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ORIGINAL PAPER

Revalorisation of broccoli crop surpluses and field residues: novel ingredients for food industry uses

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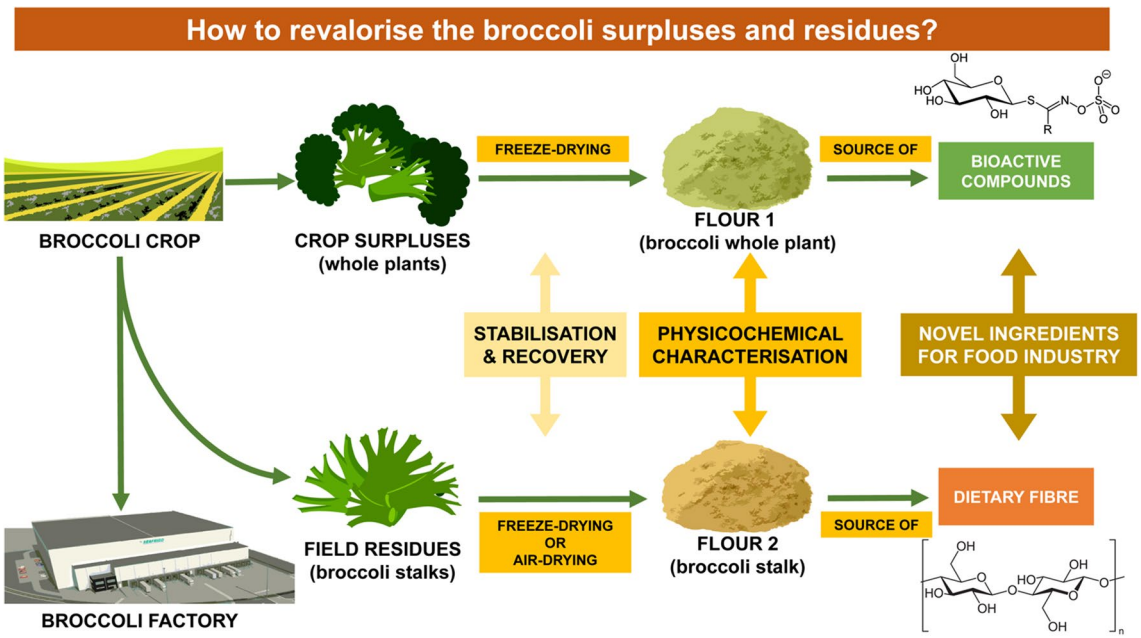
Abstract

Research on the management of broccoli crop residues and surpluses is critical for improving agricultural practices, optimizing food industrial manufacture, and contributing to better human nutrition. The objective of this study was to obtain novel ingredients based on these residues and surpluses for a wide range of applications in the food industry. The effect of air-drying (60 °C or 80 °C) applied to field residues (broccoli stalks), mimicking industrial conditions, was compared with dehydration by freeze-drying applied to these same field residues and to crop surpluses (broccoli whole plant). Thus, different broccoli flours were obtained and characterised for technological and biological properties including colour, antioxidant activity, nutrients, total polyphenol content, and content of glucosinolates. Flours from field residues showed high levels of dietary fibre (≈22% dry weight). Broccoli crop surpluses flours had relevant contents of glucosinolates (≈13 mg/g dry weight). Therefore, within the framework of the circular economy, these flours are proposed to revalorise the two main broccoli crop discarded fractions. Such flours could be used in a realistic and simple way by the agri-food industries interested in the development of healthy and conscience foodstuffs, in a cost-efficient manner.

Graphical abstract



Keywords Sustainability · Plant by-products · Biocompound recovery · Vegetable flour · Polyphenols · Glucosinolates

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Introduction

Broccoli (*Brassica oleracea*) is a plant with a valuable nutrient composition for the content of dietary fibre, minerals (potassium and zinc), vitamins (ascorbate and phylloquinone) and with a healthy profile because of the presence of bioactive compounds, specially glucosinolates (GLS) and flavonoids, among other substances [1]. The broccoli parts for human consumption match the inflorescences (florets) and the peduncles (stems), while the inedible parts such as the leaves and stalks (up to 70% of the total mass of the plant) remain as agricultural residue [2, 3]. The broccoli residue is either discarded or intended for animal feed. Spain is one of the main producers of broccoli in the European Union with around 500,000 tons in 2022 [4], and the large generation of agricultural residues [5] has led to scientific, technological, and economic interest in re-evaluating them as a source of bioactive compounds for potential health benefits [3]. Several studies have been conducted to characterise the composition of the plant parts that were discarded for human food (inflorescence, leaf, and stem) [6–13]. It is important to note that, even though the raw material used in those studies was defined as broccoli by-products, they were not actual residues of broccoli intensive farming. Only one work was based on industrial discards consisting of a mix of florets and stalks [14]. In addition, the processing of broccoli for non-food purposes requires preliminary dehydration for subsequent management. Dehydration methods such as microwaving [8, 15, 16], freeze-drying [17–19] and air-drying [9, 16, 19–21] have been applied at laboratory or pilot plant level on broccoli samples. Nevertheless, none of the samples evaluated were agri-food residues. These dehydration methods had a varied effect on the composition of broccoli parts (florets, leaves, stems, and stalks). Concerning broccoli by-products, the effect of dehydration process has also been evaluated [15, 22] and most of the studies are focused on their characterisation and further application once stabilised [6, 11, 23, 24]. Regarding GLS, it should be kept in mind that they are stable in the whole plant. However, they are partially degraded to their metabolites, isothiocyanates, during harvesting procedure [25, 26], technological processing (blanching, quick-freezing, vacuum freeze-drying, vacuum-drying, and oven-drying) [17, 27] or cooking [28, 29]. The research hypothesis of the present study was that the stabilisation of agricultural broccoli discards, including crop surpluses and residues prior to processing, allows obtaining ingredients rich in dietary fibre and bioactive compounds oriented to the food industry. The main objective of this study was to obtain novel ingredients based on these broccoli by-products with a wide range of applications in the food industry. Three specific objectives were proposed to achieve the main objective: (i) to analyse the composition of crop surpluses (whole plant) as a potential food ingredient; (ii) to characterise field residues (mostly stalks) after optimisation of the dehydration process (freeze-drying or air-drying); (iii) to determine the macronutrients and the bioactive contents and antioxidant activity of flours according to drying process.

Materials and methods

Raw materials reception

The broccoli surpluses from crop fields and the field residues were provided by a local agri-food co-operative (Grupo AN, Tajonar, Spain). About 60 kg of crop surpluses and 80 kg of field residues were received fresh. The crop surpluses were whole broccoli plants, considered to be of low quality because of their small size inflorescences, and deemed as oversupply for fresh-market distribution by the industry. The field residues were the stalks discarded after broccoli harvesting and left in the field, not submitted to further processing. The raw materials were collected in March–May 2022 from the Navarre (Spain) winter crop campaign.

Raw materials conditioning and flours obtention

The initial characterisation was performed to estimate the moisture content, as well as on the fractioning of broccoli surpluses to evaluate the percentage that each fraction (inflorescence, stalk, and stems) referred to the whole broccoli plant. Thereafter, batches of 3–5 kg of all raw materials (crop surpluses and field residues) were prepared and frozen until dehydration processing (air-drying or freeze-drying).

The air-drying (AD) of broccoli stalk field residues was carried out in a model D4AFY semi-industrial hot AD cabinet with 4 trays (Kowell Ind. Corp., Gyeonggi-do, South Korea) loaded with 3–5 kg of sample homogeneously distributed in thin layers. The dehydration was monitored at 60 °C (AD-60) and 80 °C (AD-80) until equilibrium was reached, and drying kinetics were determined from steady-state weight loss data. The drying rate (DR) was calculated from the free moisture content values obtained during drying according to formula:

$$DR\ (\text{kg water/kg dry weight/h}) = \left(X_{n-1} - X_n\right)/\left(t_n - t_{n-1}\right),$$

where X_{n-1} and X_n are the free moisture contents at two consecutive sample times t_{n-1} and t_n. The free moisture content was calculated by following formula:

$$X_n(\text{kg water/kg dry weight}) = \left(W_n - W_e\right)/W_{\text{e}},$$

where W_n and W_e are the weight of the product at drying time n and at the equilibrium, respectively. The AD rate curve (drying rate vs. moisture on dry basis) and the moisture curve (moisture on dry basis vs. time) were plotted.

The freeze drying (FD) was performed using a semi-industrial freeze dryer (LyoBeta, Telstar Spain, Terrassa, Spain) for a load of 4 kg evenly distributed in 4 trays. A three-step FD procedure was implemented: (1) a freezing step performed in a ramp of 2 h until reaching –45 °C and then maintained for 3 h; (2) a primary drying step conducted for 72 h at a tray temperature of –10 °C and a chamber pressure of 0.150 mbar; (3) a secondary drying step for 2 h at 0 °C and 0 mbar. The entire FD process was monitored by various temperature probes, which enabled process control. Broccoli surpluses were freeze-dried as well, prior to their characterisation, and were used as reference (control).

Dehydrated samples from each batch were grounded using a cooking robot (blade speed 10 for 1 min; Thermomix® TM6, Vorwerk España M.S.L., Madrid, Spain). The products obtained (hereinafter, flours) were then vacuum packed, in bags of high barrier to oxygen and moisture, for further use and characterisation. Once dried and stabilised, the broccoli samples were pulverised and characterised.

Physical characterisation of broccoli stalk flour

The flours from the processed broccoli stalks (field residues) were characterised to evaluate the impact of dehydration process on their traits. Particle size distribution (percentage by size) of the flours was assessed by sieving 50 g samples through 150 µm to 5 mm plates with a vibratory shaker (Retsch Gmgh, Haan, Germany). Colour was evaluated from 2 cm thick opaque layers of each flour and 1 cm radius area, with a DigiEye system (VeriVide Ltd., Leicester, UK) and the CIEL*a*b* colour space [30]. Water absorption index (WAI) was evaluated according to Djeghim et al. [31] with some modifications. One g of each flour was dispersed in 30 mL of deionized water. The mixture was stirred in a vortex at 1000 rpm for 30 s and left for hydration during 24 h at 20 °C. Thereafter, samples were centrifuged at 15,000 rpm for 20 min at 5 °C (Sigma 3-30KS and 12,156 H rotor, Osterode, Germany). After supernatant discard, samples were weighted, and WAI was expressed as g of water gained per g flour.

Proximate characterisation of flours

Characterisation of the flours from both broccoli surpluses and broccoli stalks was performed. The crude protein content was estimated by Kjeldahl method, according to the standard ISO 2013 [32], applying a nitrogen-protein conversion factor of 6.25. Total lipid content was quantified using Soxhlet extraction [33]. Total ash contents were estimated gravimetrically by incineration at 550 °C for 12 h, following the standard ISO 2007 [34]. Total dietary fibre was estimated with an assay kit (Megazyme Ltd, Bray, Ireland) based on enzymatic method [35].

Total polyphenol content and antioxidant activity

For total polyphenol content (TPC) determination, a 25% (w/v) ethanol hydroalcoholic (70%, v/v) extractions of each flour (30 rpm agitation for 30 min at 20 ± 3 °C, centrifuged at 10,000 rpm for 15 min at 25 °C) were previously done. The TPC was quantified from the improved Folin-Ciocalteu method [36] and results were expressed as mg gallic acid (GA) equivalents. Antioxidant activity (AA) was evaluated by the DPPH free radical method described by Brand-Williams et al. [37] and results were expressed as percentage of DPPH inhibition, as well as mmol Trolox equivalents/L. TPC and AA analyses were performed spectrophotometrically using a Multiskan GO Microplate Spectrophotometer (Thermo Fisher Scientific Inc, Madrid, Spain).

Glucosinolate content of flours

Identification and quantification of glucosinolates present in the flours was carried out following the protocol of Baenas et al. [38]. Dry samples (100 mg) were extracted with 1.5 mL of 70% (v/v) methanol by heating at 70 °C in a water bath for 30 min, with agitation every 10 min using a vortex. Subsequently, samples were cooled in an ice bath for 5 min and centrifuged (30,000*g*, 5 min, 4 °C). The supernatant was filtered through a 0.22 µm PVDF syringe filter (Filter-Lab®, Barcelona, Spain).

Chromatographic separation was performed on a Kinetex 5 µm C18 100 A°, 250 × 4.6 mm column (Phenomenex, Macclesfield, UK). The mobile phases used were A) water at 0.1% formic acid and B) acetonitrile at 0.1% formic acid, with a flow rate of 1 mL/min. The linear gradient started with 1% solvent B, reaching 17% solvent B at 15 min, 25% at 22 min, 35% at 30 min, 50% at 35 min, 9% at 40 min, which was maintained until 45 min, 1% at 47 min, which was kept until 52 min. The injection volume was 20 µL. Chromatograms were recorded at 227 nm.

HPLC–DAD-ESI-MSn analyses were carried out on an Agilent HPLC 1200 (Agilent Technologies, Waldbronn, Germany) coupled to an ion trap spectrometer (HCT Ultra, Bruker Daltonics Corp, Bremen, Germany), equipped with an electrospray ionization interface (ESI), and using Data Analysis software (Bruker Daltonics Corp, Bremen, Germany) for data processing. Gas temperature and capillary voltage were set to 350 °C and 4 kV, respectively. The nebulizer pressure and nitrogen flow rate were 65.0 psi and 11 L/min, respectively. The full mass scan covered the *m/z* range 50–600. Helium was used as collision gas with

voltage ramp cycles from 0.3 to 2 V. Mass spectrometry data were acquired in the negative ionisation mode. Identifications were carried out by the characteristic *m/z* signal of each compound, their MS^2 [M–H]− fragmentations and relative times. Then, the extracted samples were quantified in a Waters HPLC–DAD system with Millenium 32® Chromatography Manager software for data acquisition and management (Waters Cromatografía S.A., Barcelona, Spain), following their UV spectra and the elution order previously described for the acquisition conditions. The glucosinolates were quantified using sinigrin as external standard (in the case of aliphatics) and glucobrassicin (for indole GLS) (PhytoPlan Diehm & Neuberger GmbH, Heidelberg, Germany).

Statistical analysis

Three batches were performed for each drying process applied to the raw materials. Extractions and determinations were carried out in triplicate. The mean and standard deviation of the replicates were used as descriptive statistics. The effect of the drying process (FD, AD-60, AD-80) to achieve the stabilisation of the broccoli stalk residues and broccoli surpluses was analysed by means of the analysis of variance (one-factor ANOVA), together with HSD multiple comparison analysis using Tukey's test, with a significance level of 95% with Statgraphics 18.0 (Statgraphics Technologies Inc., Virginia, USA) as the associated software.

Results and discussion

Drying of broccoli field residues (stalks)

The drying kinetics curves of the broccoli field residues (stalks), at 60 °C and 80 °C, are shown in Fig. 1. These curves exhibit the changes in drying rate as a function of moisture (drying curve), as well as the change in moisture content as a function of time (moisture curve).

An initial constant drying rate period can be observed at both drying temperatures, until the free moisture of the stalks achieved a critical value of 4–4.5 kg water/kg DW (Fig. 1A). During this period, the drying rate was around two times higher at 80 °C (2.4 g water/g DW*h) than at 60 °C (1.2 g water/g DW*h). Once the free moisture decreased below the critical moisture, a period of decreasing drying rate occurred, with a faster drop being observed at 80 °C than at 60 °C. The behaviour of the moisture curve (Fig. 1B) was analogous to that obtained by Dufoo-Hurtado et al. [39], who reported a greater loss of moisture at 80 °C than at 60 °C during the drying period in the first 5 h, reaching the equilibrium earlier. The processes that govern the drying kinetics are mainly two: the surface diffusion of water in the first phase, and secondly the diffusion of liquid and/or vapor from the inner parts of the product in the falling rate period, due to the differences in moisture that are established as drying progresses [40]. Once AD process finished, the moisture content of the flour samples was around 1–2% (Table 1), lower than the 7–9% obtained by other authors [16, 21] but similar to values of 1% reported by Cao et al. [8] and Doymaz [20], and 3% by Dufoo-Hurtado et al. [39]. By contrast, flours processed by FD showed moisture values around 6%, higher than values between 2 and 4% reported by Shi et al. [12]. The higher residual moisture retention observed could be due to the actual heterogeneity of the plant material and the uneven size distribution of the material introduced into the freeze-dryer.

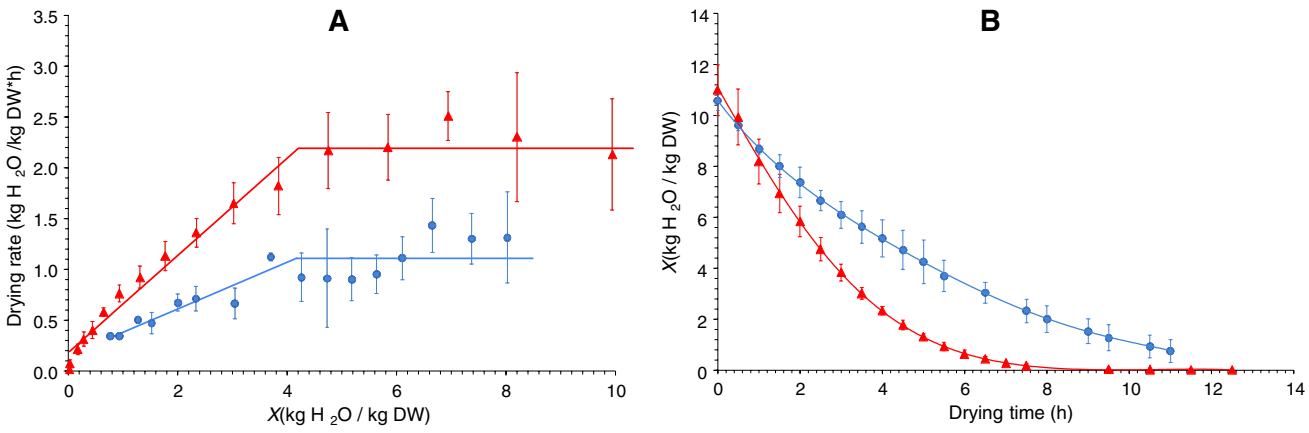

Fig. 1 The air-drying rate (**A**) and moisture curve (**B**) of the stalk residues according to temperature (square 60 °C; triangle 80 °C). Data are expressed as mean ± standard deviation

Table 1 Physical characteristics of broccoli flours from field residues (stalks) stabilised by freeze-drying (FD) and air-drying at 60 °C (AD-60) and 80 °C (AD-80)

Parameter	FD	AD-60	AD-80
Moisture (%)	5.71 ± 0.51[a]	2.22 ± 0.50[b]	1.19 ± 0.15[c]
Colour			
Lightness (*L**)	78.10 ± 0.61[a]	69.84 ± 0.69[b]	57.50 ± 0.12[c]
Redness (*a**)	− 6.29 ± 0.05[c]	4.64 ± 0.25[b]	9.05 ± 0.19[a]
Yellowness (*b**)	34.81 ± 0.73[a]	29.90 ± 0.34b	28.38 ± 0.10[c]
Particle size (%)			
< 150 µm	22.16 ± 6.78	20.37 ± 1.75	23.09 ± 0.95
150–450 µm	41.02 ± 3.62	40.62 ± 1.06	43.19 ± 0.80
450–850 µm	29.10 ± 8.59	29.95 ± 1.29	26.80 ± 0.45
> 850 µm	7.72 ± 2.37	9.07 ± 1.48	6.91 ± 0.93
WAI (g water/g DW)	8.87 ± 0.69[b]	10.13 ± 0.36[a]	7.55 ± 0.47[c]

Data expressed as mean ± SD ($n = 9$). Different superscript letters in the same row indicate statistically significant differences according to the HSD Tukey test at the 95% confidence level ($\alpha = 0.05$)

DW dry weight, *WAI* water absorption index

Technological characteristics of broccoli field residues flours

The results of the technological characterisation of broccoli field residue flours are listed in Table 1. The three flours (FD, AD-60, and AD-80) presented a similar granulometry. About 60–66% was made up of fractions with a particle size of less than 450 µm. Regarding the colour of the flour samples, as expected, the higher the drying temperature, the lower the luminosity and the greater the development of brown tones. FD flour was the only one that maintained green tones and its brightness values were significantly higher with respect to AD, in agreement with Bas-Bellver et al. [13]. Several authors have pointed out that drying broccoli with hot air reduces the chlorophyll content and, therefore, increases the values of *a** coordinate [8]. The development of reddish-brown tones in AD-80 flour could be explained because of the development of polymers (melanoidins) at that drying temperature, as a product of the oxidation and complexation of proteins, polyphenols, and polysaccharides [41]. In this sense, bleaching of broccoli prior to heat treatment would have stabilised the green colour, inhibiting browning reactions, especially in AD even at higher dehydration temperatures. In order to propose a technically and economically feasible method for valorising the by-product for an actual industrial application, it was determined not to bleach the by-product.

Regarding WAI, it is important to note that this parameter is not only a functional property relevant for rehydration, but, from a physiological perspective; high values in foods can contribute to the formation of the food bolus and facilitate gastrointestinal peristalsis [42]. AD-60 flour was the one with the highest water absorption capacity, followed by FD (which started from an already higher moisture content) and AD-80 flour. The lower WAI of the AD-80 sample can be attributed to the effect of drying temperature on cell retraction, thereby reducing the capacity to absorb water during rehydration [40]. In any case, the values obtained fall within the range specified for this particular type of flours, ranging from approximately 8–10 kg water/kg sample [24, 39].

Composition of crop surpluses and broccoli flours

As shown in Table 2, the density of macronutrients in the flours was high as a consequence of their low moisture content. All samples presented a high protein content (around 13%), in the range described in literature for broccoli by-products and comparable to foods considered source of protein [39, 43]. Fat levels were less than 2% and total carbohydrate content was around 75% (DW), of which about 20% corresponded to dietary fibre. Total ashes were around 10%, similar to those reported by other authors [24, 43]. Regarding the type of dehydration, the content of nutrients was similar in all flours. Thus, the industrial thermal process did not significantly impact the nutritional profile, in agreement with other authors [39].

Dietary fibre corresponds to the fraction of carbohydrate polymers that are neither digested nor absorbed and have beneficial properties for health, such as reducing/slowing

Table 2 Proximate composition (g/100 g DW) of flours from crop surpluses (flour 1) and field residues (flour 2) stabilised by freeze-drying (FD) and air-drying at 60 °C (AD-60) or 80 °C (AD-80)

Parameter	Flour 1 (broccoli whole plant)	Flour 2 (broccoli stalk)		
	FD	FD	AD-60	AD-80
Raw protein	19.08 ± 0.08	14.37 ± 0.41	13.36 ± 0.53	12.76 ± 0.62
Raw fat	1.67 ± 0.70	0.89 ± 0.02	1.34 ± 0.15	0.93 ± 0.54
Total carbohydrates	72.83 ± 5.33	82.27 ± 9.60	76.43 ± 1.07	76.85 ± 1.90
Dietary fibre	13.28 ± 6.50	17.71 ± 0.57	22.87 ± 2.02	22.34 ± 1.17
Total ashes	10.22 ± 0.08	10.55 ± 0.12	9.17 ± 0.55	9.59 ± 0.01

Data expressed as mean ± SD ($n = 9$)

Total carbohydrates = 100 − Σ (proteins + fats + ashes)

the absorption of cholesterol and glucose, and therefore their blood levels, and promoting intestinal peristalsis [44, 45]. In addition, it serves as a metabolic substrate for the intestinal microbiota, producing short-chain fatty acids that are absorbed in the large intestine and have been shown to modulate energy metabolism [46]. In the present study, the dietary fibre content in broccoli residues ranged from 12% for production surpluses to 16–22% in freeze-dried flours and air-dried flours at 60 °C, and 80 °C. The higher dietary fibre values of the flours could be explained by the fact that the agricultural residues correspond mainly to stalks, which contain a high amount of insoluble fibre (lignin) as well as a certain percentage of soluble fibre.

According to scientific literature, crude fibre values range 11–16% in broccoli stalks [43], dietary fibre 26–32% (DW) in by-products from broccoli leaves [47], and 38% in freeze-dried broccoli stems [24]. In all these studies, the analysis was performed in fractions obtained during broccoli plant cultivation and not from agricultural crop residues.

Broccoli flours showed dietary fibre concentrations similar to food products such as carrot, oat bran, apple or peas [48]. Therefore, these flours could be used as an ingredient to improve the nutritional profile of other foods. According to European legislation [49], the nutritional claim "source of fibre" requires an amount of 3 g of dietary fibre per 100 g product. In present case, this could be achieved with 13–14 g of these flours. Their incorporation in the foodstuffs could contribute to reaching the recommended daily intake of dietary fibre, which is between 25 and 35 g for adult people [50]. Current study is in agreement with the advantages highlighted by other authors that used broccoli by-products for product formulation. In this sense, broccoli flours from leaves and stalks have been used for the elaboration of cereal products such as gluten-free breads [10], sponge cakes or pasta [51, 52], increasing the content of proteins, minerals, and AA of the finished products [11]. In those studies, broccoli material was dried by freeze-drying, a technique of complex implementation in small and medium local companies. For present research, broccoli material was obtained from crops and production surpluses. The drying process was carried out with the conditions and equipment typically used by the food industry for this purpose, in accordance with the study design of Rivas et al. [53]. In that study, broccoli by-products derived from cultivar Parthenon in the region of Extremadura (Spain), and they were dried in a forced air oven and submitted to different treatments to obtain dietary fibre concentrates, supporting the use of broccoli by-products as a good source of dietary fibre.

Total polyphenol content from production surpluses and broccoli flours

Total polyphenol content (TPC) is shown in Table 3, and it can be observed that the drying process had a significant effect. The AD-80 flour had the highest contents and the AD-60 and FD (stalk) flours the lowest. On the other hand, the contents were intermediate in the FD broccoli surpluses (whole plant). In the plant, most polyphenols are bound to the cell wall and lignin. Some authors have pointed out that the application of temperatures up to 100 °C to vegetable structures breaks these bonds, releasing the polyphenols, which may result in higher TPC [6]. Additionally, the higher values of TPC with AD may be due to the formation of oxidation products of the polyphenolic compounds or products resulting from the interaction between amino acids and sugars present (melanoidins). In both cases, polymers with complex chemical structures are formed, which provide a brown coloration and have a reducing power. Thus, they can react with the Folin–Ciocalteau reagent, which would justify the higher TPC values observed in samples dried at 60 and 80 °C with respect to FD, as has also been described in the literature [13]. A limitation of the Folin–Ciocalteau method is that all functional groups with reducing power are quantified, both polyphenols and their reaction products, which may lead to an overestimation of the polyphenol content [54].

TPC values in broccoli production surplus samples (whole plant and its fractions) are shown in Table 4. Inflorescences represented 61% of the total biomass (in dry weight) and significant differences were observed in the

Table 3 Total polyphenol content (TPC), and antioxidant activity of flours from crop surpluses (flour 1) and field residues (flour 2) stabilised by freeze-drying (FD) and air-drying at 60 °C (AD-60) or 80 °C (AD-80)

Parameter	Flour 1 (broccoli whole plant)	Flour 2 (broccoli stalk)		
	FD	FD	AD-60	AD-80
TPC (mg GA/ g DW)	6.30 ± 0.79^{b}	3.01 ± 0.70^{c}	3.78 ± 0.18^{c}	8.37 ± 0.69^{a}
Antioxidant activity				
DPPH inhibition (%)	47.0 ± 4.03^{b}	29.0 ± 7.96^{c}	22.62 ± 0.62^{c}	66.75 ± 4.56^{a}
DPPH (mmol trolox/g DW)	16.25 ± 0.67^{b}	10.25 ± 2.60^{c}	7.86 ± 0.16^{c}	21.69 ± 1.40^{a}

Data expressed as mean ± SD ($n = 9$). Different superscript letters in the same row indicate statistically significant differences according to the HSD Tukey test at the 95% confidence level ($\alpha = 0.05$)

DW dry weight, *TPC* total polyphenol content, *GA* gallic acid equivalents

polyphenolic content of the whole plant (around 6 mg GA/g DW) with respect to stalks and stems (around 4 mg/g DW). Similar values were described by Thomas et al. [14]. A similar distribution has been reported by other authors [39, 55–57], although with quantitative differences. These differences in content may be due to factors such as the cultivar studied, plant growth conditions, ripening stage, and post-harvest processing [58, 59]. In all cases, TPC values were higher than those observed in the flours.

Antioxidant activity of crop surpluses and broccoli flours

The antioxidant activity (AA) of the different broccoli raw materials is shown in Table 3. Drying had a significant impact on this parameter, being higher in flours dried at 80 °C, followed by FD and finally air-drying at 60 °C. The increase in AA parallel to the increase in temperature has also been observed in citrus by-products (peels) [60]. The high values obtained at 80 °C could be related to the antioxidant capacity of the polymers formed as a result of the heat treatment [6], a hypothesis that needs to be confirmed in future studies. It has been shown that this type of polymers, such as the melanoidins formed in coffee roasting, have the ability to capture peroxyl radicals and chelate metals, which is greater with the higher molecular weight of the compound [61]. Pastoriza and Rufián-Henares [62] suggest that the formation of polymers derived from the interaction between polyphenols and sugars possess greater AA than those derived from the interaction between sugars and proteins (melanoproteins). These compounds were positively correlated with the level of browning of foods such as chocolate, beer, bread, and bakery products in general and could contribute significantly to the antioxidant capacity of the diet.

In the case of broccoli surpluses (whole broccoli and its fractions) (Tables 3 and 4), their AA was higher than that of the flours and the highest values corresponded to inflorescences and stems, followed by stalks and whole broccoli. This high inhibitory value against the DPPH radical of the inflorescences can be explained by their higher content of polyphenols, that are well-known scavengers of this free radical [63].

Glucosinolates from crop surpluses and broccoli flours

Glucosinolates are glycosylated organosulfur compounds and present a side chain of variable structure that allows them to be classified as aliphatic, aromatic, or heterocyclic (mainly indole) [64]. In the studied flours, four glucosinolates were identified, one belonging to the aliphatic glucosinolate family: glucoraphanin (4-methylsulfinylbutylglucosinolate), and three indole glucosinolates: glucobrassicin (3-indolylmethyl glucosinolate), 4-methoxyglucobrassicin (4-methoxy-3-indolylmethyl glucosinolate), and neoglucobrassicin (N-methoxy-3-indolylmethyl glucosinolate) (Table 5, Fig. 2). Of these, glucoraphanin was the most abundant glucosinolate, with values of 4.92 mg/g DW in freeze-dried broccoli, somewhat lower than those collected by other authors (9.7 mg/g DW) [8] and similar to those observed in inflorescences (3.52 mg/g DW) [59].

Technological and culinary treatments (blanching, cooking in water, and microwaving) affect the glucosinolate content of brassicas, due to leaching phenomena in the aqueous medium, thermal degradation, or enzymatic hydrolysis [65]. Heat treatment can exert positive effects, causing denaturation and inactivation of the myrosinase enzyme and thus preventing enzymatic hydrolysis of glucosinolates; however, elevated temperatures are harmful as they produce thermal degradation of these compounds [8, 66]. In the present study, considering the drying process applied, stabilisation of broccoli flours did not significantly affect the total amount of glucosinolates, but their particular composition. A relevant reduction was detected on indole glucosinolate contents due to the effect of drying at 80 °C. Other studies have also shown that indole glucosinolates, such as glucobrassicin, are more thermosensitive than aliphatic glucosinolates [17, 18, 67]. Glucoraphanin (aliphatic GLS) was the major compound present in

Table 4 Glucosinolate content (mg/g DW) in flours from crop surpluses (F1) and field residues (F2) stabilised by freeze-drying (FD) and air-drying at 60 °C (AD-60) or 80 °C (AD-80)

Parameter	Flour 1 (broccoli whole plant)	Flour 2 (broccoli stalk)		
	FD	FD	AD-60	AD-80
Glucoraphanin	4.92 ± 0.28^{a}	3.21 ± 0.22^{bc}	2.66 ± 0.07^{c}	3.73 ± 0.36^{b}
Glucobrassicin	3.66 ± 0.58^{a}	0.16 ± 0.03^{b}	0.47 ± 0.25^{b}	nd
Methoxyglucobrassicin	0.51 ± 0.20^{a}	0.24 ± 0.03^{b}	0.06 ± 0.01^{c}	nd
Neoglucobrassicin	4.37 ± 0.49^{a}	0.17 ± 0.00^{b}	nd	nd
TOTAL	13.27 ± 1.06^{a}	3.78 ± 0.17^{b}	3.04 ± 0.37^{b}	3.73 ± 0.36^{b}

Data expressed as mean ± SD ($n = 9$). Different superscript letters in the same row indicate statistically significant differences according to the HSD Tukey test at the 95% confidence level ($\alpha = 0.05$)

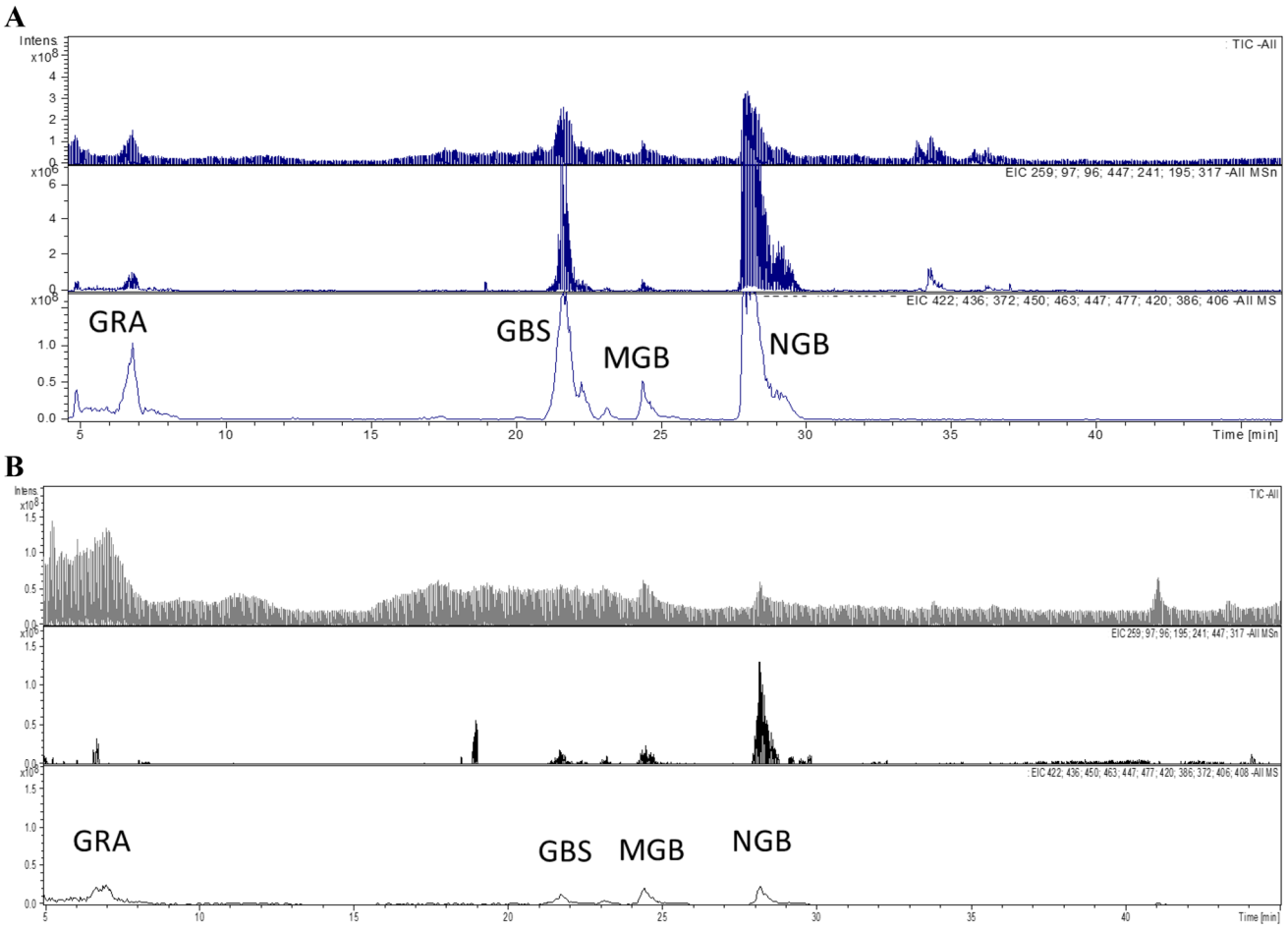

Fig. 2 Chromatographic profile of **A** freeze-drying broccoli surplus (whole plant); **B** freeze-drying broccoli field residue (stalks) using HPLC-MS^n ESI detection, revealing the presence of glucosinolates. *GRA* glucoraphanin, *GBS* glucobrassicin, *MGB* methoxyglucobrassicin, *NGB* neoglucobrassicin

the flours, being the only one detected in samples treated at 80 °C. Some authors have reported that indole glucosinolates degrade at 130 °C, reducing their contents to 92% after 80 min [68]. Processes such as blanching and subsequent drying with hot air result in losses of more than 50% [22]. Other authors have described that the amounts of GLS are maintained up to 60 °C, while at higher temperatures they decrease, especially indole compounds [69]. The observed differences may be dependent on the *Brassica* species studied, the processing temperature and time, or the conditions of the medium (presence of water, pH, Fe^{2+} ions) [70]. On the other hand, FD was the process with lowest impact on GLS content of the flours; however, it is a process with a high energy cost and is not easily accessible and implemented in the food industry. Other emerging technologies such as ultrasound-assisted heat drying, or microwave drying are equally interesting for preserving phytochemical content [8] but are not widely implemented in the agri-food field. In the present study, a thermal drying of agricultural residues on a semi-industrial scale has been carried out to evaluate a real alternative for the use of this material, mimicking the working conditions in industry.

Regarding the total GLS content of broccoli surpluses, they presented concentrations four times higher than broccoli field residues flours. This difference in concentration was expected, since their different distribution in the plant: stalks have a lower amount of glucosinolates than inflorescences [71] and, as mentioned above, the flours consisted mostly of stalks while the surpluses were composed mainly of inflorescences. Therefore, the interest in the revalorization of broccoli production surpluses lies in proposing them as ingredients rich in bioactive principles (glucosinolates). An intake of 120 mg GLS/day has been associated with anti-inflammatory effects in overweight people [72]; such a dose would be reached by incorporating 31 g of these surpluses as an ingredient in a food product.

Conclusions

Taking into account the findings of current study, flours from agricultural residues (broccoli stalks) are an interesting source of dietary fibre. On the other hand, flours from broccoli production surpluses provide relevant amounts of bioactive compounds due to their glucosinolate content. Thus, the results obtained from the identification, analysis, and characterisation of the surpluses of broccoli crops and field residues enable the proposal of the integral and mixed dosage and utilization of these materials to produce a novel stable ingredient, rich in dietary fibre and bioactive compounds, for diverse applications of the agri-food industry, in accordance with the fundamental principles of the circular economy.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest. The authors have no financial or proprietary interests in any material discussed in this article.

Compliance with ethics requirements This article does not contain any studies with human or animal subjects.

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