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## ***VALORIZACIÓN DE RESIDUOS HORTOFRUTÍCOLAS COMO INGREDIENTES FUNCIONALES EN POLVO. REVISIÓN SOBRE LA INFLUENCIA DE PRETRATAMIENTOS, PROCESADO Y DIGESTIÓN IN VITRO SOBRE LOS COMPUESTOS BIOACTIVOS PRESENTES EN SUBPRODUCTOS DE COL, ZANAHORIA, APIO Y AJO PUERRO***

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# VALORIZACIÓN DE RESIDUOS HORTOFRUTÍCOLAS COMO INGREDIENTES FUNCIONALES EN POLVO. REVISIÓN SOBRE LA INFLUENCIA DE PRETRATAMIENTOS, PROCESADO Y DIGESTIÓN IN VITRO SOBRE LOS COMPUESTOS BIOACTIVOS PRESENTES EN SUBPRODUCTOS DE COL, ZANAHORIA, APIO Y AJO PUERRO

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## ABSTRACT

Food waste is a worldwide concern as it represents a constant threat to the environment and a serious operational problem for the food industry. The by-products of fruits and vegetables being a valuable source of bioactive compounds have the potential to be reused and reintroduced in the agri-food chain. This circular approach contributes to a sustainable production system. In this context, the Institute of Food Engineering for Development (IUIAD) of the Universitat Politècnica de València (UPV) and the agricultural cooperative Agrícola Villena, work together on an eco-innovation project that seeks to integrally valorize the vegetables waste generated, particularly cabbage, carrot, celery and leek. The objective is to transform vegetable wastes into functional powder ingredients and be able to apply them in food formulations in order to improve their nutritional profile, contributing to a sustainable healthy diet. Through an exhaustive bibliographic review, this research studies the influence of pretreatments, processing and *in vitro* digestion on the bioactive compounds of vegetable residues, with the aim of identifying the appropriate production parameters to achieve an adequate functional and physicochemical profile of the final powders. It was evidenced that there is a growing interest in developing functional foods from residues, that dehydration is a suitable technology for integral recovery that may benefit from the use of pretreatments, and that the results of *in vitro* digestion are influenced by processing conditions.

KEY WORDS: bio-waste valorization, dehydration, functional food, food powders, pretreatments, *in vitro* digestion, bioaccessibility.

## RESUMEN

El desperdicio de alimentos es motivo de preocupación en todo el mundo, ya que representa una amenaza constante para el medio ambiente y un grave problema operativo para la industria alimentaria. Los subproductos de frutas y verduras son una valiosa fuente de compuestos bioactivos, los cuales tienen el potencial de ser reutilizados y reintroducidos a la cadena alimentaria. Este enfoque circular contribuye a un sistema de producción sostenible. Es por ello que el Instituto de Ingeniería de Alimentos para el Desarrollo (IUIAD) de la

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Universitat Politècnica de València (UPV) y la cooperativa Agrícola Villena, colaboran en un proyecto que persigue la valorización integral de los residuos de hortalizas, particularmente col, zanahoria, apio y ajo puerro. El objetivo es transformarlos en ingredientes funcionales en polvo y luego poder aplicarlos en formulaciones alimentarias con el fin de mejorar su perfil nutricional, y contribuir a una dieta saludable y sostenible. A través de una exhaustiva revisión bibliográfica, la presente investigación estudia la influencia de los pretratamientos, el procesado y la digestión *in vitro* sobre los compuestos bioactivos presentes en los desechos vegetales seleccionados, con el objetivo de identificar los parámetros de producción apropiados para lograr un adecuado perfil funcional y fisicoquímico de los polvos finales. Se evidenció que existe un creciente interés en desarrollar alimentos funcionales a partir de residuos, que la deshidratación es una tecnología adecuada para el aprovechamiento integral que puede beneficiarse de la aplicación de pretratamientos y que los resultados de la digestión *in vitro* están influenciados por las condiciones de proceso.

**PALABRAS CLAVE:** valorización de residuos, deshidratación, alimentos funcionales en polvo, pretratamientos, digestión *in vitro*, bioaccesibilidad.

## **RESUM**

El desperdici d'aliments és motiu de preocupació en tot el món, ja que representa una amenaça constant per al medi ambient i un greu problema operatiu per a la indústria alimentària. Els subproductes de fruites i verdures són una valuosa font de compostos bioactius i tenen el potencial de ser reutilitzats i reintroduïts a la cadena alimentària. Este enfocament circular contribuïx a un sistema de producció sostenible. És per això que l'Institut d'Enginyeria d'Aliments per al Desenrotllament (IUIAD) de la Universitat Politècnica de València (UPV) i la cooperativa Agrícola Villena, col·laboren en un projecte amb la fi de valoritzar integralment els residus generats, en particular de col, carlota, api i all porro. L'objectiu és transformar-los en ingredients funcionals en pols i després poder-los aplicar en formulacions alimentàries a fi de millorar el seu perfil nutricional, i contribuir a una dieta saludable i sostenible. A través d'una exhaustiva revisió bibliogràfica, aquesta investigació estudia la influència dels pretractaments, el processament i la digestió *in vitro* sobre els compostos bioactius presents en els residus vegetals seleccionats, amb l'objectiu d'identificar els paràmetres de producció apropiats per a aconseguir un adequat perfil funcional i fisicoquímic de les pols finals. En aquest treball s'ha evidenciat que hi ha un creixent interès a desenrotllar aliments funcionals a partir de residus, que la deshidratació és una tecnologia adequada per a l'aprofitament integral però que requerix de pretractaments capaços d'alleujar els seus efectes adversos i que els resultats de la digestió *in vitro* estan influenciats per les condicions de processament.

**PARAULES CLAU:** valorització de residus, deshidratació, pretractaments, aliments funcionals, pols d'ús alimentari, digestió *in vitro*, bioaccesibilitat.

## 1. INTRODUCTION

Food industry waste represents a constant threat to the environment and a serious operational problem for the respective production plants (Goula and Lazarides, 2015). It is a matter of concern worldwide, as a substantial amount of food that could have been consumed ends up as residues. Food waste (FW) generation takes place throughout the whole supply chain (Mirabella et al., 2014). During the processing of food, the edible parts of raw materials are decreased due to discard or degradation. Many of them have the potential to be reused, but fruit and vegetable residues are usually underused and considered as low-value material. This represents a loss of material with high biological and nutritional value. According to the Food and Agriculture Organization (FAO) of the United Nations, approximately a third of the edible parts of food produced for human consumption are lost or wasted globally. This amount represents approximately 1,300 million tons per year (Galanakis, 2012). In 2016 it was estimated that FW in the EU was around 88 million tons representing an associated cost of 143 billion euros (Stenmarck et al., 2016). Furthermore, the respective consumption of resources and the emission of pollutants implies an increase in the environmental burden.

Considering that final storage in landfills is not a sustainable option (Ojeda, 2009), it is necessary to focus production systems in terms of minimizing waste, and/or reintroducing it into the production chain. For these reasons, concepts such as “circular economy”, “industrial ecology” and “zero waste economy”, among others, have arisen to orient eco-innovation towards the use of waste as raw material for the development of new products and applications (Mirabella et al., 2014). Among the measurements adopted by the EU against FW, the Sustainable Development Goal 12 (“Ensure sustainable consumption and production patterns”), adopted in September 2015, targets to halve per capita FW at the retail and consumer level by 2030. The reduction of FW has an enormous potential for reducing the resources used to produce it, saving money and decreasing the environmental impact (Stenmarck et al., 2016). This also represents an important contribution to the Circular Economy Action Plan adopted by the European Commission within the European Green Deal, which promotes sustainable initiatives along the entire life cycle of products. A circular approach benefits sustainable food production (EU, 2019).

As defined by the FAO (2019), a sustainable food system must be able to ensure sustainable consumption and production patterns, as well as deliver safe, healthy, and nutritious diets. Accordingly, the food industry must incorporate processes and products which provide both a lower environmental impact and an increased positive impact on diets and health. Reformulation of processed foods provides a genuine opportunity to improve people’s health by modifying the nutritional characteristics of commonly consumed processed foods (Bas-Bellver et al., 2020), therefore addressing another one of the FAO sustainable development goals: “Ensure healthy lives and promote well-being for all at all ages”. This is in accordance with the definition of a sustainable healthy diet, which states that food consumption should be adequate in energy

and nutrients for growth and development, meeting the needs of an active and healthy life. This concept states that dietary patterns should promote all dimensions of individuals' health and wellbeing, while having low environmental impact and reducing food loss (FAO and WHO, 2019).

Increased awareness of the association between improper diet and health disorders has led to the development of a healthier food industry. Recommendations based on scientific evidence include increasing the consumption of plant-based foods, basically fruits and vegetables, since they represent excellent sources of fiber and other bioactive compounds (Rao and Rao, 2007). These are perishable commodities with high moisture content (~80%) which deteriorate in a short period of time when not handled properly. Dehydration is a suitable method to extend the shelf life of these foods, since partial water removal halts the growth of spoiling microorganisms. Thus, the current food industry shows a growing interest in the application of dried fruits and vegetables in powder form (Karam et al., 2016). In 2006 Zhang et al., already mentioned the rapid growth of this market. Applications of fruit and vegetable powders include their use as ingredients in the confectionary, bakery, sweet, and distilling industries to improve the nutritional value of foodstuff (Camire et al., 2007), but they also have application in others such as dressings, soups or ready-to-eat products. Applications of fruit and vegetable powders is not limited to the use of the whole food, but the waste generated in the transformation industry is also a valuable source of bioactive compounds such as fiber, antioxidants and other phytochemicals (Tseng & Zhao, 2013). Fruit and vegetables waste powders can also be used to fortify food products and improve their technological properties (Elleuch et al., 2011). In this way, FW can be considered a source of valuable components at a low cost.

In this context, the cooperative Agrícola Villena and the Institute of Food Engineering for Development (IUIAD) of the Universitat Politècnica de València (UPV) collaborate in a project to recover the waste generated in the vegetables lines and reuse it as raw material in the production of powder ingredients with functional properties. The project, which is funded by the European Agricultural Fund for Rural Development within the Rural Development Program 2014-2020 of the Valencian Community (Spain), aims to define the processing conditions better preserving the functional properties of the waste generated in the manufacturing lines of four selected vegetables: cabbage, carrot, celery and leek. These vegetables, were chosen considering the ease of obtaining a clean (or easy to wash) residue, as well as according to the expected bioactive components. The annual production of these vegetable residues estimated by the Villena Agri-Food Cooperative is ~ 178 tons in the case of leeks (some of the white part and the green leaves on top) and ~ 250 tons in the case of cabbage (outer leaves). The amount of carrot residues generated is significantly greater (~ 3000 tons) since most of this residue comes from the ready-to-eat line or IV Range, in which the discard percentages increase dramatically (Bas-Bellver et al., 2020). According to the cooperative, the amount of leek residue produced last year was around ~ 1500 tons, including discarded product. This project seeks to reach an integral use

of the by-products by applying different dehydration technologies, and it is not focused on extracting the bioactive compounds, thus avoiding solvent extraction and the generation of other residues.

This Master's thesis seeks to contribute to the achievement of the objectives of the previously mentioned project, through an exhaustive bibliographic review that allows detailing the appropriate process conditions for obtaining food powders as functional ingredients. This is highly relevant because the physical, chemical, nutritional, functional and organoleptic profile of the final product, as well as its possible subsequent applications, is largely determined by the processing conditions. The purpose of this research is to provide scientific bases for the optimal integral recovery of vegetable residues, and reintroduce them in the food chain, so as to contribute to food chain circularity and the development of sustainable food diets.

Thus, considering the heterogeneity and the different macrostructural, microstructural and compositional characteristics existing among the different vegetables, as well as the impact of the processing conditions on the final characteristics of the food powders, the present research has been divided into three categories which allows the objectives to be addressed effectively. First, the current context of obtaining functional powder ingredients from fruit and vegetable wastes will be presented, with special attention to the products of interest (Category A). Subsequently, the effects of processing on the functional and physicochemical profile of food powders will be exposed in order to identify possible pretreatments or additional steps that could increase the biological or nutritional value, and thus define the appropriate production parameters (Category B). And finally, the response to the *in vitro* digestion of the various bioactive compounds of interest and their effects on human health will be detailed (Category C). Considering the above, bibliographic research will allow to define ideal process conditions for producing vegetable powders of high quality, with a physicochemical profile suitable for subsequent application in food formulations and with functional properties capable of exerting beneficial effects on consumer health. Consequently, food products contributing to the principles of a sustainable healthy diet could be developed.

## **2. METHODOLOGY**

The bibliographic research was carried out in three phases: planning, writing and editing. During the information search, specific quality and relevance criteria were applied. Data collected were analyzed in a critical and well-founded manner, comparing various studies and relating the authors' approaches. In addition, a database was designed and structured with the spreadsheet program Microsoft Excel (supplementary material) as a tool to collect and record most relevant information. This Excel file was organized according to the three categories (A, B, and C) previously described. A model spreadsheet was designed to compile the most relevant data from the various articles examined. Additionally, an Excel sheet was created within the same

file to record the definition of key concepts, and four more sheets were created to gather relevant information for each vegetable. The methodological approach for the literature review was structured in the three categories mentioned and, according to each one, the research criteria was determined. Annex 1 illustrates the established categorization of the research and the respective selection parameters.

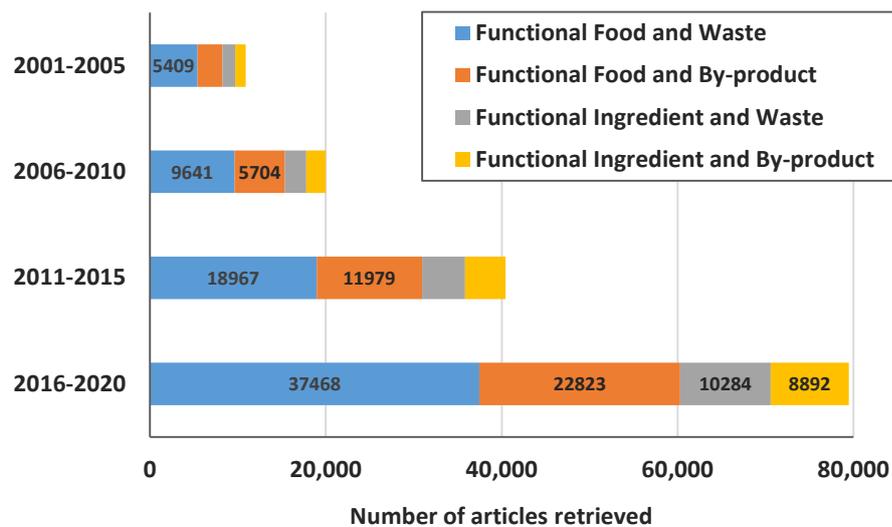
Selected keywords were entered into the most important databases of scientific journals, such as Science Direct, Scopus, Emerald Insight, Springer Link, Taylor & Francis Group, and Wiley Online Library. Additionally, tools such as Google Scholar and technical dictionaries such as IATE (Terminology of the European Union) and CAB Thesaurus were used. The titles and abstracts of more than 120 publications were selected and examined. Subsequently, the most relevant documents were chosen based on the previously defined criteria. In short, a total of 98 documents and investigations were selected and are the basis of the present bibliographic review. These works come from a total of 18 scientific journals, classified in the fields of food research, biotechnology, chemistry, and waste management. At this point, it can be mentioned that the collection of information was equivalent for each category (A, B, C). With respect to each vegetable, it was possible to find more studies related to cabbage and carrot than to celery and leek. No geographic or time restrictions were applied in the initial search process. According to the aforementioned categorization criteria (Annex 1), temporality of the selected studies was determined, showing that 80% of the investigations selected for this review had been published in the last ten years (2011-2020) and 45% belong to the last five years (2016-2020). Therefore, it can be affirmed that recent and updated information related to the subject of this study was used.

### **3. RESULTS AND DISCUSSION**

#### **3.1. Current context of obtaining ingredients and functional foods from fruit and vegetable wastes, with special attention to the products of interest**

Figure 1 shows the evolution in the last 20 years of the number of published papers dealing with functional food development from food waste (retrieved from ScienceDirect). For this search, the terms “functional food” or “functional ingredient” have been combined with “food waste” or “by-product”. Other terms used in the literature to define food waste such as “residue” or “co-product” were not specific and retrieved a significant number of papers not dealing with the topic of interest, for which they were neglected for this analysis. According to the results plotted in figure 1, the research interest in developing functional foods and obtaining functional ingredients from food waste has increased dramatically in the last years, the total number of published articles doubling every 5 years.

At the beginning of the 21<sup>st</sup> century food industry and researchers were already aware of the need for developing valorization processes which reduced the environmental impact of the food industry and contributed to food industry sustainability. Nevertheless, it is the recent definition of the FAO sustainable goals for development that have boosted this research trend which contributes to the idea of sustainable food systems from a social, economic, and environmental point of view. The definition of this concept states that a sustainable food system must ensure sustainable consumption and production patterns, as well as deliver safe, healthy and nutritious diets (FAO, 2019).



**Figure 1.** Evolution in the number of published articles (retrieved from Science Direct) using the combination of keywords “*Functional Food*” and “*Waste*”, “*Functional Food*” and “*By-product*”, “*Functional Ingredients*” and “*Waste*”, “*Functional Ingredient*” and “*By-product*”

Nevertheless, concerns related to FW have been presented in the scientific community since the 90s (Mirabella et al., 2014), such is the case of the research carried out by Kroyer (1995), who studied the impact of food processing on the environment highlighting the importance of waste treatment efficiently, reusing those by-products that are unavoidable to produce and turning them into new edible foods, thus rescuing biomass and valuable nutrients while reducing environmental burdens caused by inadequate management of waste. In parallel, health benefits associated to dietary fibers (DF) has led to the development of a large and potential market for fiber-rich products and ingredients, thus boosting the search for new sources of DF. This is one of the reasons why the by-products of fruits and vegetables that have been traditionally devalued are today considered a valuable source of this compound of interest (Rodríguez et al., 2006).

This increasing attention to DF thanks to its beneficial physiological effects on humans and its contribution to several functional properties when incorporated in various food products, has led to the development of functional powder ingredients obtained from pomace or bagasse by-products generated in the beverage industry. It was seen that the amount of studies found dealing

with this topic was also influenced by the levels of consumption and DF amount. Therefore, fruits like orange, apple or grape, and vegetables such as carrot and cabbage retrieved more papers. On the other hand, products like blueberry, lulo fruit, celery and leek had been studied to a lesser extent.

As for apple and citrus residues, Figuerola et al. (2005) used them to obtain fiber concentrates with functional properties to use them in food fortification. In particular, the powders obtained had a particle size ranging between 460 and 600  $\mu\text{m}$ , which allowed them to have high water retention and fat adsorption capacity. The powders also had a high DF content and a relatively low caloric value. Regarding the effectiveness of its incorporation in the development of foods reduced in calories and rich in dietary fiber, it was found to depend not only on the characteristics of each concentrate, but also on the way of adding them to the food matrix (particle size, temperature, ionic strength). Authors also stated that the characteristics of each concentrate determine the volume replacement and their thickening or texturizing effect.

Other authors (Tseng and Zhao, 2013) used grape pomace from the wine industry to produce antioxidant DF. Results showed that the content of phenolic compounds was affected by many factors such as grape variety, growth climate and location, harvest time, processing and storage conditions or extraction procedure. Wine grape pomace was reported to contain 61% of DF which, together with its content of antioxidant compounds, remained constant after 16 weeks of storage. Researchers also studied the application of this powder as a food ingredient to increase the nutritional value and improve the storage capacity of a yogurt and a salad dressing. Results showed that the addition of wine grape pomace powder resulted in a 35-65% reduction in peroxide values in all samples and that the products had a DF content of 0,94-3,6% (w/w).

Lulo fruit is a tropical species rich in antioxidant compounds, minerals and fiber. Duarte et al. (2019) used lulo fruit bagasse, a by-product generated during lulo fruit juice manufacturing, to obtain a functional powder ingredient. They studied the dehydration process of lulo bagasse through freeze-drying and hot air-drying technologies. The results showed that lyophilized samples had better antioxidant properties due to milder process conditions. The obtained lulo bagasse powder was rich in fiber, phenols and flavonoids with antioxidant capacity, which makes it an interest functional ingredient to enrich different types of food matrices. In a similar study, Roig et al. (2017) used blueberry juice bagasse, to obtain a functional powder rich in antioxidants such as polyphenols and anthocyanins, which may provide benefits for human health by preventing chronic diseases (Ismail et al., 2004). The researchers evaluated the incorporation of blueberry powder in a cookie formulation with good acceptance up to 30% addition. In Bas-Bellver et al. (2018) the production of a sustainable and functional powder ingredient from persimmon bagasse is proposed. Spain is the main producer of persimmon fruit in Europe, nevertheless, the overproduction and growing industrialization of this crop generates huge amounts of residues. In this case, a probiotic powder obtained

from bagasse inoculated with *Lactobacillus salivarius* spp. *salivarius* was also obtained. Maintaining the viability of the probiotic microorganism in the dehydrated powder is a challenge since there are many factors (e.g. temperature, humidity, pH or the process of obtaining the product) that can reduce its viability.

Other examples found in the literature include obtaining a powder from mango skins and subsequently applied it in macaroni preparations to improve their antioxidant properties (Ajila et al., 2010). These and other papers dealing with the recovery of fruit and vegetable by-products, and their reuse for the development of functional foods are shown in Annex 2. Thus it becomes clear that FW can be transformed to functional foods thanks to its bioactive compounds with benefits for human health, and thereby contribute to the development of a sustainable food system.

Going deeper into the vegetables under study, identifying their main bioactive compounds is a key factor when it is intended to use them in obtaining food powders with optimal functional properties. As shown in Annex 3, the four vegetables have a nutritional profile that is important for a healthy and sustainable diet. Cabbage is rich in sulforaphane, a product of hydrolysis of glucosinolates that is known to be the most potent food-derived anticancer substance (Tanongkankit et al., 2011). Carrots are a rich source of  $\alpha$ - and  $\beta$ -carotene (Chen et al., 2016; Hiranvarachat et al., 2011), which are important precursors of vitamin A in the human metabolism (Barzee et al., 2019). As for celery, it stands out for its high apigenin content, a flavone that has the ability to decrease pre- and postprandial blood glucose levels (Yusni et al., 2018). Finally, leek is an important source of sulfides (Luo et al., 2014).

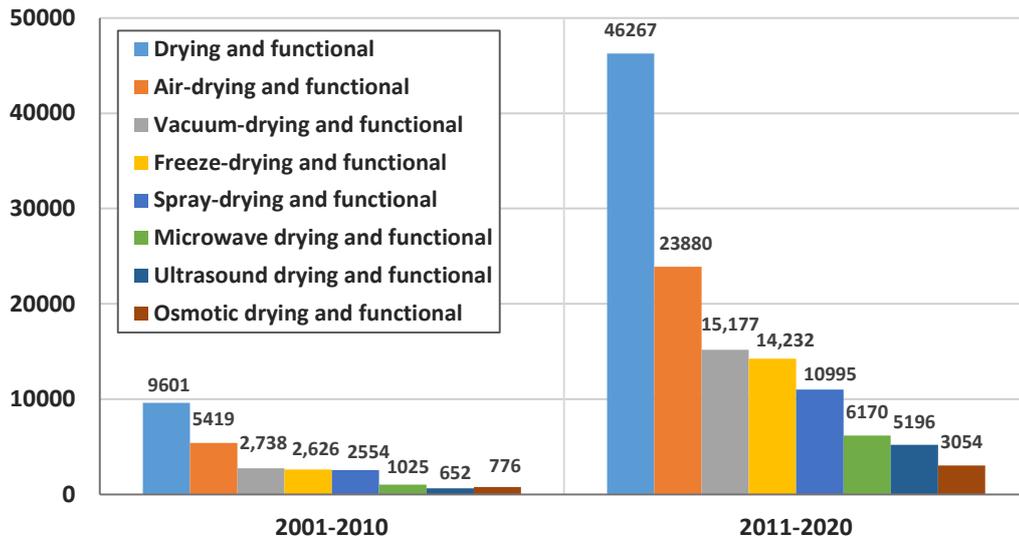
Focusing on the vegetables of interest, it was possible to find more detailed information on obtaining functional powder ingredients from cabbage and carrot residues as compared to celery and leek, possibly due to their production and consumption levels. Therefore, in the case of cabbage Nilnakara et al. (2009) evaluated the production of antioxidant DF powder from the outer leaves and kernel. This specific by-product represents the 40% of the total weight and it is often used as fertilizer or animal feed. The researchers studied the effects of blanching in hot water and hot air drying at different temperatures on the quality of the final product. As for carrot, one of the most important root crops in the world because of its  $\beta$ -carotene content, a large proportion is consumed as juice (Chau et al., 2004). However, after juice extraction thousands of tons of carrot pomace are produced and are also generally disposed of as animal feed. Approximately 80% of the carotenes remain in this by-product (Baljeet et al., 2014). It also has a DF content of approximately 63.6 g/100 g of dry matter (Chau et al., 2004). Therefore, Nouri et al. (2017) sought to reuse it and develop a functional powder ingredient. They evaluated the application of carrot pomace powder in the formulation of a fried pastry product for mass consumption, with the aim of improving its nutritional profile and thus creating low-fat donuts with high fiber content.

In contrast, information found related to celery and leek was very limited. Specific research dealing with the recovery of by-products of these respective vegetables and their reuse for the development of functional foods was scarce. Researchers Wang et al., 2020 studied the effects of wheat flour replacement by celery powder (CP) at different levels (1, 2, 3 and 5/100 g of flour) on bread quality. Results showed that the higher CP content, the greater the water absorption of the dough; however, the protein network, the maximum viscosity and the crystallinity of the starch decreased with an increase on the CP content. On the other hand, a higher level of CP increased the hardness and chewiness of the bread. It is important to highlight that the addition of CP significantly increased the total phenol content of the bread and, therefore, caused a significant improvement of its antioxidant properties. Ozgur et al., 2011 studied the changes in some physicochemical properties and the variations in the antioxidant compounds of leek caused by the drying process. Leek drying resulted in a loss of ascorbic acid and phenolic compounds, so that the antioxidant capacity was reduced by more than 50% after drying.

### **3.2. Vegetables and fruit wastes as functional powder ingredients. Influence of processing variables and pretreatments**

Food processing is one of the main factors affecting bioaccessibility of nutrients and bioactive compounds by increasing or decreasing its content (Cilla et al., 2018). It is crucial to study the effect of different treatments on the functional properties of the powders, given that functional foods are those that, in addition to providing nutrients, are capable of positively affect specific biological functions and thus improving the general state of people's health (Rodríguez et al., 2006). For this reason, processing techniques are increasingly sophisticated and diverse, in response to the growing demand for quality food, with functional properties. The final characteristics of the food powders also depend on the composition and physicochemical properties of each vegetable. To maximize the value of the waste, the general processing steps must be well defined.

Dehydration, as a fundamental stage for the production of powder ingredients, involves the transient transfer of heat and mass accompanied by physical, chemical and phase change transformations. Unfortunately, these transformations can cause changes in product quality, as well as in heat and mass transfer mechanisms, and in its technological properties of interaction with water and oil. Figure 2 summarizes the evolution in the number of papers which study the application of different dehydration techniques to obtain functional foods or ingredients, and their impact on the bioactive compounds present in the food material. As it can be seen, dehydration is a common technique applied to obtain functional food ingredients. Research interest on the impact of drying on the development of ingredients and functional foods, and its effects on the bioactive compounds has increased significantly in the last decade.



**Figure 2.** Dehydration techniques used to obtain functional food or ingredients. Average number of articles retrieved when using each technique combined with the terms “functional food powder”, “functional ingredient” or “bioactive compound”.

In order to select the most suitable dehydration technique to obtain functional powder ingredients from the vegetable wastes considered in this research, it is necessary to detail the different drying methods and analyze their advantages, disadvantages and limitations. It is also relevant to compare different studies and observe the impact of each dehydration technique on the bioactive compounds present in different fruit and vegetable matrices. These compounds that have nutritional benefits are a key factor in determining the functional properties of powdered foods, and this subsequently influences their application and commercial value. Table 3 collects main dehydration techniques applied to develop functional ingredients and functional foods.

Within the context of this research and considering the information gathered in table 3, the effects of hot air drying over the bioactive compounds present in the vegetable wastes under study will be presented in detail below. This technology is commonly applied in the food industry due to its lower processing and investment costs. From an industrial point of view, it is a suitable technology for the recovery of solid waste from the fruit and vegetable sector. On the other hand, freeze-drying is a technology of limited industrial application due to its high operating costs. Other drying techniques such as spray-drying is limited to liquids, emulsions or suspensions, for which it is not considered a viable option for this project which seeks integral valorization avoiding extraction. Thus, the use of hot air drying is proposed for the integral recovery of the vegetable wastes generated in the cooperative.

**Table 3.** Dehydration techniques applied to the development of functional ingredients and functional foods.

| Dehydration Technique            | Products and conditions   | Impact on bioactive compounds  | Advantages   | Disadvantages  | Limitations  | References   |
|----------------------------------|---|--|--|--|--|--|
| Air-drying (AD)                  | Fresh carrot cubes   Chopped cabbage outer leaves (both dried at 70-90 °C)                                | High temperatures can decrease the content of thermolabile substances and reduce the total antioxidant activity  | Most widely used drying technique as it is inexpensive and easy to operate. It allows integral recovery of vegetable waste   | Long drying times and surface overheating cause color darkening, loss of flavor, decreased rehydration capacity and loss of nutritional value  | Very heat sensitive bioactive compounds present in vegetable waste. Therefore, reduction of the nutritional value                                    | Chen et al., 2016; Hiranvarachat et al., 2011; Nilnakara et al., 2009.                           |
| Vacuum-drying (VD)               | Carrot slices (dried at 65-75 °C, 0.02 and 0.03 MPa)   Eggplant (dried at 30 to 50 °C, 2.5, 5 and 10 kPa) | Beneficial to improve the quality and nutritional value of the powder. Ideal for thermal and/or oxygen sensitive materials   | Higher drying rate, lower drying temperature, and oxygen-deficient processing environment  | Batch process, has very small throughput, and involves a residence time of the order of 20–100 h   | Limited energy transmission because the transfer of thermal energy to the workload becomes difficult since convection is ineffective at low pressure | Chen et al., 2016; Ghandi et al., 2013; Jiang et al., 2013.                                      |
| Freeze-drying (FD)               | Purple carrot roots and apple slices  | Negligible loss of nutrients, retention of original color and hence higher quality   | Less damage in the product due to oxygen limitation and low temperatures. Allows to preserve to a greater extent the properties of the product   | It has poor heat transfer rate. The long drying time required and its batch nature add costs and lower the potential for commercial application  | Expensive process due to the cost of maintaining the pressure and temperature conditions at which it takes place                                     | Dorota et al., 2009; Huang et al., 2009; Jiang et al., 2013; Ratti, 2001.                        |
| Spray-drying (SD)                | Bayberry juice and orange juice   | Product deterioration can occur due to the combined effects of dehydration, thermal, and oxygen stresses imposed on bioactive components   | Widely used technique. It is rapid, continuous, reproducible, single-step, and thus, scalable without major modifications  | The yield strongly depends on the work scale. Low yield is due to the loss of product in the walls of the drying chamber   | Only applicable to suspensions, solutions or emulsions. It does not allow an integral use of solid waste, generally applied to extracts.             | Anal and Singh, 2007; Fang and Bhandari, 2011; Goula and Adamopoulos, 2010; Santos et al., 2018. |
| Microwave drying (MD) *Assisted  | Trabzon Persimmon (460 W for 7 mm slice thickness)  | Even though this method might not be better than others in keeping composition and nutritional capacities of the food products, their functional properties could be maintained more effectively | Faster reduction in humidity caused by absence of heat movement from the surface to the product due to absence of convection. A reduction in drying time of up to 90% has been reported. Higher profit margins due to less chemical and water usage, plus more production time available | Non-uniform temperature distribution. The product must be in constant movement within the cavity to avoid hot spots. Internal temperature can increase to values that can burn it, therefore it is difficult to control the temperature in the final phase | Relatively slow development of equipment capable of operating efficiently on large scales.   | Çelen, 2019; Dehnad et al., 2016; Menéndez, 2017.  |
| Ultrasound drying (UD) *Assisted | Carrot slices (power rates of 10-1000 W/cm <sup>-2</sup> at a frequency between 20 and 1000 kHz)          | Heat-sensitive components are damaged less. Shorter processing time and lower air temperatures protect the bioactive compounds   | Better quality of the final powder. It can reduce the energy consumption. It is quicker and more powerful.   | The effectiveness of the treatment depends on the correct intensity applied.   | More effective in porous materials, in conditions of low temperatures and low air speeds.  | Dehnad et al., 2016; Guo et al., 2020; Jiang et al., 2013; Xu et al., 2009.                      |
| Osmotic drying (OD)              | White cabbage (aqueous solution of NaCl and sucrose)  | Loss of most nutrients is avoided. It also brings the maximum stability for phytochemicals   | Non-thermal process that can significantly reduce the drying time and therefore achieve a higher food quality. It does not have a high energy requirement  | Partial dehydration of food. Results depend on the osmotic solution composition  | A semipermeable membrane is required   | Cvetković et al., 2019; Prosapio and Norton, 2017.   |

When applying HAD at a temperature range of 60 to 69 °C in fresh samples of cabbage a decrease in the glucosinolate content was observed (Tanongkankit et al., 2012). Other researchers reported that the optimal temperature to maximize myrosinase activity in white and red cabbage varieties was approximately 60 °C and that it was destroyed after heating to 70 °C during 30 min (Yen and Wei, 1993). It was also reported that the formation of sulforaphane (SF) occurred when the temperature of the vegetable was in the range of 25 to 53,5 °C, and that thermal degradation occurred when this temperature was exceeded (Tanongkankit et al., 2011). Therefore, it has been proposed a gradual change in the temperature of the drying medium, to achieve greater retention of SF in the DF powder of the outer leaves of cabbage (Lekcharoenkul et al., 2014). Hence, in the early drying period the heating rate should be high to rapidly increase the temperature of the material to a value above 40 °C. After this period, the temperature of the material must be controlled so that it does not exceed 50 °C. To preserve the functional properties of the powder it should have a minimum SF content of only approximately 15 µm (2.7 mg/L) (Gamet-Payraastre et al., 2000).

In the case of carrot, HAD at 60 °C and under blanching conditions (pre-treatment) allows to obtain a higher DF content (Chantaro et al., 2008). At 90 °C, 20% of  $\beta$ -carotene breaks down (Mayer-Miebach et al., 2005). In order to achieve a moisture content of 0.12 kg water / kg dry matter, fresh carrot cubes (1 cm<sup>3</sup>) were dried at 70, 80 and 90 °C during 5, 4 and 3 h, respectively (Hiranvarachat et al., 2011). Moreover, the HAD technique can alter the cell wall and induce an anticipated improvement in the release of carotenoids, this leading to higher bioaccessibility values (Zhang et al., 2018). The most stable individual carotenoid of this vegetable is lutein, because its content its reduced by only 15.1% at 80 °C. In contrast, under the same conditions  $\beta$ -carotene is heavily degraded. On the other hand, combining ultrasound and vacuum processes to dehydrate carrot slices can decrease the drying time in 41 to 53%. This novel technique called ultrasonic vacuum drying can also improve the rehydration potential, nutritional value, color, and texture properties of the samples (Chen et al., 2016).

Regarding leek, Ozgur et al. (2011) investigated changes in some physicochemical properties and variations in antioxidant compounds caused by HAD at  $63 \pm 2$  °C for 3 h with an air velocity of 2.5 m/s. They observed that the antioxidant capacity decreased by more than 50%, there was a loss of ascorbic acid and the dehydrated samples showed a large total color difference ( $\Delta E = 12.53$ ) mainly due to the effect of temperature on heat sensitive compounds. However, the  $\alpha$  and  $\beta$  chlorophyll content was higher in dried leeks than in fresh leeks. Regarding celery, no detailed studies were found on its dehydration process with the use of HAD technology, but the research carried out by Priecina et al. (2018) on celery roots (*Apium graveolens* var. *Rapaceum*) showed that their small amount of carotenoids significantly decreased after drying. HAD samples were determined to have a

higher total phenolic content than those dehydrated with microwave vacuum drying, as long as a steam blanching pretreatment is applied.

One way to alleviate the adverse effect of HAD is to pretreat a product, either physically or chemically, before drying (Górnicki and Kaleta, 2007). The main purpose of pretreatment is generally to inactivate enzymes such as polyphenoloxidase, peroxidase, and phenolase, and to inhibit some undesirable chemical reactions, which cause many adverse changes in a product. Many pretreatment techniques have been used in the food industry, including blanching and the use of weak acids (Prakash et al., 2004; Górnicki and Kaleta, 2007). Natural whitening causes some changes in the sensory and nutritional qualities of the products (Nilnakara et al., 2009). Generally, it is done with hot water, although it is also possible to apply steam. Other type of pretreatments can improve the drying process conditions and therefore it is possible to obtain better results in the physicochemical and functional characteristics of the vegetable wastes. The following table is a synthesis of the pretreatments applied to obtain functional powders, according to the review of the scientific literature and that have been applied in the various selected studies. It summarizes the process conditions and the potential impact on the fruit or vegetable matrix, as well as on its bioactive components (Table 4).

In order to obtain a high-quality product, it is important to monitor bioactive compounds and physicochemical characteristics so that the powders obtained are suitable to be used as functional ingredients for subsequent applications. The pretreatments summarized in table 4 have different advantages and limitations, depending on the food matrix. Within the context of the present research, milling is a key step to achieve the desired results and improve the drying process. Steam cooking or blanching are recommended to soften the structure of the vegetable and facilitate water removal during drying. To accurately determine the most appropriate method between the two previously mentioned, it is necessary to carry out experimental tests that allow measuring the evolution of the bioactive compounds of interest and the particular effects on the physicochemical properties of the vegetable powders. Novel technologies such as electric pulses or ultrasounds may be an interesting option thanks to their positive effects, but their industrial application are more limited.

As observed, it is essential to rigorously determine the process conditions, to reduce the negative impacts of drying and improve the results. With regard to post-treatments applied to obtain powders, it is worth mentioning the importance of grinding and its role in determining particle size, which has a relevant impact on flow properties and subsequent applications of the powder as a food ingredient. Researchers such as Chantaro et al. (2008) and Tanongkankit et al. (2012), ground the dehydrated carrot and cabbage, respectively, to obtain a specific particle size for particular applications. Sieving is an additional post-treatment useful to fix the desired particle size. At this point, rehydration is an additional parameter to consider as an indicator of quality (Hiranvarachat et al., 2011; Ozgur et al., 2011; Chen et al., 2016).

**Table 4.** Pretreatments discussed in the literature and potential impact on fruit and vegetable tissue and its bioactive compounds.

| Pre-treatments       | Process Conditions   | Outstanding results   | Reference   |
|----------------------|--|---|---|
| Milling              | Chopping or grounding fresh vegetables   | It determines particle size characteristics; the size reduction improves the drying process. It is an essential step to obtain high-quality powders, with proper nutritional and physicochemical characteristics.   | Bas-Bellver, et al., 2020; Djantou et al., 2011; Erenturk et al., 2005.       |
| Freezing             | Vegetable wastes were stored in a freezer at -22 °C until processing   | It has different impacts on water activity values, depending on the food matrix. It maintains nutritional and sensory quality of products. When frozen and later HAD, the samples reached higher water content possibly due to the reduction of porosity thus generating a compacted bed with increased resistance to drying.   | Ando et al., 2016; Bas-Bellver, et al., 2020; Peng et al., 2018               |
| Steam cooking        | From 1 to 20 min in a closed bath and a single layer of vegetable waste  | In the case of brassica vegetables it does not produce a significant loss of glucosinolates. The content of lutein, $\alpha$ -carotene, $\beta$ -carotene, total carotenoids, and vitamin A remain high. Heating alters plant tissues, making bioactive compounds more available. A long steam cooking time prior to HAD can reduce nutrient content.   | dos Reis et al., 2015; Tanongkankit et al., 2012.                             |
| Blanching            | Cooking in water at 90, 93 and $95 \pm 2$ °C during 1 or 2 min with a 1:7 ratio (vegetable waste vs. water). Immediate cooling in cold water at 4 °C   | It reduces the drying time, due to the softening of the structure which facilitates water removal. It enables to achieve higher content of fiber (pectin), due to the loss of dry matter. The WRC and SWC can be improved, which is good for food applications. The retention of TPC can also be improved and enzymatic browning does not occur   | Chantaro et al., 2008; Ismail et al., 2004; Nilnakara et al., 2009            |
| Acid pretreatment    | Blanching vegetable waste with 0,7% (w/v) of citric acid during 2,5 min (pH 4-5)   | It improves product quality by inactivating enzymes and modifying the texture. The color can also be maintained due to the chelating properties of the acids. Better water rehydration capacities, and a redder color can be obtained   | Branem et al., 2002; Hiranvarachat et al., 2011; Zhu et al., 2007             |
| Microwaves           | Fresh vegetables samples are pretreated with MV at different powders and in a variable time  | It increases the nutritional value of the dry samples. It leads to the maximum content of extractable phenols. Better functional properties can be obtained. Particular case of the cabbage: the content of sulforaphane increased by 6.23 times compared to the fresh samples  | Bejar et al., 2011; Nieto Calvache et al., 2015; Pongmalai et al., 2018       |
| Osmotic pretreatment | Immersion of the vegetable sample in a hypertonic solution (45 ° Brix at $30 \pm 2$ °C). Stirred at 740 rpm and removed in different timings. Ratio of 1:25.   | It reduces nutritional losses due to the decrease of the drying time. It also inhibits enzymatic browning, allows to achieve higher volatile compounds during further dehydration and the natural color can be maintained   | Bozkir and Ergün, 2020; Nimmanpipug et al., 2013; Mosquera-Vivas et al., 2019 |
| Ultrasounds          | Ultrasonic parameters: sonication time, amplitude and ultrasound power. Particular case (Carrots): Water immersing; f = 25 kHz, P = 700 W, T = 23 °C, t = 30 and 60 min  | Non-thermal technology that has the potential to greatly decrease the drying time, it increases the drying kinetics, decreases the water activity, improves the product color and reduces the nutrient loss.  | Dehnad et al., 2016; Huang et al., 2020; Xu et al., 2009                      |
| Electric pulses      | Food by-product (e.g. pulp, seeds, shells, etc.) is subjected to an intermittent application (< 300 Hz) of electric fields at moderate-high intensity (0.1-20 kV/cm) and short duration (from a few $\mu$ s to several ms) | It has great potential to improve the recovery of compounds quickly, economically and environmentally sustainable. It causes electroporation, which involves the formation of localized pores in the cell membranes of eukaryotic and prokaryotic cells. This improves the subsequent applied technological processes. The results depend on the physicochemical properties of the treated matrix and its specific tissues. | Barba et al., 2015; Puértolas and Barba, 2016; Vorobiev and Lebovka, 2011.    |

SWC: Swelling capacity. WRC: Water retention capacity. TPC: Total phenolic content.

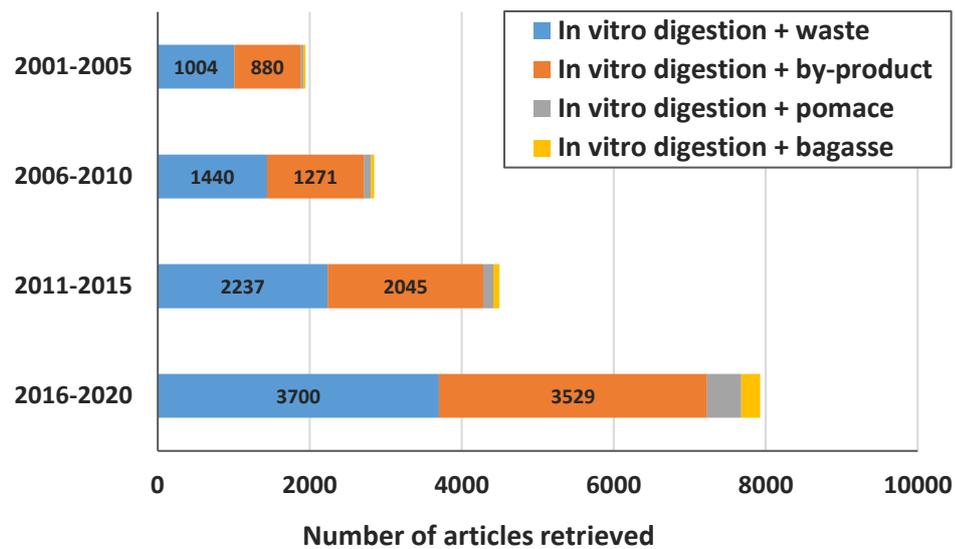
As observed, it is essential to rigorously determine the process conditions, in order to reduce the negative impacts of drying and improve the final results. In reference to the post-treatments applied in obtaining powdered food, it is worth highlighting the importance of grinding and its role in determining particle size. This is a key aspect in the flow properties and subsequent applications of the powder as a food ingredient. The researchers Chantaro et al. (2008) ground the dehydrated carrot to a specific particle size, which was between 125 and 425  $\mu\text{m}$ . Tanongkankit et al. (2012), also ground the dehydrated cabbage obtaining a particle size in the range of 150 to 450  $\mu\text{m}$ . Sieving is additionally a possible post-treatment useful to fix the desired particle size. At this point, rehydration is an additional parameter to consider as an indicator of quality (Hiranvarachat et al., 2011; Ozgur et al., 2011; Chen et al., 2016).

### **3.3. Response of the *in vitro* digestion of the main bioactive compounds present in the vegetables of interest and their effects on human health**

The goals of nutrition are to provide nutrients in such quantity and quality as are necessary to meet bodily requirements. Optimal nutrition should include bioactive compounds such as fiber, antioxidants, phytochemicals, among others, since these have the capacity to improve health, generate well-being and are useful to prevent the risk of suffering diseases (Cilla et al., 2018). The potential of these physiologically active components present in food to exert beneficial effects depends on their release from the matrix, changes during digestion, absorption, metabolism and biodistribution. In order for these compounds to exert their bioactivity, they must be bioavailable. Other factors such as dose and host must also be considered (Bohn et al., 2015).

The term bioavailability has various working conditions and there is no universally accepted definition. From a nutritional point of view, it is defined as the fraction of ingested component that is available for use in normal physiological functions. This concept includes two additional terms, which are bioaccessibility (BA) and bioactivity (Bact). BA is defined as the fraction of a compound that is released from its food matrix in the gastrointestinal tract and, therefore, is available for intestinal absorption. Bact includes events related to the way in which the bioactive compound reach the systemic circulation and are transported until reaching the target tissue; it also refers to the interaction with the metabolism of biomolecules in these tissues and the entire cascade of physiological effects it generates. *In vitro* methods can evaluate the BA of the compounds of interest, because they simulate gastrointestinal digestion (Cilla et al., 2018). These are very useful for evaluating the effect of processing and in this way designing functional foods with greater health promoting effects.

As shown in figure 3, the interest in understanding the response of the bioactive compounds present in products or ingredients obtained from fruit or vegetable waste has increased considerably in the last years. Main terms used by researchers are, again, “waste” and “by-product”. The use of other keywords such as “pomace” or “bagasse” is marginal.



**Figure 3.** Evolution in the number of published articles (retrieved by Science Direct) which combine the keywords “*in vitro digestion*” and “waste”, “by-product”, “pomace” or “bagasse”, in the last twenty years.

Taking into account that the objective of the present study is to develop functional powder ingredients from vegetable by-products of the food industry, particularly cabbage, carrot, celery and leek, research on the digestion of powder, alone or as a part of a formulated product, is essential. A method commonly applied in the study of the gastrointestinal digestion of food powders obtained from fruit and vegetable residues, is the practical static digestion methodology. It consists of mouth mastication, stomach digestion, and intestinal digestion. The authors Gouw et al. (2017) used this method to study the digestion process of the bioactive compounds present in different food powders obtained from fruit bagasse. The research was conducted by mixing 1 g of powder with 8.5 mL of 0.05 M phosphate buffer (pH 7.0) at the first stage of mouth mastication. Results evidenced that bound polyphenols from food powders are liberated in the simulated gastrointestinal digestion and that phenolics can be released from the food matrix by direct solubilization in the intestinal fluids and through the action of digestive enzymes. Low molecular weight polyphenols are partially absorbed in the small intestine, while high molecular weight can be fermented in the colon by the microflora and partially absorbed to gut epithelial cells, acting as the counteract of prooxidants.

The information found on the *in vitro* digestion of food powders obtained from the particular vegetable wastes of the present research was limited. A close study is that of Bas-Bellver et al. (2020b) who evaluated the changes in bioactive compounds throughout *in vitro* gastrointestinal digestion of blueberry and persimmon residue powders. The researchers conducted the study directly with the powder without prior dilution or addition of water or other liquid or solid food matrix. In a first stage, the powder was mixed with human saliva in a ratio of 1:1 (w/v) and then the established protocol was continued. Authors concluded that the release of antioxidants along gastrointestinal digestion was dependent on the type of residue, the drying process, and the fiber content

and type. *In vitro* colonic fermentation also carried out in this study showed that the carotenoids and anthocyanins present in the digested powders can modify the microbiota and promote a benefit for human health.

Similarly, Zhang et al. (2018) applied an *in vitro* digestion model in which they evaluated the interaction of fiber with carotenoids. Fresh and dried carrot slices (60, 70 and 80 °C) were ground for 1 min to simulate the chewing process. One-half of the test food was used for *in vitro* digestion, the other half was supplemented with 5% (w/w) of olive oil. Both test foods with and without added lipids were stored at -80 °C until use. To simulate the digestion process, an aliquot of 3 g of the thawed test food was combined with 10 mL of simulated salivary solution. Results obtained showed that the cell wall acts as the main natural structural physical barrier that governs the release of carotenoids. Pectin composition and the presence of other polysaccharides in the cell wall influence the BA of carotenoids by interacting differently with target compounds. Addition of lipid to dried samples during digestion had a marked positive effect on the BA of individual carotenoid.

The study of the digestion of powders can also be carried out by dissolving the powder in water. Such is the case of the research of Gullon et al. (2015), who evaluated the effect of *in vitro* gastrointestinal digestion on the recovery rates and BA indexes, changes in antioxidant activity and the production of short-chain fatty acids from two powdered extracts rich in DF, such as date seed meal and apple bagasse obtained from agro-industrial by-products. The powders (2.4 g / each) were diluted in 9 mL of water before they were mixed with 1 mL of simulated saliva. From there, the established protocol was continued. The BA of phenolic compounds was 78.54% for date seed powder and 91.58% for apple bagasse powder. The fermentation of both extracts by colonic bacteria generated short chain fatty acids such as formate, succinate, acetate, propionate and butyrate.

The evaluation of the *in vitro* digestion of powders incorporated in a food matrix can produce different results, due to the interaction of the powder with the other ingredients throughout the gastrointestinal digestion. In the bibliographic review, it was identified different studies that evaluate the digestion of the vegetables studied in the present research, from the products themselves and not in powder form. These studies are useful to evaluate the digestion of the bioactive compounds of interest, as well as the impact of the addition of other ingredients, in order to determine the effect of the interaction between ingredients on the BA of the compounds. Therefore, Hornero and Mínguez, (2007) affirm that both the processing and the content mainly of lipids (cooking oil in this case) significantly improve the BA of carotenoids of carrots and, therefore, can increase the bioavailability in humans. In their research, carrots were cooked to 100 °C in a water bath for 15 min. Although the heat treatment during cooking showed to have a negative impact on the carotenoid content, a positive effect was found on the micellarisation of the carotenes and therefore on their BA.

On the other hand, Sarvan et al. (2017) studied the form of conversion of glucoraphanin (GR) and the BA of the decomposition products released, evaluating the effect of steam cooking, as well as the composition of food (addition of proteins or lipids) with an *in vitro* digestion model (mouth, stomach, intestine, but not colonic digestion). They were able to determine that the main formation of sulforaphane (SF) and sulforaphane nitrile (SFN) occurred during *in vitro* chewing. Furthermore, they state that SF concentrations were up to 10 times higher in raw and steamed broccoli samples of 1 min after digestion, compared to longer cooking samples. The addition of proteins or lipids had no influence on the formation and BA of SF or SFN. They have concluded that the main factor for the formation of SF and its BA from broccoli in the *in vitro* upper digestive tract is the presence of active myrosinase in the vegetable.

In the particular case of celery and leek, no studies related to the digestion of these powdered or fresh vegetables were identified. A search was then carried out according to the bioactive compound of interest present on each vegetable, in order to evaluate the impact of digestion on the BA of the nutrients present in these food matrices and also their effects on human health. Although no specific study was identified with the *in vitro* digestion assessment method, the researchers Kowalska et al. (2020) studied the influence of apigenin and its derivatives on certain coagulation parameters in human plasma. The results indicated that the tested phenolic compounds have different influences on the processes of oxidative stress and coagulation. These dissimilarities can be attributed to differences in their chemical structure. With the *in vitro* method they demonstrated the antioxidant and anticoagulant activity of apigenin. On the other hand, a fact of particular interest about the leek is that it has a positive effect when mixed with cereals such as rice and wheat, or legumes such as beans and soybeans, since it improves the BA of iron and zinc, minerals present in these foods (Luo et al., 2014).

#### **4. CONCLUSION**

The recovery of valuable compounds from food waste is a major challenge for scientists related to the field, also considering that commercial implementation is a complex approach that depends on several parameters to evaluate. Furthermore, it must be possible to scale without affecting the functional properties of the target compound and develop a product that meets the standards of high quality, safety and organoleptic characteristics demanded by consumers. Many studies have indicated that the agricultural by-products of fruits and vegetables could be promising sources of dietary fibers and other functional compounds. The way of processing agricultural by-products determines the characteristics and functionality of the final products, which is why the research should focus on the development of safer and more personalized processes, avoiding extreme processing conditions.

Dehydration as a food preservation technology, allowing the extension of shelf life, is a useful tool for the transformation of plant-based food residues into functional powder ingredients, intended for use in the food industry. It is advisable to consider pretreatments such as blanching and milling, to alleviate the adverse effects of dehydration and improve the efficiency of the process. The selection of the temperatures and the drying time must also consider the effect on the content of the bioactive compounds of interest and their subsequent impact on the bioaccessibility throughout the digestion process; this because the process conditions can also have an impact on the beneficial effect derived from consumption of the vegetable powders.

On the other hand, an integral recovery is important to avoid other residues and rescue the main bioactive compounds. As key indicators of the final quality of the vegetable powders it must be evaluated the physicochemical composition, the functional properties and the physiological effects on human health through *in vitro* digestion, in order to guarantee the quality of the powders and their effective application in food formulations. A comprehensive study should include recovery protocols, specific applications, and preservation trials to ensure sustainable industrial production of functional powder ingredients from vegetable waste.

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**ANNEX 1**  
Research categorization

| <b>RESEARCH CATEGORIZATION</b> |   |  |  |
|--------------------------------|---|--|--|
| <b>Category</b>                | <b>Description</b>  | <b>Search criteria</b>   | <b>Key words</b>   |
| A                              | Current context of obtaining ingredients and functional foods from fruit and vegetable wastes, with special attention to products of interest     | General approach to obtaining functional food powder from the recovery of fruits and vegetables by-products from the food industry   | Bio-waste valorization; Food powder; Functional food; Functional ingredient; Fruit and vegetable by-products; Vegetables waste   |
| B                              | Vegetables and fruit wastes as functional powder ingredients: influence of processing variables and pre-treatments                                | Articles related to the impacts of the vegetable matrix on the drying process, time and other relevant results. Emphasis on pre-treatments that can increase biological or nutritional value of the vegetable powders studied in this research and that can be included in this project. The articles chosen do not necessarily have to be intended to obtain a food powder. | Blanching; Dehydration; Electrical pulses; Electrotechnologies; Food waste processing; Freeze drying/lyophilization; Hot air drying; HPP; Maximizing bioactive compounds; Milling (Chopping/Grinding); Steam cooking |
| C                              | Response to the <i>in vitro</i> digestion of the main bioactive compounds present in the vegetables of interest and their effects on human health | Absorption process of the bioactive compounds of interest, and studies of their bioavailability/bioaccessibility. Studies of its effects on human health. Interaction during digestion of the main compound of interest with fiber. Studies related to the digestion of powders and the application of the powder in different food formulations                             | Antioxidant activity; Apigenin; Carotenoids; Fiber; Glucosinolates; <i>in vitro</i> digestion; Phenolic compounds; Sulfoxides.   |

## ANNEX 2

### Papers dealing with functional food development from food waste

| Food waste                                       | Compound of interest   | Ingredient or functional food that is obtained  | Applied technology                    | Reference                 |
|--|--|---|---------------------------------------|---------------------------|
| Apple pomace and citrus peel                     | Dietary fiber  | Powdered fiber concentrates   | HAD at 60 °C                          | Figuerola et al., 2005    |
| Blueberry bagasse                                | Dietary fiber, polyphenols and anthocyanins                      | Functional blueberry bagasse powder applied in a cookie formulation                     | HAD at 70 °C                          | Roig et al., 2017         |
| By-products of cooked pear, apple and dried date | Phenolic compounds and dietary fiber                             | Pear, apple and date powder   | FD                                    | Aguedo et al., 2012       |
| Cabbage outer leaves                             | Sulforaphane and dietary fiber                                   | Dietary fibre powder  | HAD at 40, 50, 60 and 70 °C           | Tanongkankit et al., 2011 |
| Carrot peels                                     | Carotenes and dietary fiber                                      | Antioxidant high dietary fiber powder   | HAD at 60-80 °C                       | Chantaro et al., 2008     |
| Carrot pomace                                    | Carotenes and dietary fiber                                      | Carrot pomace powder applied in a biscuit formulation                                   | HAD at 60 °C                          | Baljeet et al., 2014      |
| Carrot pomace                                    | Carotenes and dietary fiber                                      | Carrot pomace powder for development of low-fat donut with high fiber content           | HAD at 60 °C                          | Nouri et al., 2017        |
| Carrot pomace                                    | Carotenes and dietary fiber                                      | Carotene-rich functional food ingredient  | Enzymatic liquefaction                | Stoll et al., 2003        |
| Grape pomace                                     | Phenolic compounds and dietary fiber                             | Wine grape pomace powder applied in a yogurt and a salad dressing formulation           | FD under 55 °C and vacuum of 17.33 Pa | Tseng and Zhao, 2013      |
| Lulo bagasse                                     | Antioxidants and dietary fiber                                   | Functional lulo bagasse powder  | FD and HAD at 60 and 70 °C            | Duarte et al., 2019       |
| Mango skins                                      | Polyphenols, carotenoids and dietary fiber                       | Mango powder applied in macaroni preparations   | HAD at 50 °C                          | Ajila et al., 2010        |
| Outer leaves and kernel of the cabbage           | Ascorbic acid, phenolic compounds, tocopherols and dietary fiber | Antioxidant dietary fibre powder  | HAD at 70, 80 and 90 °C               | Nilnakara et al., 2009    |
| Persimmon bagasse                                | Polyphenols, flavonoids, carotenoids and dietary fiber           | Persimmon peel powder and the development of a probiotic powder                         | FD and HAD at 60 y 70 °C              | Bas-Bellver et al., 2018  |
| Raspberry pomace                                 | Dietary fiber  | Dried dietary fibre-rich raspberry pomace used in the formulation of shortcrust cookies | HAD                                   | Górecka et al., 2010      |
| Tubers from Jerusalem artichoke                  | Inulin   | Jerusalem artichoke powder applied in a bread formulation                               | HAD in two-steps at 120 and 60 °C     | Praznik et al., 2002      |

*HAD: Hot air drying; FD: Freeze-drying*

### ANNEX 3

#### Compounds of interest present in the vegetables of this study

| Vegetable | Scientific name and Family                                      | Bioactive compounds and benefits for human health  |
|-----------|---|--|
| Cabbage   | <i>Brassica oleracea</i> L. var. <i>Capitata</i> , Brassicaceae | It has significant amounts of antioxidants such as ascorbic acid, phenolic compounds, and tocopherols (Kim et al., 2004; Wennberg et al., 2004). Sulforaphane, one of the most potent food-derived anticancer substance, is a product of hydrolysis of glucosinolates, which are abundant in Brassica vegetables (Tanongkankit et al., 2011). That is why it is an excellent source of bioactive phytochemicals that can reduce the risk of chronic diseases (dos Reis et al., 2015). Epidemiological studies show that a high consumption of it is associated with a lower risk of certain types of cancer, such as lung cancer, colorectal cancer, breast cancer, and prostate cancer (Ciska and Pathak, 2004; Higdon et al., 2007). |
| Carrot    | <i>Daucus carota</i> , Umbelliferae                             | Rich source of $\beta$ -carotene (Chen et al., 2016; Hiranvarachat et al., 2011). Its distinctive color is due to its high phenolic content ( $\alpha$ and $\beta$ ), important precursor of vitamin A in the human metabolism that is involved with the development and healthy function of teeth, bones, skin and eyes (Barzee et al., 2019).  |
| Celery    | <i>Apium graveolens</i> , Apiaceous                             | Rich source of protein, vitamins, carotenoids, fibers, phenolics, flavonoids and tannins with flavoring and healthy properties (Ingallina et al., 2020). It is characterized by the content of antioxidant compounds such as the flavones apigenin, luteolin and the flavonol quercetin, these having the ability to decrease pre and postprandial blood glucose levels in prediabetic elderly people without affecting plasma insulin levels, which could interfere with oxidative stress, associated with hyperglycemia (Yusni et al., 2018).  |
| Leek      | <i>Allium ampeloprasum</i> var. <i>Porrum</i> , Liliaceae       | Characterized by having a rich content of thiosulfonates, sulfides, polysulfides, methanethiol and other odorous sulfur compounds (Luo et al., 2014). It also contains significant levels of lutein, $\beta$ -carotene, vitamin C and vitamin E (Bernaert et al., 2012). It has antidiabetic properties and it can improve cardiovascular risk biomarkers (Benítez et al., 2017; Colina-Coca et al., 2017).  |