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Turning Agri-Food Cooperative Vegetable Residues into Functional Powdered Ingredients for the Food Industry

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Abstract: Current food transformation processes must face the food waste issue by developing valorization processes to reintroduce by-products in the economic cycle and contribute to circular economy, generating social and economic value, and ensuring permanence of agricultural and rural activities. In the present paper, the results of a collaboration project between a regional agri-food cooperative and university are summarized. The project aimed to revalorize a series of vegetable wastes (carrot, leek, celery, and cabbage) from the fresh and ready-to-eat lines of the cooperative, by producing functional powders to be used as functional food ingredients. Vegetables residues were successfully transformed into functional ingredients by hot air drying or freeze-drying, and variables such as storage conditions and grinding intensity prior to drying were considered. Twenty-five vegetable powders were obtained and characterized in terms of physicochemical and antioxidant properties. Results showed that drying (mainly hot air drying) allowed obtaining stable powders, with very low water activity values, and a significantly increased functionality. Vegetable waste powders could be used in the food industry as coloring and flavoring ingredients, or natural preservatives, or either be used to reformulate processed foods in order to improve their nutritional properties.

Keywords: vegetables waste; agri-food by-products; bio-waste valorization; bio-waste processing; food system sustainability; functional food ingredients

1. Introduction

Present food transformation processes need not only to focus on proper waste management, but they also need to be rethought to contribute to circular economy through the valorization of by-products, which are reintroduced in the economic cycle [1]. Continuity of agricultural and rural activities requires the development of new processes for the valorization of food industrial residues in order to generate social and economic value [2,3]. In this context, integral valorization, circular economy, process efficiency, biorefinery, or zero residue economy concepts contribute to describe present trends [4]. Valorization of food process by-products with the aim of contributing to food industry sustainability as well as developing circular economy systems cannot be considered an option but a must.

Agriculture is the world's most globalized industry. According to a recent publication by AgroPress and TOMRA sorting solutions foods [5], almost a third of worldwide food production is never eaten, which represents ~1.3 billion tons of food waste per year. In the particular case of fruits and vegetables the percentage of wastes is 45%, the United Nations Food and Agriculture Organization (FAO) having suggested values as high as 60%. More than a significant figure if considered that, in the particular case of Spain, over 13.4 million tons of vegetables and 18.4 million tons of fruit were produced in 2017 [6].

Fruits and vegetables are the most used commodities among horticultural crops, but their processing residues are still generally infra-utilized and considered a low value material. Fresh and processing industries generate significant losses and wastes which are becoming a serious nutritional, economical, and environmental problem [7]. The need for reducing the generation of food wastes and developing processes that allow their reuse by re-introducing the wastes or their components in the productive cycle has been discussed during years [8], but it still challenges all the agents involved. The sustainable development goals defined by the Food and Agricultural Organization of the United Nations especially focus on sustainability of food systems, as “sustainable consumption and production patterns” must be ensured, and especially mentions the coordination of global initiatives, activities, and projects on food losses and waste reduction.

Fruits and vegetables are consumed raw, minimally processed, or processed, because of their nutrients and health-promoting constituents [7]. In addition, agroindustrial wastes (such as seeds, peels, or pulp) generated in the different steps of the processing chains, present a high content of bioactive compounds, often even higher than that of the whole fruit [9,10]. Although the market currently offers a variety of foods which contain artificial preservatives, additives and/or taste improvers, modern consumers are more health-committed, buy food products more consciously, carefully read labels and decide in favor of natural foods, preferring low-processed foods without the addition of artificial preservatives or other additives. There is a continuous growing interest in such food, leading to new products permanently appearing on the market, including those which may contain fruit and vegetable industrial wastes [11]. This trend forces food producers to design and formulate foods containing natural additives, thus improving the nutritional value of juices, beverages, purees, or other food and, simultaneously, extending their shelf-life [7]. Nevertheless, the use of plant ingredients by the food and drink industry is still scarce, for which generating knowledge of their phytochemical properties is expected to be useful to increase their use as food ingredients.

Fruit and vegetable residues are perishable and have a very short shelf life. They are easily fermented while accumulated in the fresh processing plant facilities. For this reason, the preliminary treatment of the residues in the place where they are produced is expected to improve their durability as well as enable a better use and, additionally, help preserve the bioactive compounds it contains. The pretreatment (e.g., drying) of fresh residues immediately after processing to extend their durability could eliminate wastes and reduce environmental pollution.

On the other hand, reformulation of processed foods provides a genuine opportunity to improve people’s health by modifying the nutritional characteristics of commonly consumed processed foods [12], therefore addressing another one of the FAO sustainable development goals: “Ensure healthy lives and promote well-being for all at all ages” [13]. According to this goal, consumers must be encouraged to shift to nutritious and safe diets with a lower environmental footprint [13].

Fruit and vegetable powders have become a new way of consuming these products, which has gain importance along years [11,14,15]. Powdered foods are usually stable and presented in a concentrated and versatile form, for which they can be used in food formulation. Using food waste (vegetable waste in this case) to produce food powdered ingredients is an approach that contributes to the food chain sustainability and system circularity, by reusing a waste material and re-introducing it in the production cycle. In addition, vegetable waste powdered ingredients would have potential applications as coloring, savoring, or preservative agents, but they could also be used to increase the nutritional value of (processed) food, contributing to the development of nutritious and safe diets with a reduced environmental impact. Manufacturing powders from food waste may be a challenge considering the heterogeneity and structural differences among materials and edible/non-edible part of the same residue. From a technological point of view, parameters of unit operations such as cleaning, drying and milling (pre and post-drying) may have a relevant impact on the functional properties of the powder.

As described in the regional order “ORDEN 3/2018” (Conselleria de Agricultura, Medio Ambiente, Cambio Climático y Desarrollo Rural, Comunitat Valenciana, Spain) [16], the agricultural sector in the C. Valenciana bears a productive structure which is characterized by small, fragmented,

and disperse units. Agricultural cooperativism contributes to unify interests and improve the sector's competitiveness. Likewise, the Federation of Agri-Food Cooperatives of this region (Federació de Cooperatives Agroalimentàries de la Comunitat Valenciana) posts on the innovation and technologic development of this primary sector.

The present contribution summarizes a case study of collaboration of the Institute of Food Engineering for Development (IUIAD) of the Universitat Politècnica de València (UPV) and the Villena agrifood cooperative (Agrícola Villena, Coop. V), in the context of the Rural Development Program 2014–2020 of the C. Valenciana, funded by the European Agricultural Fund for Rural Development. The aim of the project is to develop processes to obtain functional powders to be used as food ingredients, from the wastes generated in the manufacturing lines of four selected vegetables (celery, carrot, cabbage, and leek), prepared raw to be cooked or, in the case of celery and carrot, ready-to-eat formats. These particular vegetables were chosen considering the ease of obtaining a clean (or easy to wash) residue as well as the expected bioactive constituents' content in the food. Yearly production of these vegetables residues is estimated by Agrícola Villena, Coop.V. to be ~178 tons in the case of leek and ~250 tons in the case of cabbage. The amount of carrot residues generated is significantly higher (almost 3000 tons) since most of this residue proceeds from the ready-to-eat line, in which discarding percentages increase dramatically.

To develop the processes, the effect of different process variables (fresh/freeze-dried, chopped/ground, air-dried/freeze-dried) on physicochemical and antioxidant attributes of powders, alongside with the study of the drying curves of the wastes, were studied. The present approach is a clear example of collaboration between primary sector-fresh vegetable industry and university to address the concept of global sustainability of food systems, focused not only on the environment, but also on food access and the development of technologies that increase bioavailability of bioactive compounds. Both functional food development and sustainability of the food system meet at this point.

2. Materials and Methods

2.1. Vegetables Waste Processing for Powder Manufacturing

In a first stage of the project, vegetable wastes were partially processed in the cooperative and further processed at the IUIAD facilities, as the objectives of the project included the study of drying conditions and drying curves, and the characterization of the powders obtained. In a second stage of the project, it is expected that the milling and drying stages are completed at the cooperative, once conditions of milling and drying are conveniently fixed and a solid recommendation for the equipment to be acquired is obtained as a result of the project.

2.1.1. Vegetables Waste Processing at the Cooperative Facilities

Among the different fresh products prepared and commercialized by Agrícola Villena, Coop. V, the ones identified as good candidates for the project were the cabbage and leek used for preparing combo vegetable trays (washed and cut, not chopped or minced) and the ready-to-eat lines (carrot and celery sticks). As introduced previously, the amount of wastes generated in the former is ~200 tons/year, whereas the amount of waste materials generated in the ready-to-eat lines can be higher than 2500 tons/year.

Leek and celery are washed with tap water and processed by hand (cutting with knife) to separate the edible part from the part that will be considered residue. In the particular case of cabbage, dirty or damaged leaves are removed by hand, and no washing is used. Carrots are processed by first washing with tap water, mechanically peeled, and cut in sticks in a mechanical cutter. Finally, carrot sticks undergo a second washing step with an ascorbic acid-water solution. Those sticks not meeting the quality standards are discarded and considered residue.

2.1.2. Vegetables Waste Processing after Reception at Iuiad Facilities

The vegetable wastes received were processed freshly (Fre) or stored in a freezer at $-22\text{ }^{\circ}\text{C}$ (Fro) until processing. When not washed at the cooperative facilities, residues were washed with chlorinated tap water before further processing. Prior to drying, particle size was reduced to pieces of medium size $\leq 10\text{ mm}$, called hereinafter chopped (C), or ground to particles $\leq 5\text{ mm}$ (G). Particle size reduction was performed in a lab mill according to the conditions specified in Table 1.

Table 1. Conditions for particle size reduction before drying, for the different vegetable wastes being processed.

	Carrot	Celery	White Cabbage	Leek (w)	Leek (g)
Ground (G)	10,000 rpm, 10 s (350 g)	10,000 rpm, 10 s (200 g)	10,000 rpm, 10 s + 10 s (150 g)	10,000 rpm, 10 s + 20 s + 10 s (150 g)	10,000 rpm, 10 s + 10 s (150 g)
Chopped (C)	5000 rpm, 5 s (350 g)	5000 rpm, 5 s (200 g)	5000 rpm, 5 s (150 g)	10,000 rpm, 5 s (150 g)	5000 rpm, 10 s (150 g)

Once milled, vegetable wastes were hot air dried (HAD) or underwent a freeze-drying process (FD). Air drying took place in a convective tray dryer (Pol-ekoAparatura, Katowice, Poland) at $70\text{ }^{\circ}\text{C}$, until water activity (a_w) was reduced to values below 0.3. Freeze-drying took place in a freeze dryer (Lioalfa-6, Telstar) for 24 h under freezing conditions and sub-atmospheric pressure ($P = 0.1\text{ mbar}$), with a previous freezing of the samples at $-40\text{ }^{\circ}\text{C}$ during 24 h. After dehydration, representative samples of each dehydrated residue were crushed to a fine grain size (10,000 rpm, 2 min at 30 s intervals) using a food processor (Thermomix[®], Vorwerk) to obtain the final powder. A total of 25 powders were obtained, 5 from each type of waste: Carrot (Ca), Celery (Ce), White cabbage (WC), Leek white portion (Lw), and Leek green portion (Lg). Powders were also identified by initial storing conditions: Fresh (Fre) or Frozen (Fro); the pretreatment applied: Ground (G) or Chopped (C); and the drying method used: Hot air drying (HAD) or Freeze-drying (FD).

2.1.3. Matrix Behavior during Air Drying: Drying and Drying Rate Curves

Kinetics of drying during the hot air-drying process are important as it is relevant to elucidate the duration of the constant drying rate period (CDRP), during which resistance for water transfer in the product is low as compared to evaporating rate and adiabatic conditions of drying prevail. It also allows estimation of the critical moisture content at which drying rate starts to decrease, indicating the beginning of the falling drying rate period (FDRP), when there is an internal control of mass (water) transfer. For this purpose, drying and drying rate curves of all the vegetable matrices were obtained during air drying at $70\text{ }^{\circ}\text{C}$. The analytical procedure consisted of placing each residue in two different trays for gravimetric determinations, and a third one for a_w measurements. Samples were weighed along the whole drying process (i.e., until reaching a water activity value below 0.3), at increasing time intervals, starting from 30 min. When drying finished, final moisture content of samples (x_{wf}) was determined by the following the official method established by the AOAC 934.06 [17], and the moisture content along the treatment was calculated (x_{wt}) by stating a dry matter balance (Equation (1)) along the drying process. Water mass fractions (x_w) were then transformed into wet basis (X_w) to plot both the drying curve (X_w/X_0 vs. time) and the drying rate curve ($-\Delta X_w/X_0/\Delta t$) vs. X_w/X_0 . Drying curves were obtained for the chopped and ground residues directly processed after reception (fresh) at the IUIAD pilot plant.

$$M_t \cdot (1 - x_{wt}) = M_f \cdot (x_{wf}) \quad (1)$$

2.2. Analytical Determinations of Raw Materials and the Obtained Powders

Powders were characterized in terms of physicochemical and antioxidant properties. Physicochemical characterization consisted of determining moisture content, water activity, total

soluble solids, and particle size. Antioxidant properties of powders were measured by qualifying phenol and flavonoid compounds, as well as antioxidant activity by the DPPH and ABTS methods.

2.2.1. Physicochemical Properties of Vegetable Wastes and Powders

Water activity (a_w) was measured by means of a dewpoint hygrometer (Aqualab 4TE, Decagon devices, Inc., USA); moisture content (x_w) was determined following the official method established by the AOAC 934.06 [17], which consists of determining the weight loss of the samples by drying them in a vacuum oven (Vaciotem, JP Selecta) ($P = 10$ mm Hg) at 60 °C, until constant weight. Total soluble solids (x_{ss}) were estimated from the Brix degrees measurement performed at 20 °C, obtained with a thermostatic refractometer (ABBE ATAGO 3-T, Japan). When necessary, water extraction of soluble solids was performed adding water in a 1:10 (w/v) ratio, for Brix measurements. All three properties were measured in triplicate. Particle size distribution was determined by laser diffraction using a Malvern Mastersizer equipment (Model 2000; Malvern Instruments Limited, UK), in dry and wet conditions. For the dry method, a dispersion unit Sirocco 2000 with air as dispersant at 2.5 bar of pressure and 60% speed were used. For wet measurements, a 1.52 refraction index was used for the sample, whereas 1.33 was used for the dispersed phase (water), the particle absorption index being 0.1. Results are the mean of five replicates and are given as equivalent volume diameter $D[4,3]$ and surface area mean diameter $D[3,2]$, together with the distribution percentiles d_{10} , d_{50} , and d_{90} .

2.2.2. Antioxidant Properties of Vegetable Wastes and Powders

Antioxidant properties of vegetable wastes and corresponding powders were evaluated by determining their phenol and flavonoid content, as well as their DPPH and ABTS antioxidant activities. Determinations were carried out on extract of samples, by using an 80% (v/v) methanol/water solution as solvent, and an extraction ratio of 0.5:10 (m/v). The mixture was continuously stirred during 1 h in a horizontal stirrer (COMECTA WY-100), and further centrifuged for 5 min at 10,000 rpm in a microcentrifuge (5804R, Eppendorf®). Measurements were performed on the separated supernatants (extracts).

Total phenolic content of samples was determined with the Folin–Ciocalteu method [18]. An aliquot of 0.125 mL of the previously prepared extract was mixed with 0.5 mL of distilled water and 0.125 mL of the Folin–Ciocalteu reagent (Sigma Aldrich). The mixture was allowed to react for 7 min in darkness before adding 1.25 mL of a 7% sodium carbonate solution to stop the reaction and 1 mL of distilled water until completing a volume of 3 mL. After 90 min in darkness, absorbance was measured at 760 nm with a spectrophotometer (Helios Zeta UV/Vis, Thermo scientific, UK). Results are given in mg of Gallic Acid Equivalents (GAE) per g of dry matter, and are the average of three replicates.

The colorimetric method of aluminium chloride [19] was applied to determine total flavonoid content of samples. One-and-a-half milliliters of the extract was mixed with 1.5 mL of a 2% w/v aluminum chloride in methanol solution, and vigorously shaken after mixture. The absorbance was measured at 368 nm after 10 min of reaction, and compared to a standard curve of quercetin (purity $\geq 95\%$, Sigma-Aldrich). Flavonoid content is given in mg of quercetin equivalents (QE) per gram of product, in dry basis, as the average of three replicates.

Antioxidant activity (AO) of the obtained powders and vegetable wastes was measured as Trolox Equivalents, by the DPPH and ABTS methods. The former evaluates the radical scavenging ability of sample against 1,1-diphenyl-2-picryl hydrazyl (DPPH \cdot), applying the method proposed by Brand-Williams et al. [20], which measures the change in color of a DPPH-methanol solution when reacting with the sample. An aliquot of 100 μ L of the extract was mixed with 200 μ L of a 0.1 mM solution of DPPH in methanol and 900 μ L of methanol. After 60 min in darkness, absorbance was measured at 517 nm in a spectrophotometer. Results were expressed in mg of Trolox Equivalent (TE) per gram of sample in dry basis. The ability to scavenge the ABTS $^+$ cation (2,20-azobis-3-ethyl benzthiazoline-6-sulphonic acid) was measured as described in Re et al. [21]. The radical ABTS $^+$ was released by reacting 7 mM of ABTS with potassium persulfate (2.45 mM) during 16 h at room

temperature and darkness. ABTS+ was mixed with phosphate buffer (pH 7.4) to reach an absorbance of 0.70 ± 0.02 at 734 nm. An aliquot of 100 μL of the sample was added to 2900 μL of the solution ABTS+ in phosphate buffer with an absorbance of 0.70 ± 0.02 and the absorbance of samples was read after 7 min. Distilled water was used as a reference. Presented results are the average of three replicates and are expressed in mg of Trolox Equivalent (TE) per gram of dry matter.

2.3. Statistical Significance of Results

Results were statistically analyzed using Statgraphics Centurion XVI Centurion XVI.I, Statpoint Technologies, Inc.) with a confidence level of 95% (p -value ≤ 0.05). Data were processed by performing simple and multifactor ANOVA. All analytical determinations were performed at least in triplicate.

3. Results and Discussion

3.1. Physicochemical Properties of the Vegetable Waste Powders

Physicochemical parameters including water activity (a_w), moisture content (% grams of water/100 total grams), and soluble solids content (x_{ss}) of the raw waste materials and dried powders are summarized in Table 2. Before processing, all vegetable wastes showed very high a_w values, indicating their perishability and high risk of spoilage. Drying of these wastes to produce the powders allowed reduction of a_w values below the target (0.3), with few exceptions. Carrot and celery freeze-dried powders exhibited water activities slightly higher than the expected, but in any case, safe levels were reached. As for moisture content, similar results were obtained, and a higher moisture content was obtained for carrot and celery freeze-dried products. On the other hand, more variability was observed among the different processing applied to the vegetable wastes, which suggested that pretreatments (freezing and milling) had different impacts on x_w values depending on the structure of the processed material. Raw vegetable wastes presented very high moisture contents (86–92%), which presumably contributed to the high a_w values and subsequent water availability to participate in spoiling reactions. Moisture content values were in the range of other fruit or vegetable (waste) powders such as blackcurrant, apple, or mango fruit pomace, as deduced from the literature [22–24]. Therefore, all the powders obtained were considered stable, since the amount of water present in the samples was very low, and not available to participate in spoilage reactions.

With regard to the drying process, air drying usually provided lower a_w and moisture content than freeze drying. This could be a consequence of air drying being prolonged until reaching the target a_w value, whereas a 24 h lyophilization cycle was applied in all cases, for which a_w was not registered until the end of the process when releasing vacuum and opening the chamber. It is also postulated that freezing prior to sublimation could have produced a more compacted bed, from which water was more difficult to sublimate and desorb, when completing the freeze-drying cycle. A similar behavior was observed for the HAD wastes that had been previously stored at freezing conditions: final water content of frozen and further HAD powders was higher in most cases, except for celery, which showed almost no significant differences, and carrot, which behaved differently to the rest of vegetables. Freezing could have an impact on the sample density by reducing porosity and thus generating a compacted bed with increased resistance to drying. The different behavior shown by the carrot waste could be explained by the particular (micro)structural characteristics of carrots, which present a more rigid matrix, less susceptible to compact [25].

Table 2. Water activity (a_w), moisture content (x_w) and soluble solids content (x_{ss}) of carrot (Ca), celery (Ce), white cabbage (WC), leek-white (Lw), and leek-green (Lg) waste powders obtained. Fro: frozen, Fre: fresh; HAD: air dried, FD: freeze dried; C: chopped; G: ground. Mean \pm standard deviation.

Waste/Powder	a_w	Moisture Content (%)	x_{ss} (g _{ss} /g _{dm})
Carrot (Ca)	0.996 \pm 0.004 ^c	87.32 \pm 0.02 ^f	1.155 \pm 0.013 ^c
CaFD_FroG	0.358 \pm 0.013 ^b	5.04 \pm 0.06 ^e	0.645 \pm 0.007 ^a
CaHAD_FreG	0.190 \pm 0.006 ^a	2.40 \pm 0.18 ^c	0.647 \pm 0.017 ^a
CaHAD_FreC	0.204 \pm 0.013 ^a	3.2 \pm 0.5 ^d	0.694 \pm 0.007 ^b
CaHAD_FroG	0.20 \pm 0.02 ^a	0.84 \pm 0.10 ^a	0.665 \pm 0.017 ^a
CaHAD_FroC	0.18 \pm 0.03 ^a	1.25 \pm 0.06 ^b	0.658 \pm 0.017 ^a
Celery (Ce)	0.994 \pm 0.008 ^e	91.7 \pm 0.4 ^d	0.522 \pm 0.007 ^b
CeFD_FroG	0.336 \pm 0.001 ^d	4.71 \pm 0.18 ^c	0.368 \pm 0.006 ^a
CeHAD_FreG	0.172 \pm 0.017 ^b	2.00 \pm 0.12 ^b	0.64 \pm 0.03 ^d
CeHAD_FreC	0.152 \pm 0.011 ^a	1.49 \pm 0.09 ^a	0.69 \pm 0.02 ^e
CeHAD_FroG	0.156 \pm 0.010 ^{ab}	1.1 \pm 0.3 ^a	0.579 \pm 0.017 ^c
CeHAD_FroC	0.208 \pm 0.005 ^c	1.26 \pm 0.17 ^a	0.546 \pm 0.017 ^b
White cabbage (WC)	0.999 \pm 0.003 ^d	90.2 \pm 0.2 ^e	0.725 \pm 0.007 ^e
WCFD_FroG	0.27 \pm 0.02 ^c	3.11 \pm 0.14 ^d	0.491 \pm 0.006 ^a
WCHAD_FreG	0.219 \pm 0.007 ^b	1.70 \pm 0.13 ^c	0.590 \pm 0.017 ^b
WCHAD_FreC	0.169 \pm 0.002 ^a	1.18 \pm 0.18 ^b	0.643 \pm 0.013 ^c
WCHAD_FroG	0.30 \pm 0.03 ^c	0.301 \pm 0.012 ^a	0.661 \pm 0.011 ^c
WCHAD_FroC	0.286 \pm 0.011 ^c	0.50 \pm 0.13 ^a	0.688 \pm 0.007 ^d
Leek (white) waste (Lw)	0.999 \pm 0.003 ^d	86.62 \pm 0.07 ^c	0.97 \pm 0.03 ^e
LwFD_FroG	0.088 \pm 0.013 ^a	1.3 \pm 0.3 ^b	0.48 \pm 0.02 ^a
LwHAD_FreG	0.200 \pm 0.019 ^c	1.02 \pm 0.11 ^b	0.549 \pm 0.011 ^{bc}
LwHAD_FreC	0.20 \pm 0.02 ^c	1.3 \pm 0.3 ^b	0.534 \pm 0.006 ^b
LwHAD_FroG	0.186 \pm 0.013 ^c	0.3 \pm 0.3 ^a	0.658 \pm 0.013 ^d
LwHAD_FroC	0.124 \pm 0.003 ^b	0.46 \pm 0.11 ^a	0.571 \pm 0.019 ^c
Leek green waste (Lg)	0.999 \pm 0.003 ^d	91.69 \pm 0.16 ^c	0.404 \pm 0.007 ^b
LgFD_FroG	0.079 \pm 0.003 ^a	1.6 \pm 0.5 ^b	0.34 \pm 0.02 ^a
LgHAD_FreG	0.264 \pm 0.009 ^c	1.38 \pm 0.10 ^b	0.501 \pm 0.013 ^d
LgHAD_FreC	0.261 \pm 0.013 ^c	1.6 \pm 0.5 ^b	0.432 \pm 0.017 ^c
LgHAD_FroG	0.14 \pm 0.02 ^b	0.51 \pm 0.16 ^a	0.435 \pm 0.017 ^c
LgHAD_FroC	0.100 \pm 0.014 ^a	0.28 \pm 0.09 ^a	0.446 \pm 0.013 ^c

Note: ^{a,b,c...} Different superscript letters for a similar residue indicate statistical significant differences at the 95% confidence level (p -value $<$ 0.05).

Particle size of powders was also determined, and characteristic parameters calculated, as previously described. This parameter was finally not obtained for the vegetable wastes stored at freezing conditions, as during the course of the project, it was decided to proceed only with fresh samples according to the results obtained (previous physicochemical and next antioxidant), and the economic and technical convenience of processing the samples directly after being generated at the cooperative facilities. Characteristic parameter values of fresh processed samples are summarized in Table 3.

There were statistically significant differences among samples, with regard to particle size and particle distribution. In general terms, powders obtained from a vegetable matrix ground prior to dehydration exhibit a smaller particle size, as compared to chopped samples. These results confirm that not only milling after drying, but also grinding/chopping prior to drying, determines particle size characteristics. As indicated by Djantou et al. [26], elucidating the interdependence of drying and milling results essential to obtain high-quality powders, with proper nutritional and physicochemical characteristics. The green part of leek was an exception, since the previously ground vegetable waste led to a bigger particle size than the chopped one. As occurred with carrot before, structure of the green part of leek significantly differs from the rest of vegetable wastes being processed. As indicated

later in this document (Section 3.3; drying curves), the green portion of leek dried significantly faster when chopped as compared to ground, which could have resulted in a more brittle structure, more easily milled after dehydration. Characteristic parameters obtained by the wet procedure were more heterogeneous among samples. In general, solubilization of soluble solids constituents in water during this determination led to a reduction in particle size parameters. Carrot powders were an exception. A possible explanation would be that smaller carrot powder particles would have completely solubilized, the bigger ones contributing more significantly to the average equivalent diameter calculated by the equipment. Graphs of particle size distributions are given as supplementary material in order to not to duplicate results; instead, percentiles d10, d50, and d90 are shown as an approximation of the distribution pattern. Distributions obtained by the wet procedure were more heterogeneous and, in occasions, a bimodal function was obtained after soluble solids solubilization. This suggests the existence of two main groups of particles with two different (average) particle sizes.

Table 3. Particle size characteristic parameters obtained by the wet and dry procedures: equivalent volume diameter D[4,3], surface area mean diameter D[3,2] percentiles d10, d50, and d90. Carrot (Ca), celery (Ce), white cabbage (WC), leek-white (Lw), and leek-green (Lg) waste powders obtained. Fre: fresh; HAD: air dried, FD: freeze dried; C: chopped; G: ground. Mean \pm standard deviation.

		DRY PROCEDURE				
		D[4,3]	D[3,2]	d10	d50	d90
Carrot	CaHAD_Ground	190 \pm 3 ^d	27.3 \pm 0.6 ^b	9.9 \pm 0.2 ^a	153 \pm 5 ^d	434 \pm 6 ^{ef}
	CaHAD_Chopped	300 \pm 15 ^g	35 \pm 4 ^d	12.0 \pm 0.9 ^{bc}	260 \pm 12 ⁱ	660 \pm 35 ^h
White cabbage	WCHAD_Ground	190 \pm 6 ^d	37 \pm 4 ^d	14.6 \pm 1.3 ^d	165 \pm 6 ^e	407 \pm 11 ^d
	WCHAD_Chopped	213 \pm 4 ^e	30.7 \pm 1.3 ^{bc}	11.3 \pm 0.7 ^{ab}	197 \pm 3 ^g	444 \pm 9 ^f
Celery	CeHAD_Ground	183 \pm 3 ^d	34.2 \pm 0.5 ^{cd}	14.9 \pm 0.2 ^d	136 \pm 3 ^c	428 \pm 8 ^{ef}
	CeHAD_Chopped	266 \pm 25 ^f	60 \pm 8 ^e	26 \pm 5 ^f	235 \pm 28 ^h	553 \pm 40 ^g
Leek (white)	LwHAD_Ground	110.6 \pm 1.6 ^a	22.9 \pm 0.6 ^a	9.7 \pm 0.4 ^a	82.4 \pm 1.6 ^a	259 \pm 3 ^a
	LwHAD_Chopped	156.0 \pm 1.8 ^c	34.8 \pm 0.4 ^d	16.7 \pm 0.3 ^e	128.1 \pm 1.5 ^c	339 \pm 4 ^c
Leek (green)	LgHAD_Ground	207 \pm 3 ^e	63 \pm 2 ^e	32.0 \pm 1.5 ^g	182 \pm 3 ^f	416 \pm 7 ^{de}
	LgHAD_Chopped	137 \pm 4 ^b	30.6 \pm 1.0 ^{bc}	13.2 \pm 0.6 ^{cd}	107 \pm 4 ^b	312 \pm 8 ^b
		WET PROCEDURE				
		D[4,3]	D[3,2]	d10	d50	d90
Carrot	CaHAD_Ground	165 \pm 26 ^{bc}	33 \pm 2 ^a	13.2 \pm 0.9 ^a	109 \pm 11 ^{bc}	398 \pm 68 ^{bc}
	CaHAD_Chopped	147 \pm 6 ^{ab}	32.5 \pm 1.3 ^a	13.6 \pm 0.6 ^a	96 \pm 6 ^{ab}	364 \pm 18 ^b
White cabbage	WCHAD_Ground	285 \pm 27 ^d	55 \pm 3 ^d	22.9 \pm 1.8 ^c	242 \pm 20 ^e	610 \pm 61 ^d
	WCHAD_Chopped	305 \pm 13 ^d	56.8 \pm 1.3 ^d	24.1 \pm 0.8 ^c	276 \pm 8 ^f	625 \pm 31 ^d
Celery	CeHAD_Ground	178 \pm 35 ^c	37 \pm 4 ^b	15.7 \pm 1.6 ^b	106 \pm 22 ^{bc}	447 \pm 82 ^c
	CeHAD_Chopped	151 \pm 17 ^b	33.6 \pm 1.6 ^a	14.0 \pm 0.6 ^a	99 \pm 10 ^{ab}	369 \pm 45 ^b
Leek (white)	LwHAD_Ground	155 \pm 9 ^{bc}	38.8 \pm 1.3 ^{bc}	15.9 \pm 0.5 ^b	120 \pm 7 ^c	349 \pm 20 ^{ab}
	LwHAD_Chopped	148 \pm 25 ^b	37.3 \pm 1.8 ^b	16.3 \pm 0.7 ^b	98 \pm 7 ^{ab}	334 \pm 52 ^{ab}
Leek (green)	LgHAD_Ground	165 \pm 6 ^{bc}	40.4 \pm 0.9 ^c	17.2 \pm 0.5 ^b	139 \pm 6 ^d	353 \pm 20 ^b
	LgHAD_Chopped	122 \pm 14 ^a	32 \pm 4 ^a	13.7 \pm 1.8 ^a	84 \pm 12 ^a	291 \pm 31 ^a

Note: ^{a,b,c...} Different superscript letters (dry or wet procedure) indicate statistical significant differences at the 95% confidence level (p -value $<$ 0.05).

3.2. Antioxidant Properties of the Vegetable Waste Powders: Phenols, Flavonoids, Dpph, and Abts Antiradical Capacity

Vegetable processing, including not only drying, but also pretreatments or storage conditions, has an impact on the antioxidant properties of vegetables. Processing conditions may promote the formation of compounds with novel antioxidant properties, thus maintaining or even enhancing the antioxidant potential of foods. In contrast, postharvest processing and storage can cause loss of

antioxidants or formation of compounds with pro-oxidant action, which may also lower the antioxidant capacity [27].

Figures 1 and 2 show the phenol and flavonoid content of the vegetable waste powder and their original waste, per gram of dry matter. White cabbage and leek (green portion) presented the highest phenolic concentration, calculated as gallic acid equivalents (GAE) per gram of dry matter. As compared to non-processed waste, total phenolic content usually increased when processed and, especially, when air drying was used to remove water (Figure 1). This was especially significant for white cabbage and carrot, and less significant for leek. The green part of leek and, especially, the celery, experimented a decrease in their phenolic content after processing. Phenolic constituents of celery were especially sensitive to high temperature, as this was the only case in which HAD produced a decrease in the phenolic content for all the powders, whereas FD resulted in an increased value. In this particular case, low temperature and vacuum conditions applied during FD would have better preserved phenolic constituents. The multifactor ANOVA analysis performed to the data obtained suggested that both pretreatment parameters (particle size reduction and storage conditions) significantly affected the results in a different way depending on the vegetable being dried (p -value of interaction ≤ 0.05). In the case of carrots, freezing had a significant negative impact on the phenolic content, whereas the effect of chopping vs. grinding was not significant. On the contrary, the white cabbage residue did not present significant differences with regard to storage conditions, but the chopped samples better preserved their phenolic content.

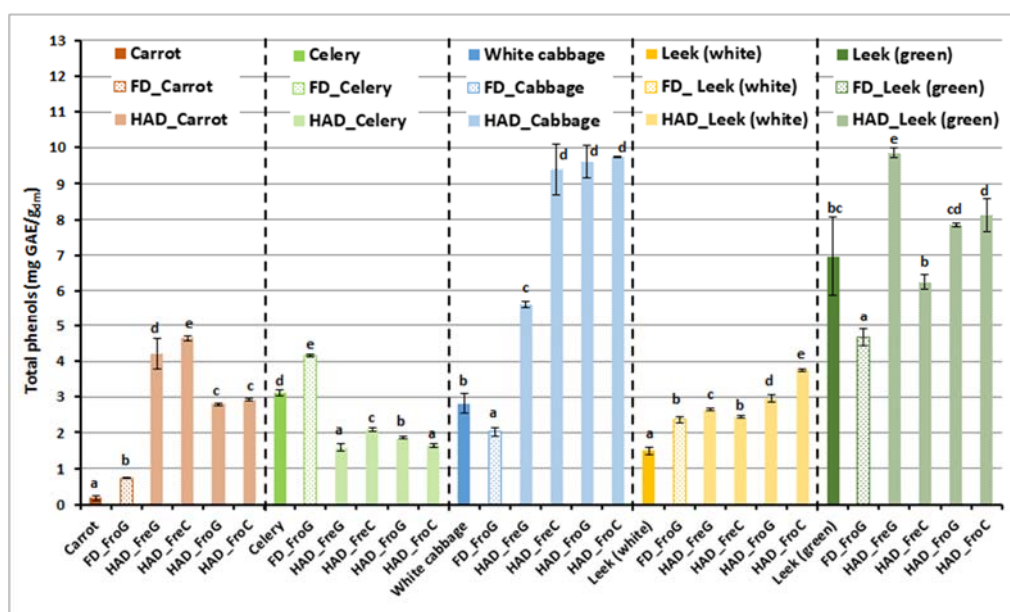


Figure 1. Total phenols content, expressed in mg of gallic acid equivalent (GAE) per gram of dry matter, in the raw bagasse (colored full bars) and vegetable waste powders. FD: freeze dried; HAD: air dried; Fro: frozen; Fre: fresh; G: ground; C: chopped. Mean values of three replicates. Bars indicate standard deviation. ^{a,b,c,d,e} Different superscript letters indicate statistical significant differences at the 95% confidence level (p -value < 0.05), for each vegetable waste.

As for flavonoid content (Figure 2), results were more heterogeneous. Flavonoids were especially present in the green part of leek and white cabbage, as in the case of phenols, the other residues and corresponding powders presenting a lower content. The effect of the variables analyzed was not statistically significant. This spectrophotometric procedure is especially influenced by the difference in absorbance between the major flavone in the sample and that one use as a reference value [28], although it is useful for comparison purposes.

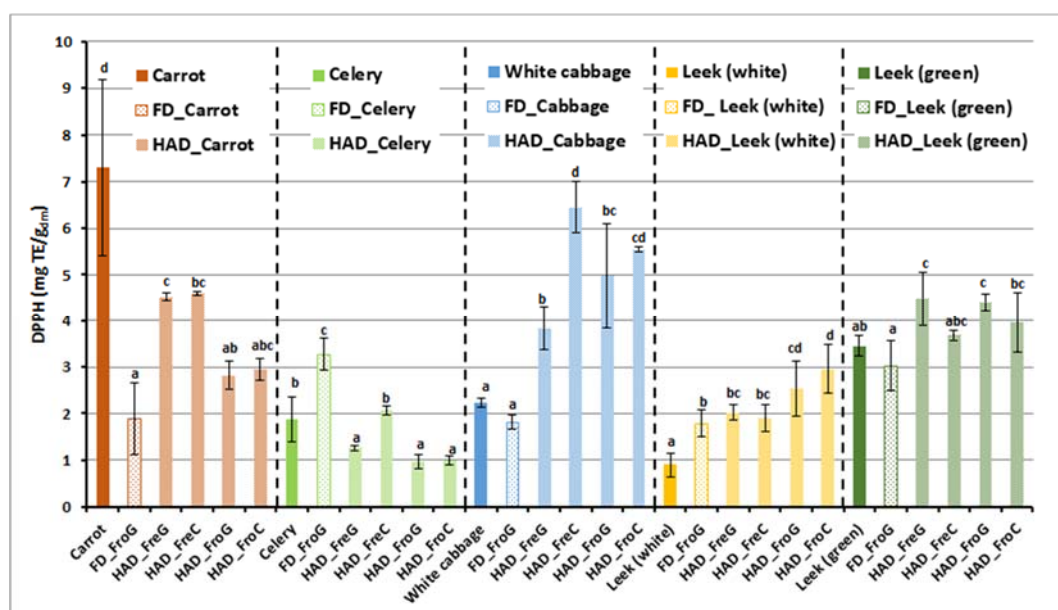


Figure 2. Total flavonoid content, expressed in mg of quercetin equivalents (QE) per gram of dry matter, in the raw bagasse (colored full bars) and vegetable waste powders. FD: freeze dried; HAD: air dried; Fro: frozen; Fre: fresh; G: ground; C: chopped. Mean values of three replicates. Bars indicate standard deviation. ^{a,b,c,d} Different superscript letters indicate statistical significant differences at the 95% confidence level (p -value < 0.05), for each vegetable waste.

DPPH and ABTS antioxidant (AO) activities are summarized in Figures 3 and 4. Again, AO properties of cabbage and green leek vegetable wastes and powders were significantly higher. In this case, also carrots, mainly non-processed, exhibited significant antioxidant properties. This difference with the previous analysis (phenols and flavonoids) was most probably due to carotenoids, one of the main bioactive compounds in carrots that exhibited significant AO activity, but are not detected by the previous methods [29,30]. The effect of processing variables depended again on the vegetable matrix being analyzed. In most cases, processing resulted in an increased AO capacity of samples. Storing in freezing conditions did not significantly affect leek and cabbage, although a negative effect was evidenced for carrot and celery. Except for celery, freeze-drying did not improve the AO capacity of powder, thus suggesting that, more than preserving the bioactive compounds present in the raw wastes, hot air drying would be inducing the formation of new antioxidant compounds. Grinding or chopping did not significantly affect the AO of powders in most cases. This implies that conditions of this stage will be selected as a function of drying behavior patterns, rather than functional properties of powders. Nevertheless, it must be taken into account that other important functional parameters such as fiber content may be affected by particle size [31], for which this parameter needs to be known before making a decision. In addition, more detailed information on specific bioactive compounds potentially present in the vegetable wastes and their powders, such as carotenoids in carrots [29], apigenin in celery [32], glucosinolates in white cabbage, [33] or sulfoxides in leek [34], would be convenient to know, to discriminate more precisely which components are negatively affected by processing and which others enhanced. For this purpose, High-Performance Laser Chromatography (HPLC) analyses are planned to be performed in the following months.

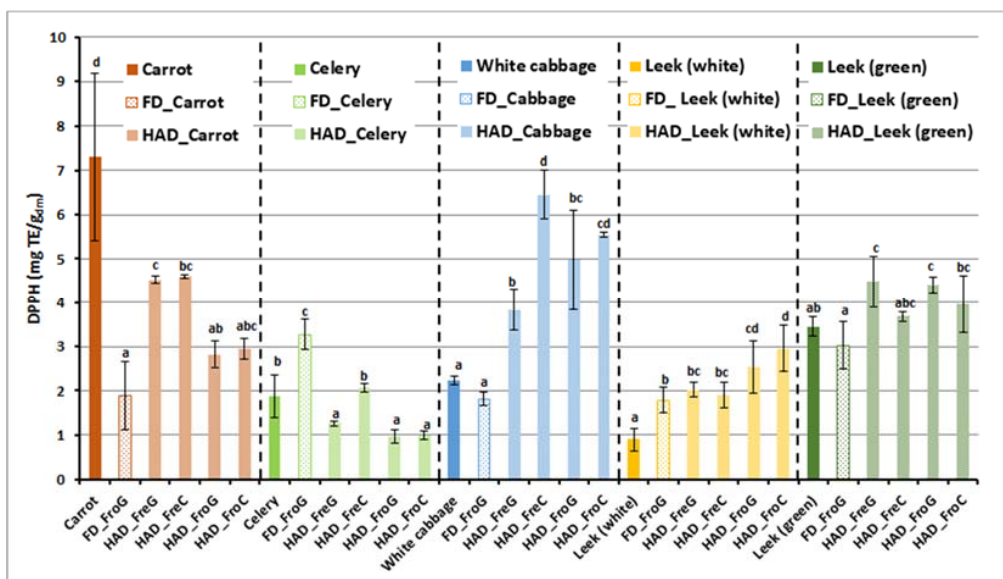


Figure 3. Results of antioxidant activity by the DPPH method, expressed in mg of trolox equivalent (TE) per gram of dry matter, of the raw bagasse (colored full bars) and vegetable waste powders. FD: freeze dried; HAD: air dried; Fro: frozen; Fre: fresh; G: ground; C: chopped. Mean values of three replicates. Bars indicate standard deviation. ^{a,b,c,d} different superscript letters indicate statistical significant differences at the 95% confidence level (p -value < 0.05), for each vegetable waste.

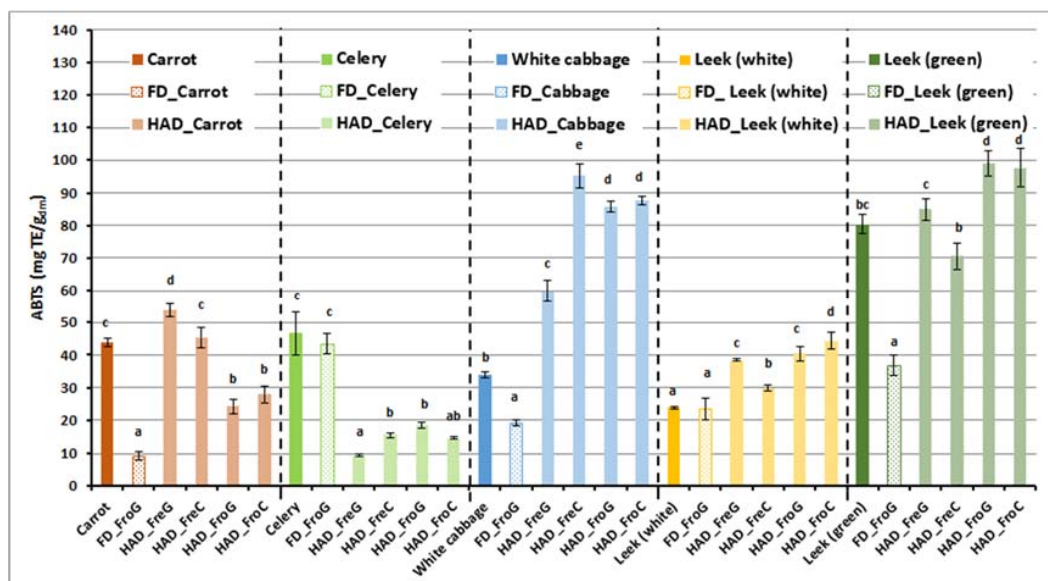


Figure 4. Results of antioxidant activity by the ABTS method, expressed in mg of trolox equivalent (TE) per gram of dry matter, of raw bagasse (colored full bars) and vegetable waste powders. FD: freeze dried; HAD: air dried; Fro: frozen; Fre: fresh; G: ground; C: chopped. Mean values of three replicates. Bars indicate standard deviation. ^{a,b,c,d,e} different superscript letters indicate statistical significant differences at the 95% confidence level (p -value < 0.05), for each vegetable waste.

3.3. Drying and Drying Rate Curves of Vegetable Wastes, As Affected by Each Vegetable Matrix

Figure 5 shows the drying curves obtained, i.e., the variation in water content (dry basis) in the reduced form (X_w/X_0) against processing time, during air drying at 70 °C of a 10 mm thick layer of the vegetal wastes subjected to different pretreatments which affect the structure matrix. Drying curves provide information about the exposure time to air under certain conditions that is required to reach a specific moisture content. Obviously, samples requiring less time would dry faster. In all the analyzed

products, except for carrot waste, chopping instead of grinding was recommended to shorten the drying time. A more compact structure might have resulted after crushing, so that the superficial area in direct contact with the air stream would be reduced as compared to the respective chopped sample. This effect was particularly evident in the green portion of leek, in which the drying time required to reduce the initial moisture content by 80% increased from 260 min (4.3 h) for chopped samples to 470 min (7.8 h) for ground samples. However, these differences were much less pronounced in the case of white cabbage residue and the white portion of leek residue, thus suggesting that the structure of chopped and ground samples were more similar for those products. As for carrot residue, the time required to reduce the initial moisture content by 80% decreased from 420 min (7 h), for chopped samples, to 350 min (5.8 h), for ground samples. Carrot residue is known to have a considerable rigid structure, as indicated previously, for which grinding may have favored the breakage of such structures and the release of the water retained inside [25].

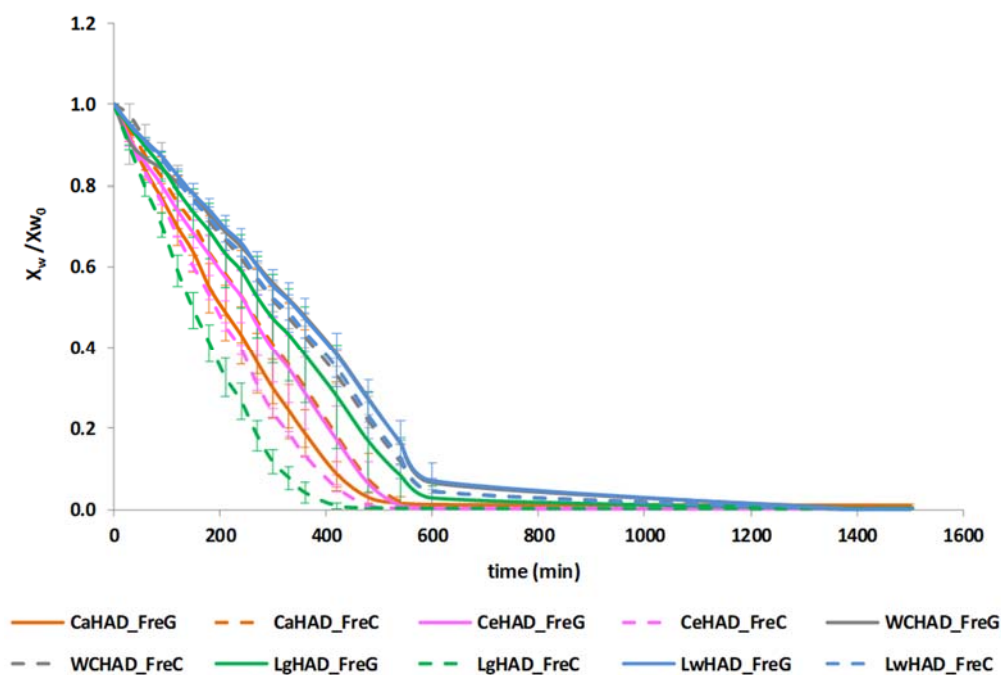


Figure 5. Drying curves (moisture content in g_w/g_{dm}) of chopped (C) and ground (G) vegetable wastes. Ca: carrot; Ce: celery; WC: white cabbage; Lg: leek green; Lw: leek white.

Drying rate curves (Figure 6) not only confirmed the already mentioned results with regard to matrix and pretreatment impact on drying behavior of samples, but also evidenced that most of the water in the residues was eliminated at the constant drying rate period (CDRP) under constant drying conditions. Drying rate during the CDRP ranged between $0.019 g_w \cdot g_{dm}^{-1} \cdot \text{min}^{-1}$, for both chopped and ground white portions of leek residue, and $0.053 g_w \cdot g_{dm}^{-1} \cdot \text{min}^{-1}$, for chopped celery. The study of drying kinetics is also relevant because it allows discriminating between the CDRP in which resistance to water transfer in the product is low as compared to evaporating rate, for which there is external control of mass transfer and adiabatic conditions of drying prevail, from the falling drying rate period (FDRP) in which there is internal control of mass (water) transfer. The inflection point between both periods indicates the critical moisture content, at which drying rate starts to decrease. In the vegetable wastes studied, critical moisture content was in most cases below 80% of the initial one. As long as the water content of the solid is higher than the critical one, the product could be dried at high temperature without significantly affecting their functional properties; thus, temperature effect on dried matrices would become more important during the FDRP. According to these results, if thermolabile compounds are to be preserved, the drying process could be improved by reducing the dryer temperature when reaching critical moisture content.

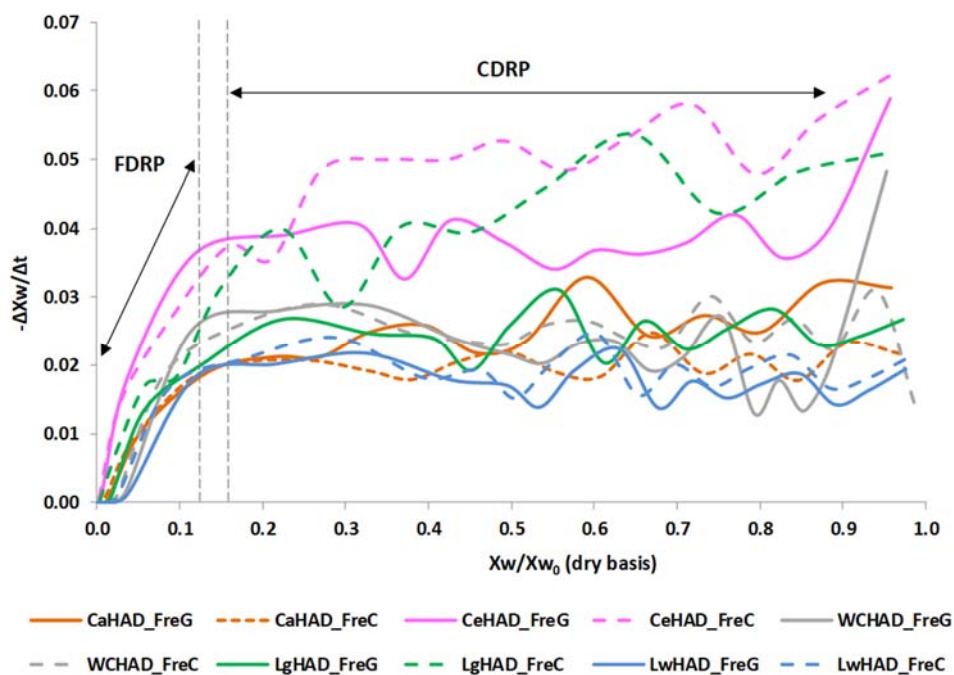


Figure 6. Drying rate curves (moisture content in g_w/g_{dm}) of chopped (C) and ground (G) vegetable wastes. Ca: carrot; Ce: celery; WC: white cabbage; Lg: leek green; Lw: leek white. CDRP: constant drying rate period; FDRP: falling drying rate period.

4. Conclusions

The sustainable development goals defined by the Food and Agricultural Organization of the United Nations especially focus on the sustainability of food systems and especially mentions the coordination of global initiatives, activities, and projects on food losses and waste reduction. Fruit and vegetables industries (fresh and processed) significantly contribute to food waste numbers, participating of an increasing nutritional, economical, and environmental problem. There is urgent need for the development of processes which allow their reuse by re-introducing the wastes in the productive cycle, contributing to the circular economy, engaging all the agents involved.

The present study is an example of successful collaboration between primary producers (agri-food cooperative) and university, together with regional government as facilitators of European funds for rural development and sustainable development. Vegetables residues have been successfully transformed into functional ingredients by a series of processes involving pretreatments and drying stages, aimed at maximizing bioactive compounds and their antioxidant activity. Results showed that drying allowed obtaining stable powders, with very low water activity values and a significantly increased functionality. The powders obtained could be used in the food industry as coloring and flavoring ingredients, or either as natural preservatives. In addition, they also have application in the reformulation of processed foods to improve their nutritional properties, and thus participate of another of the FAO goals: Ensure healthy lives and promote well-being for all at all ages. At present, the project continues on evaluating the effect of processing parameters on the concentration of specific bioactive constituents and dietary fiber, both being important phytochemicals in the fight against non-communicable diseases such as obesity and related disorders, which are becoming an epidemic and a major public health concern. A proposal of pilot plant is also being developed so as to manufacture the powders at the cooperative facilities, where wastes are produced, to evaluate economic feasibility of the processes.

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L.S., and C.B.; Writing—reviewing and Editing: L.S.; Supervision and Project administration: L.S. All authors have read and agreed to the published version of the manuscript.

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