP	CSP		CP	CPP	CSP
	dw							fw		
TPC (g GAE/100g)	0.09 ± 0.01 ^{d}	0.03 ± 0.00 ^{e}	0.24 ± 0.01 ^{a}	0.20 ± 0.01 ^{b}	0.13 ± 0.01 ^{c}	0.10 ± 0.01 ^{d}		0.011 ± 0.003 ^{C}	0.114 ± 0.006 ^{A}	0.048 ± 0.005 ^{B}
TFC (g CE/100 g)	0.002 ± 0.00 ^{d}	0.007 ± 0.00 ^{d}	0.234 ± 0.017 ^{a}	0.212 ± 0.016 ^{b}	0.030 ± 0.002 ^{c}	0.026 ± 0.002 ^{c}		0.003 ± 0.000 ^{C}	0.122 ± 0.009 ^{A}	0.013 ± 0.001 ^{B}
FRAP (g FSE/100 g)	0.28 ± 0.01 ^{d}	0.25 ± 0.01 ^{d}	3.47 ± 0.18 ^{a}	3.01 ± 0.13 ^{b}	1.10 ± 0.05 ^{c}	0.99 ± 0.05 ^{c}		0.096 ± 0.004 ^{C}	1.732 ± 0.072 ^{A}	0.491 ± 0.025 ^{B}
DPPH• (g Trolox/100 g)	ND	ND	0.10 ± 0.02 ^{a}	0.09 ± 0.02 ^{a}	0.01 ± 0.00 ^{b}	ND		ND	0.05 ± 0.01	ND

In each line, different lowercase letters represent significant differences (p<0.05) between samples with results expressed in dry weight.
In each line, different capital letters represent significant differences (p<0.05) between samples with results expressed in fresh weight.

4.4. Sensory analysis results

Sensory evaluation is a scientific method that uses human senses (sight, smell, touch, taste, and hearing) as an instrument of analysis and provides unique information about a food product acceptance (Yang and Lee, 2019). The descriptive tests used were helpful to identify specific sensory attributes present in pasta samples and the preference test showed the consumer acceptance of a product. With this sensory evaluation, it was possible to include people's perceptions and predict the consumer acceptance.

Figure 20 shows the gender distribution of the 71 consumers. The majority of consumers were women (38 women and 33 men).

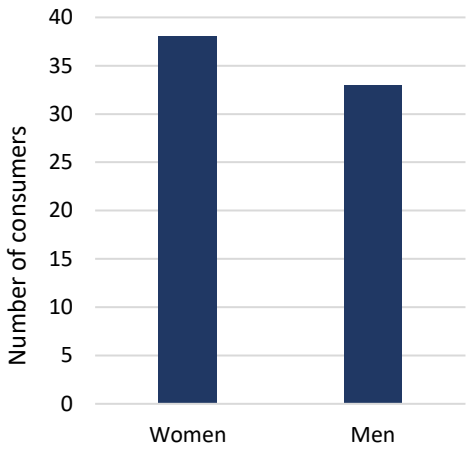

Figure 20. Gender distribution of the 71 evaluating consumers

The consumers ages varied among ≤18 and 69 years old, with predominance of individuals with ages between 31 and 49 years (**Figure 21**).

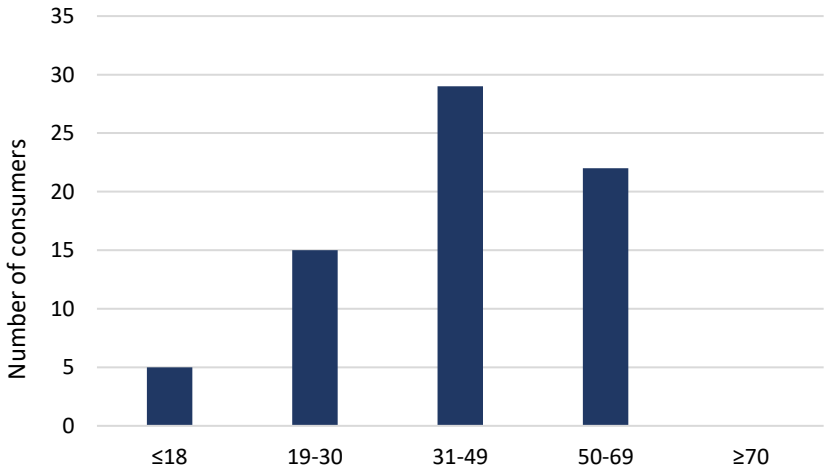

Figure 21. Age distribution of the 71 evaluating consumers

Figure 22 shows the frequency of pasta consumption. The great majority of consumers, consume pasta "frequently". Some consumers consume pasta only "occasionally" or "rarely", but no one answered "never".

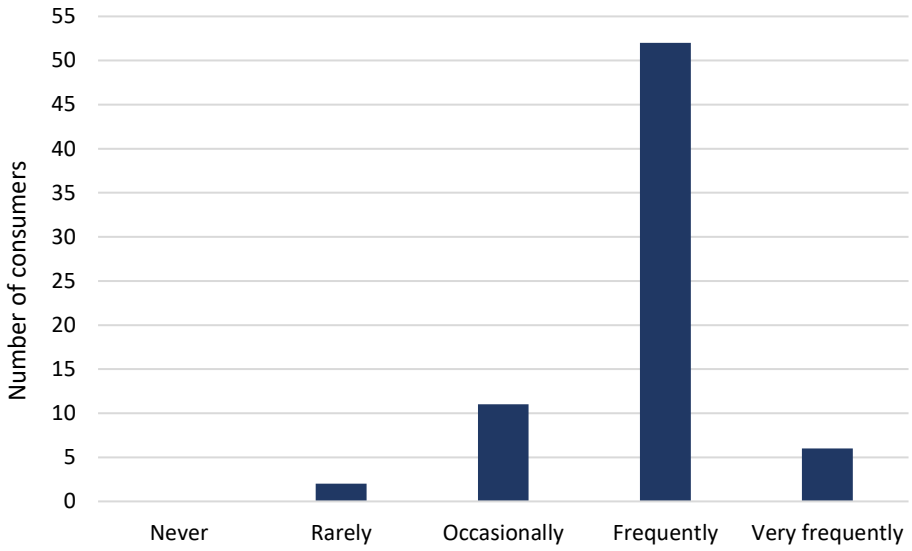

Figure 22. Frequency of pasta consumption distribution of the 71 consumers

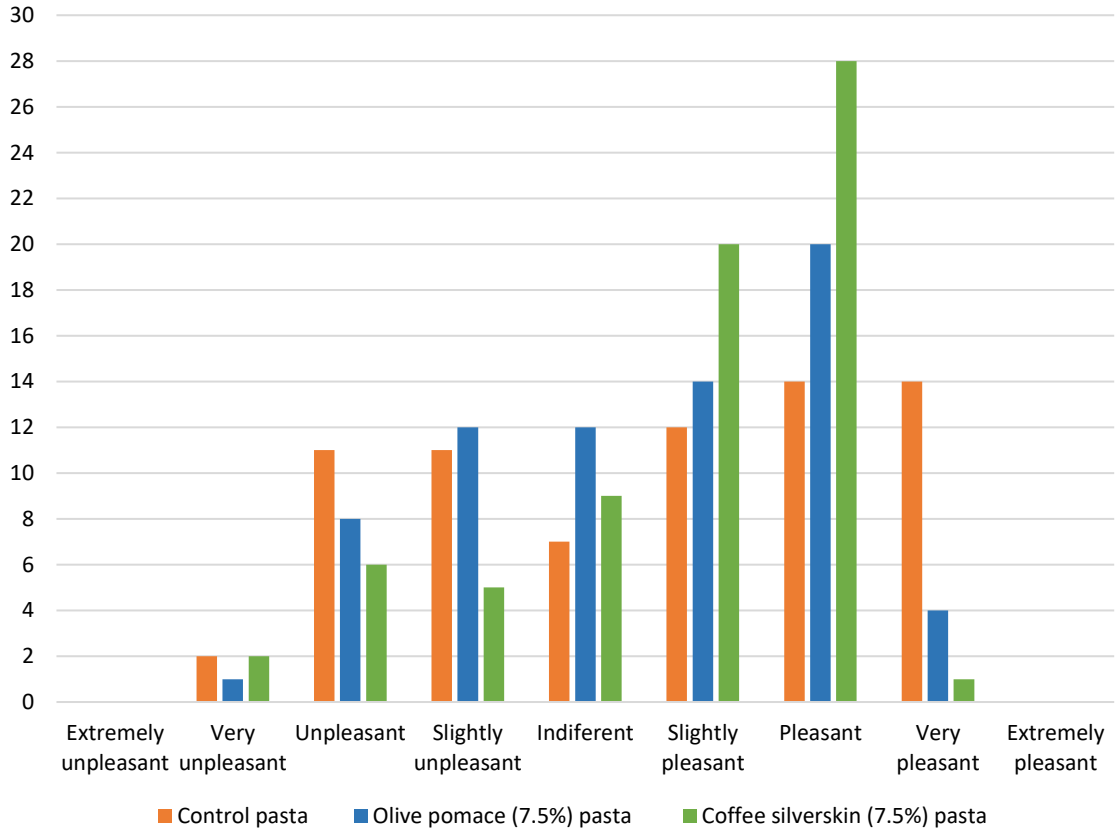

Figure 23. Evaluation scores for aspect

Independently of the formulation, most of the tasters liked the appearance of pastas (**Figure 23**). The coffee silverskin formulation was more appreciated considering aspect than the control pasta, since 49 individuals liked the appearance compared to the 40 of control pasta and 38 of olive pomace pasta. Some consumers also found “indifferent” the aspect of pasta (12 consumers on olive pomace pasta, 9 consumers on coffee silverskin pasta and 7 consumers on control pasta). Consumers disliked more the aspect of the control pasta than olive pomace and coffee silverskin pasta. This is a strong indicator of the potential acceptability of the addition of coffee silverskin to pasta. No consumer has classified pastas as “Extremely unpleasant” or “Extremely pleasant”.

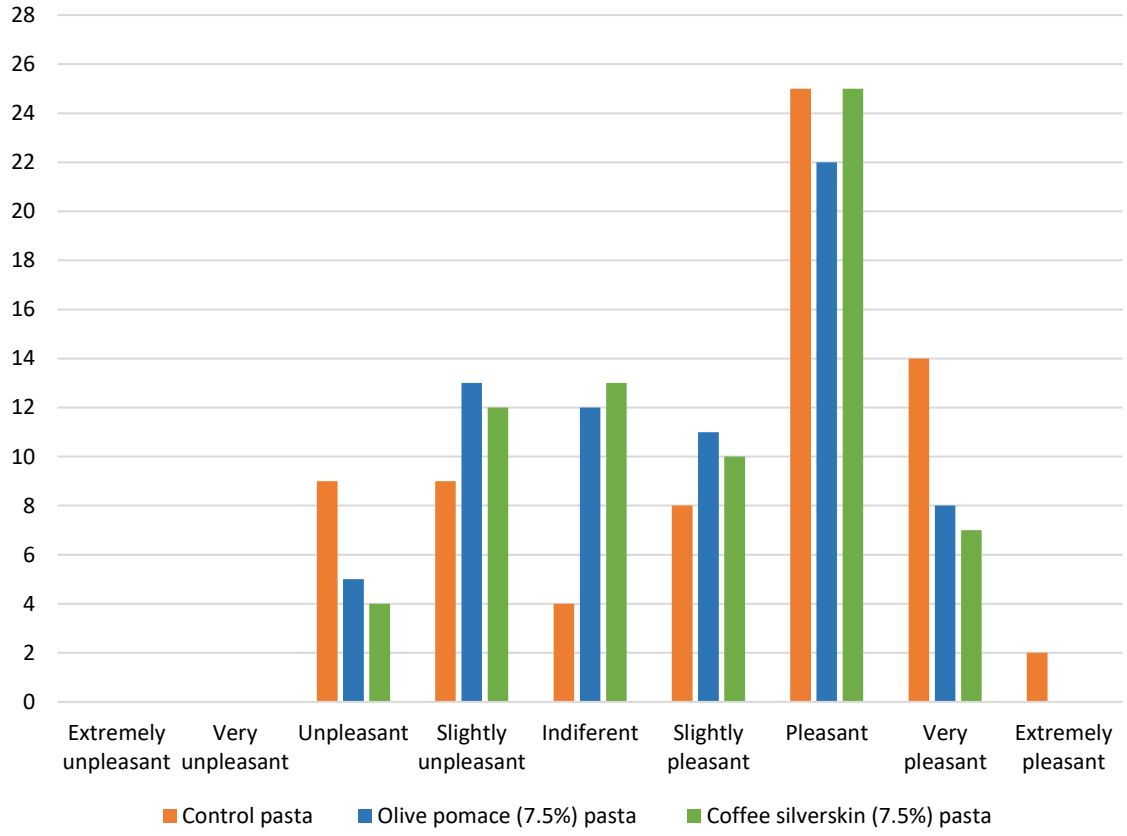

Figure 24. Evaluation scores for colour

Similar to the appearance most of the consumers liked the colour of all pastas (**Figure 24**). However, in this case the most appreciated was the control pasta probably because it has a more similar colour to those present in the market and individuals are used to consume that. 49 consumers liked the control pasta while 42 liked the coffee silverskin pasta and 41 liked the olive pomace pasta. Some consumers also found “indifferent” the colour of pasta (13 consumers on coffee silverskin pasta, 12 consumers on olive pomace pasta and 4 consumers on control pasta). Less colour rejection of consumers was reported by coffee silverskin pasta. The control pasta and olive pomace pasta reported similar

rejection. Pasta colour was not classified as “Extremely unpleasant” or “Very unpleasant”. For control pasta, there were 2 consumers that classified it as “Extremely pleasant”.

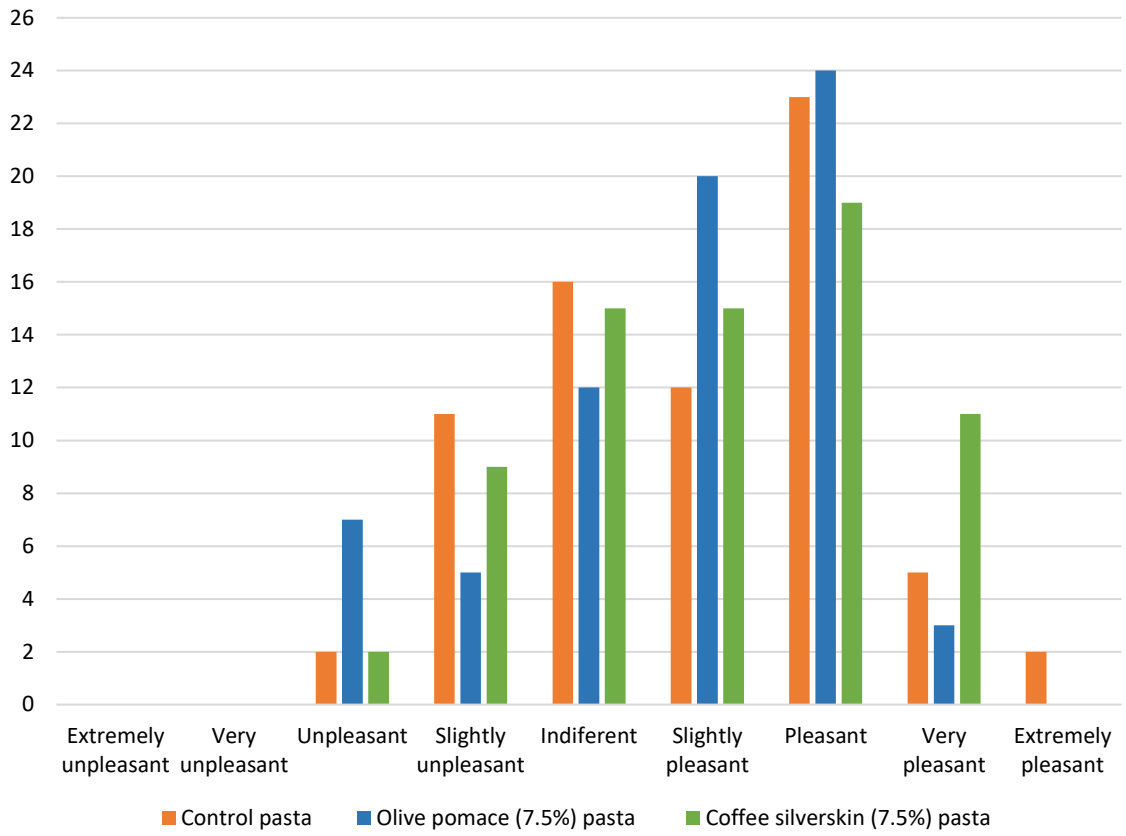

Figure 25. Evaluation scores for shine

Most consumers also liked the shine of all pastas (**Figure 25**). In this case, the most appreciated was also de olive pomace pasta with 47 consumers attributing more than 5 in a 9 points scale. Olive pomace pasta was followed by coffee silverskin pasta with 45 consumers liking the shine of pastas. The least appreciated was control pasta but on the other hand it was the one to get 2 consumers that classified it as “Extremely pleasant”. Some consumers also found “indifferent” the shine of pasta (16 consumers on control pasta, 15 consumers on coffee silverskin pasta and 12 consumers on olive pomace pasta). Considering the pasta shine, less rejection of consumers was also reported by coffee silverskin pasta followed by olive pomace pasta. Control pasta was the one with higher rejection. Pasta shine was not classified as “Extremely unpleasant” or “Very unpleasant”.

Most consumers liked the odour of all pastas (**Figure 26**). Olive pomace pasta and control pasta were the most appreciated with 51 consumers liking it. Only 42 consumers liked the odour of coffee silverskin pasta. Some consumers also found “indifferent” the odour of pasta (17 consumers on control pasta, 12 consumers on coffee silverskin pasta and 10

consumers on olive pomace pasta). Less rejection of consumers was reported by control pasta followed by olive pomace pasta. 17 consumers rejected the odour of coffee silverskin pasta. No consumer classified pasta odour as “Extremely unpleasant”.

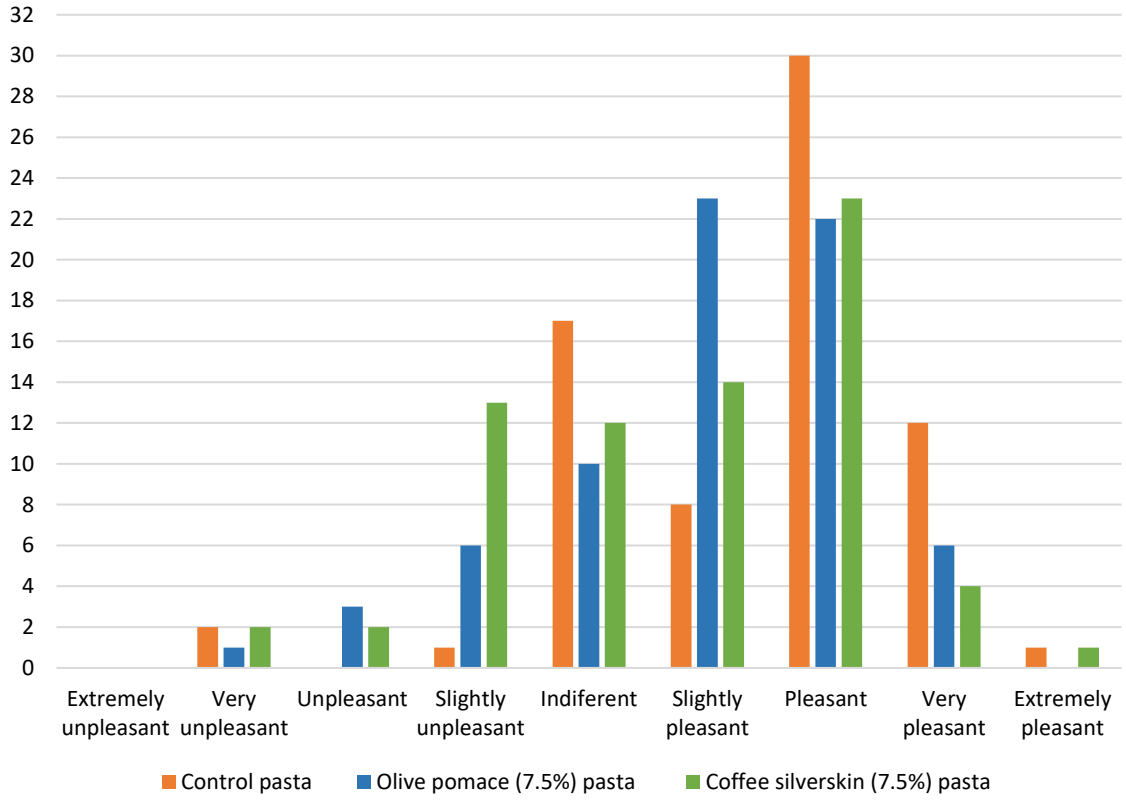

Figure 26. Evaluation scores for odour

Considering flavour, there were differences between the control sample and enriched pastas (**Figure 27**). The most appreciated pasta was the control with 51 consumers liking it. Only 30 and 37 consumers liked the flavour of coffee silverskin pasta and olive pomace pasta, respectively. Some consumers also found “indifferent” the flavour of pasta (13 consumers on control pasta, 4 consumers on olive pomace pasta and 10 consumers on coffee silverskin pasta). Less rejection of consumers was reported by olive pomace pasta. Olive pomace pasta and coffee silverskin pasta reported a similar rejection with 37 and 32 consumers disliking it. Olive pomace pasta and coffee silverskin pasta were in fact classified as “Extremely unpleasant” by 9 and 2 consumers, respectively.

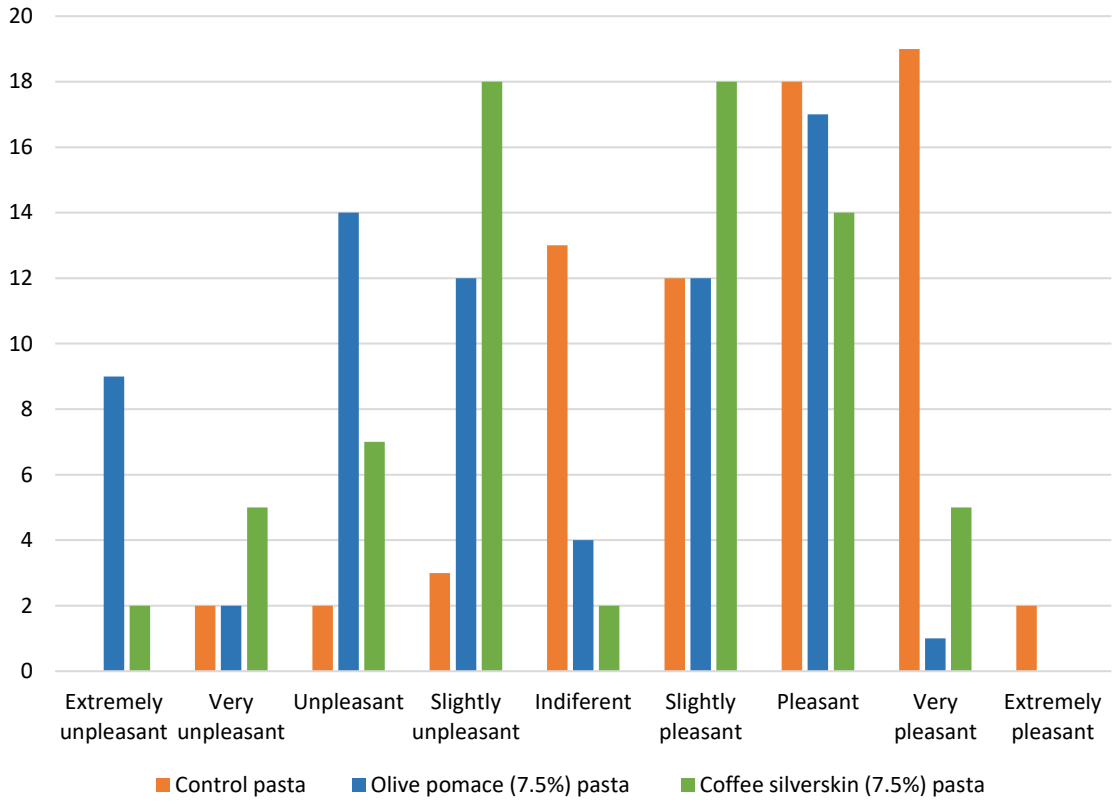

Figure 27. Evaluation scores for flavour

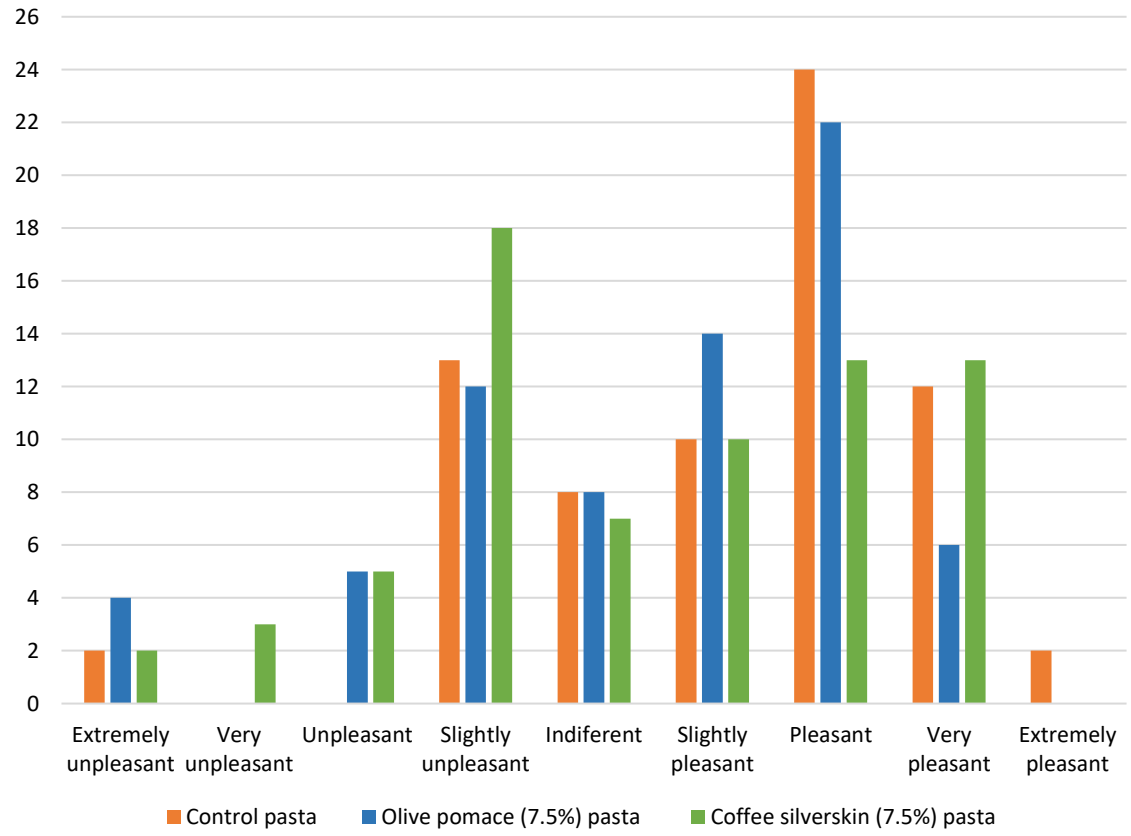

Figure 28. Evaluation scores for texture

Most consumers liked the texture of all pastas (**Figure 28**). The most appreciated was also the control pasta with 48 consumers liking it. Consumers liked the texture of olive pomace pasta than the coffee silverskin pasta. Some consumers also found “indifferent” the texture of pasta (8 consumers on control pasta and on olive pomace pasta and 7 consumers on coffee silverskin pasta). Less rejection of consumers was reported by control pasta, followed by olive pomace pasta. However, there were more consumers to classify olive pomace pasta as “Extremely pleasant” than coffee silverskin pasta. Control pasta was the only classified as “Extremely pleasant”.

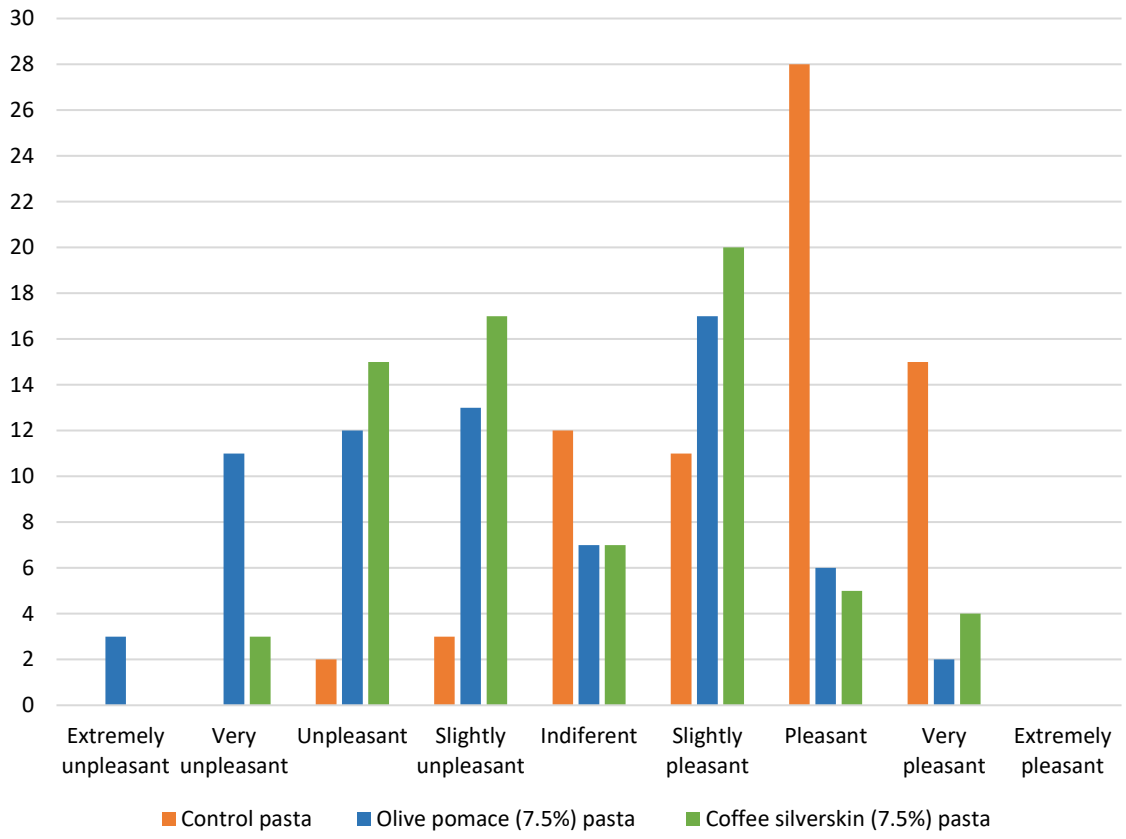

Figure 29. Evaluation scores for flavour persistence

As the **figure 29** show, the flavour persistence of control pasta was the most liked by consumers, with 54 consumers attributing a positive score. Coffee silverskin pasta and olive pomace pasta only had 29 and 25 consumers liking it which is less than half consumers that participated in this study. Some consumers found the flavour persistence “Indifferent” (12 consumers for control pasta and 7 for coffee silverskin pasta and olive pomace pasta). The most disliked pasta was the olive pomace pasta with 39 consumers attributing a negative score, with 3 consumers classifying it as “Extremely pleasant”. 35 consumers also attributed a negative score to coffee silverskin pasta. Pasta flavour persistence was not classified as “Extremely pleasant”.

Regarding the global analysis (**Figure 30**), consumers preferred the control pasta with 53 consumers attributing a positive score to this parameter. Less than half consumers liked the coffee silverskin pasta and olive pomace pasta (30 and 28 consumers, respectively). There were also some consumers that found it "indifferent", 11 consumers for control pasta, 8 consumers for coffee silverskin pasta and 4 consumers for olive pomace pasta. The most disliked sample was the olive pomace pasta followed by coffee silverskin pasta. A consumer actually classified olive pomace pasta as "Extremely unpleasant" while control pasta was classified it as "Extremely pleasant" by 2 consumers.

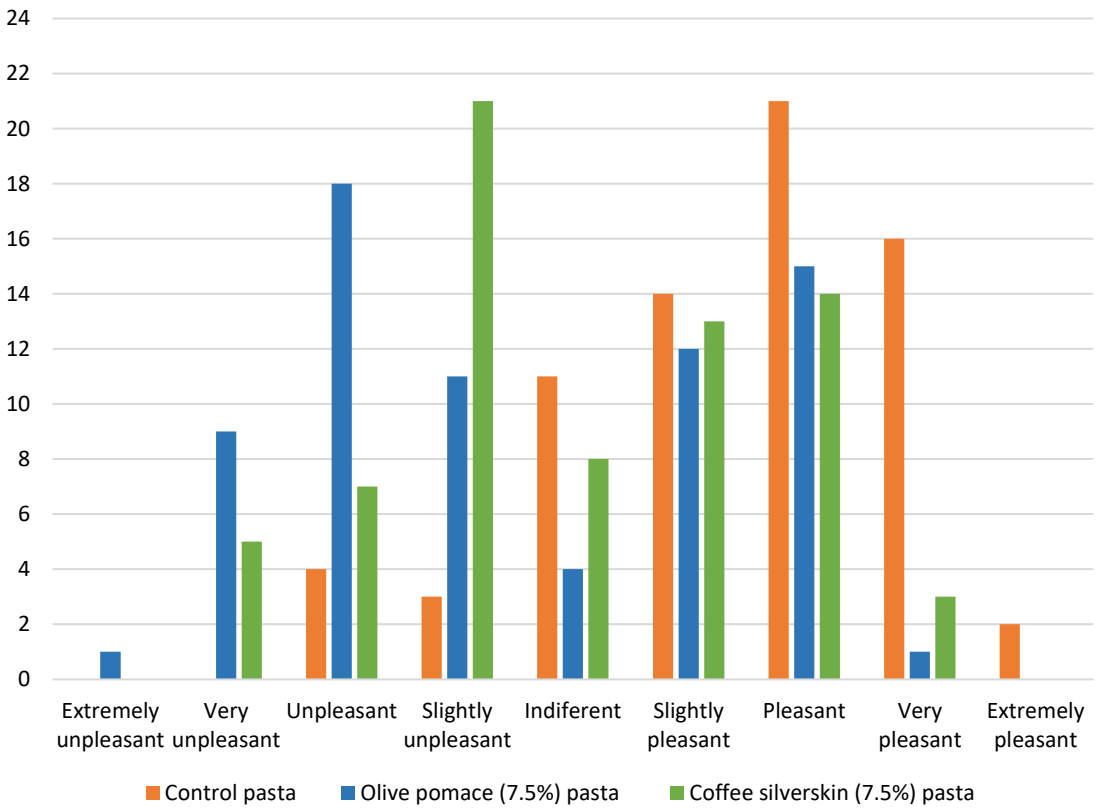

Figure 30. Evaluation scores for global analysis

Considering the buying probability all pastas were classified with all options (**Figure 31**). The control pasta was the one with better probability of buying followed by coffee silverskin pasta. Olive pomace pasta was the sample with less probability of consumers buying.

Figures 32 and **figure 33** shows the results of the preference test. As it is possible to see, consumers preferred the control pasta. This result is expected as it is the most similar pasta to the ones present in the market. However, there were also consumers that preferred coffee silverskin pasta or olive pomace pasta and chose these samples as their favourite. As a second preference consumers preferred coffee silverskin pasta to olive pomace pasta.

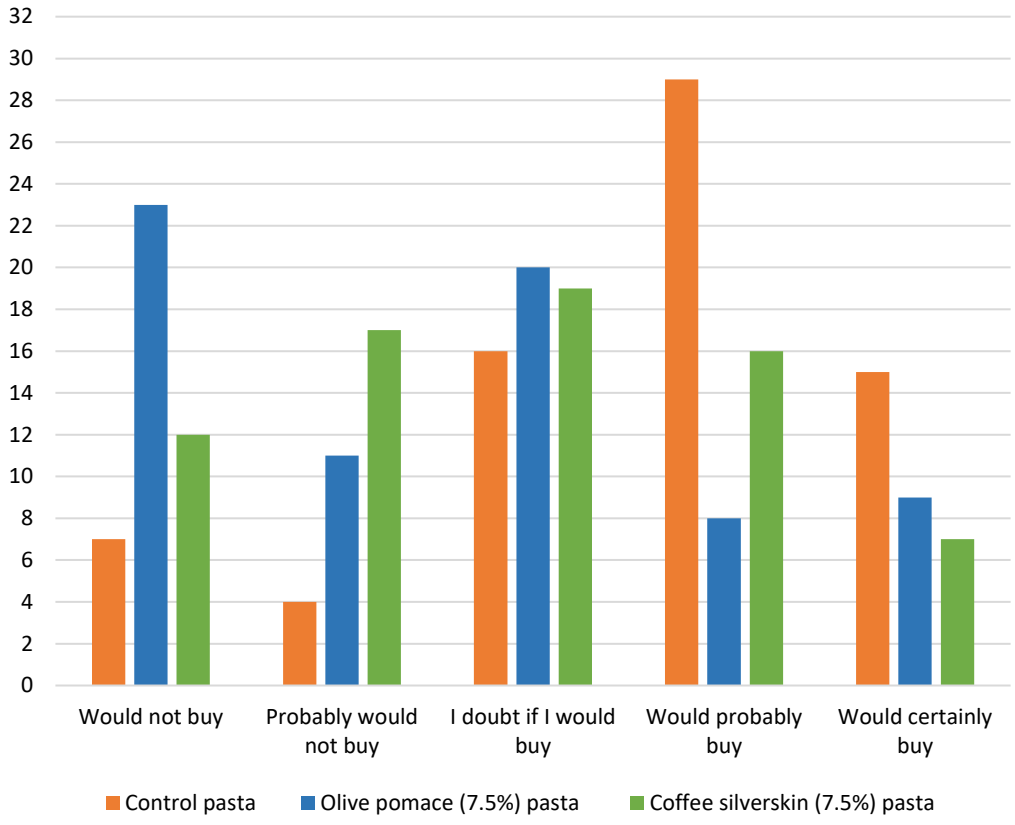

Figure 31. Evaluation scores for buying probability

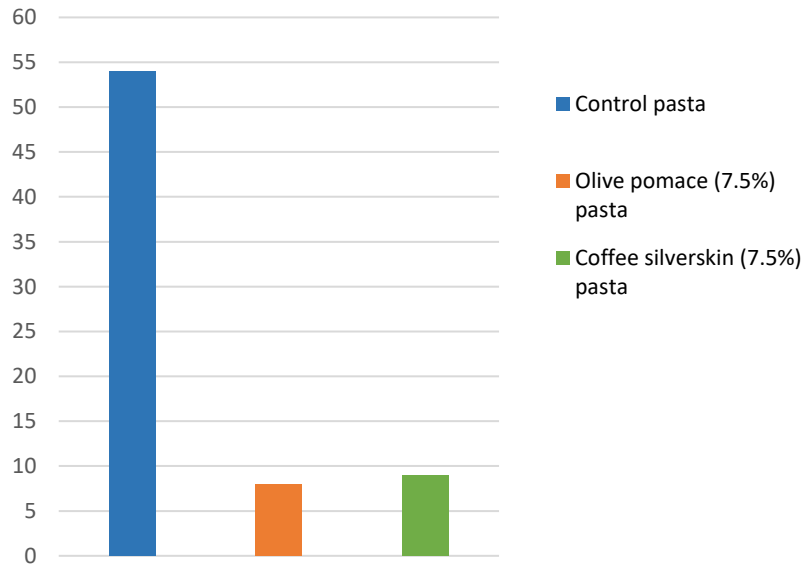

Figure 32. First preference of consumers

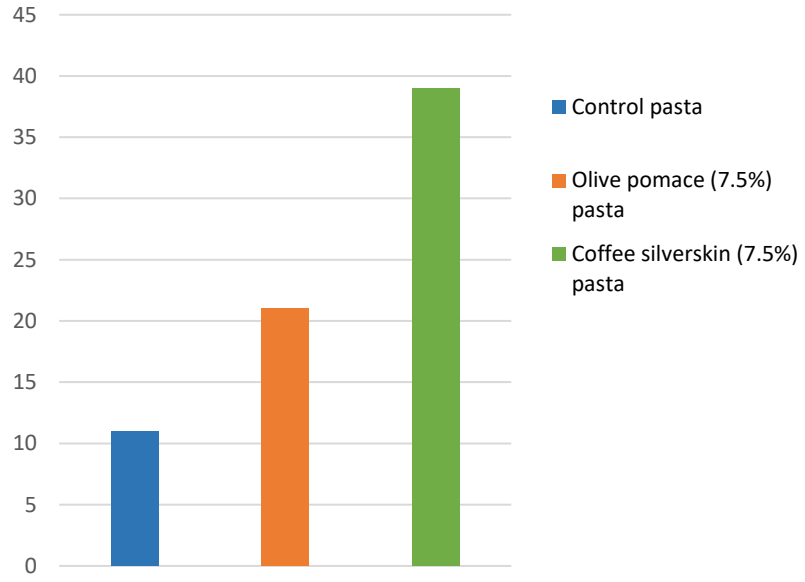

Figure 33. Second preference of consumers

In **Figure 34**, it is possible to see the relation between parameters and samples. Very similar results were obtained for parameters like aspect, colour, and shine between pastas. In odour and texture, the control pasta started to show better results than enriched pastas. The difference is even more remarkable in flavour, flavour persistence, global analysis and buying probability. Comparing the enriched pastas, better results were obtained for coffee silverskin pasta in parameters like aspect, flavour, flavour persistence, global analysis and buying probability. Odour was the only parameter that olive pomace pasta showed better results compared to the coffee silverskin pasta.

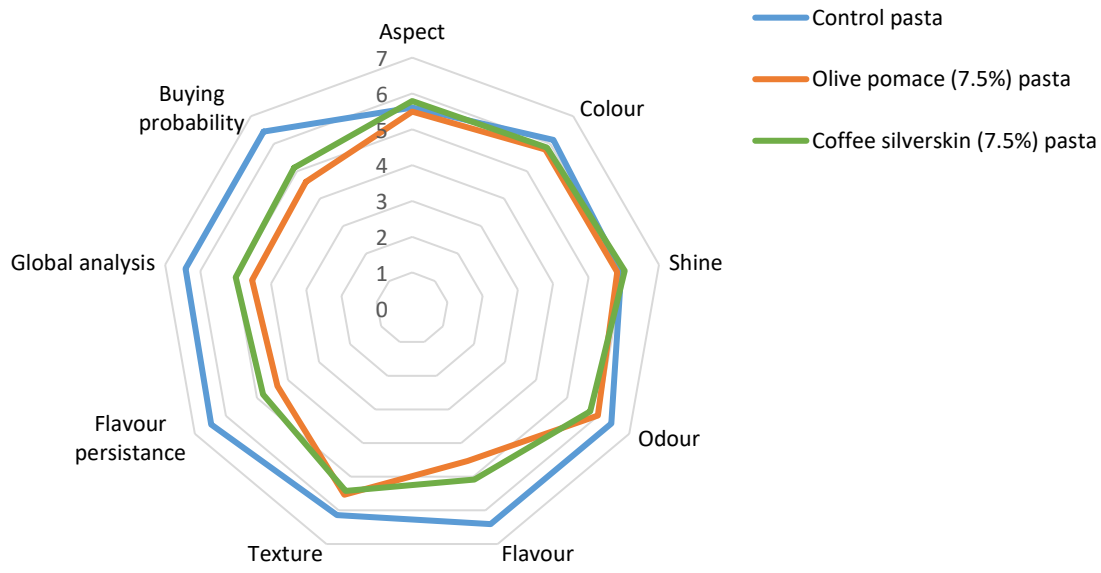

Figure 34. Overall scores for the evaluated criteria (Results obtained for "Buying probability" were multiplied by a 9/5 factor)

5. Conclusions

It is possible to conclude that olive pomace is rich in total fiber, especially insoluble fiber and has a considerable amount of fat. The main vitamer present is α-tocopherol and the major fatty acid present is oleic acid. The process of drying and removing the stone increases protein, fat, ash and soluble fiber, but also decreases total fiber, insoluble fiber and available carbohydrates. This process does not affect the vitamin E profile, but cause an increase in total vitamin E and vitamers, with highest results for OP70˚C. The fatty acids profile is also not affected and neither result in quantitative differences. Olive pomace shows a higher value of TPC, and better DPPH scavenging activity comparing to OP70˚C that presented a higher content than OP40˚C. The highest result of TFC and FRAP assay was obtained for OP70˚C.

The enrichment of pasta with olive pomace contributes to the increase of fat, ash, total fiber, soluble fiber and insoluble fiber, with higher results in fat, soluble fiber and available carbohydrates than coffee silverskin enrichment. Regarding the vitamin E, incorporation of olive pomace in pasta also resulted in an increase for all vitamin E vitamers and a change in the profile. Olive pomace pasta is richer in total vitamin E compared to control pasta and coffee silverskin enriched pasta. Considering fatty acid profile, the pasta enrichment results in some variations with the presence of palmitoleic, arachidic and behenic acids as well as, an increase in some fatty acids. The results of all antioxidant parameters are higher in olive pomace pasta compared to the control pasta. However, these bioactive compounds are partially lost or degraded during the cooking procedure.

On the other hand, coffee silverskin is rich in total fiber (mostly insoluble fiber), protein and ash. Nutritionally, coffee silverskin has higher content for protein, ash, total fiber and insoluble fiber while OP70ºC shows significant higher results for fat and available carbohydrates. The vitamer present in highest concentration is also α-tocopherol but coffee silverskin has a lower amount of total vitamin E and a different profile compared to olive pomace samples. The fatty acids profile of coffee silverskin differs from olive pomace samples, with the major fatty acids present in coffee silverskin being palmitic and behenic acids. Coffee silverskin also showed lower results than olive pomace samples for all antioxidant parameters accessed.

The enrichment with coffee silverskin resulted in the increase of protein, ash, total fiber and insoluble fiber. This enrichment has better results in protein, ash and insoluble fiber than olive pomace enrichment. The incorporation of coffee silverskin in pasta results in an increase for all vitamin E vitamers and total vitamin E. It also makes a difference in the fatty acids profile but the fatty acid present in highest concentration is the same as in the control

pasta sample. The enrichment caused differences in terms of profile and in quantitative compared to the olive pomace enrichment. Pasta enriched with coffee silverskin is richer in PUFA while the pasta enriched with olive pomace pasta richer in MUFA. The enrichment with coffee silverskin also results in significant higher results for all antioxidant parameters, compared to the control but after cooking, they are also lost or degraded.

Overall, antioxidant properties are not a benefit of pasta enrichment with these by-products. On the other hand, they contribute to an improvement of the nutritional profile by increasing macronutrients, vitamin E and make a good difference in the fatty acids profile.

Despite all the nutritional and chemical benefits described, sensory analysis revealed that consumers preferred control pasta, followed by coffee silverskin pasta. However, regarding the aspect, the coffee silverskin pasta was preferred to the control pasta. Olive pomace pasta was preferred by consumers in what respects to the shine.

More studies are needed in order to improve the enriched pastas and to obtain a higher consumers approval. It would be interesting to test a pasta enriched with a mixture of the two by-products, coffee silverskin and olive pomace, since they are complementary. Nevertheless, this study proves that it is possible to utilize these by-products and develop new food products with great nutritional advantages.

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Annex I

FICHA DE ANÁLISE SENSORIAL

(Assinale com um X)

Sexo: ☐ Masculino ☐ Feminino

Idade: ☐ ≤ 18 anos ☐ 19-30 anos ☐ 31-49 anos ☐ 50-69 anos ☐ ≥ 70 anos

Com que frequência costuma consumir massa?

☐ Nunca ☐ Raramente ☐ Ocasionalmente ☐ Frequentemente ☐ Muito frequentemente

Antes da degustação

(Assinale com um X no quadrado que mais se enquadra na sua análise)

Aspeto:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável; 4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável; 7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Cor:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável; 4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável; 7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Brilho:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável;
4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável;
7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Cheiro:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável;
4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável;
7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Durante a degustação

(Assinale com um X no quadrado que mais se enquadra na sua análise)

Sabor:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável;
4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável;
7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Textura:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável;
4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável;
7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Persistência do sabor:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável;
4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável;
7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Após a degustação

(Assinale com um X no quadrado que mais se enquadra na sua análise)

Análise global:

1-Extremamente desagradável; 2-Muito desagradável; 3-Desagradável;
4- Ligeiramente desagradável; 5-Indiferente; 6-Ligeiramente agradável;
7-Agradável; 8- Muito agradável; 9-Extremamente agradável;

737	1	2	3	4	5	6	7	8	9
647	1	2	3	4	5	6	7	8	9
566	1	2	3	4	5	6	7	8	9

Probabilidade de compra:

1-Não compraria; 2-Provavelmente não compraria; 3-Tenho dúvidas se compraria;
4-Provavelmente compraria; 5-Certamente compraria;

737	1	2	3	4	5
647	1	2	3	4	5
566	1	2	3	4	5

Ordene por preferência todas as amostras:

(Colocar o número no quadrado pela ordem correspondente)

Menos preferível				Mais preferível

Obrigada.