

Article

Bio-Products Obtained from Broccoli and Cabbage Wastes Are Proposed as Functional Food Ingredients and Bioherbicides for Sustainable Weed Management

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Abstract: Developing processes that contribute to the valorization of vegetable wastes is of great importance since these residues are characterized by being of high quality, having a huge potential for valorization. However, upcycling vegetables residues and defining specific applications for the value-added products obtained might be a challenge, and they should be tackled by means of different and complementary innovations. In the present study, broccoli and white cabbage discards were transformed into powdered products by means of selected techniques and conditions, which have been explored for applications in the agri-food sector. The obtained brassica powders were rich in bioactive compounds such as phenolics and isothiocyanates. Their antioxidant properties in response to in vitro digestion were evaluated to assess the potential of the products as functional food ingredients. On the other hand, brassica powders were tested as bioherbicides. For that purpose, inhibition tests on weed germination and growth of weeds from agricultural soil seedbank and selected species (*Lolium rigidum*, *Papaver rhoeas*, *Portulaca oleracea*, and *Echinochloa crus-galli*) were performed under controlled greenhouse conditions. In vitro simulated digestion studies demonstrated that bioactive constituents of powders were progressively released during digestion, and consequently, a part of them could be finally absorbed and, thus, provide their beneficial effect. Brassica bioproducts significantly reduced the germination of weeds from the agricultural soil seedbank, and the selected weed species tested, namely *L. rigidum*, *P. rhoeas*, *P. oleracea*, and *E. crus-galli*. Powders also showed a negative effect on the root weight and length of dicotyledonous weeds from the soil seedbank and *L. rigidum*, whereas a stimulatory effect was observed on the spring–summer species, *E. crus-galli* and *P. oleracea*. The results of this work contribute to extending the range of applications for brassica industrialization wastes.

Keywords: vegetable waste; upcycling; sustainable diets; brassicaceae; glucosinolates; organic mulching; weed control; circular economy



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1. Introduction

Reducing food loss and waste and the valorization of agri-food residues are key objectives that respond to Food 2030 priorities, which are as follows: nutrition and health,

climate and sustainability, circularity and resource efficiency, and innovation and communities [1]. Primary production and processing contribute to almost half of the losses generated along the whole food chain, with fruit and vegetables as the food materials that contribute the most to these percentages [2].

Fruits and vegetables lead the ranking of food wastes and losses (40–50% of their production) [3], thus resulting in the use of non-renewable resources to obtain food products that will not be consumed [4]. In the first stages of processing, vegetables and fruit losses mainly consist of discards due to high standards of commercialization and edible parts removed by peeling or cutting. Besides, in developing countries fruit and vegetable excess production also offers unique opportunities for adding value to these wastes or losses. Valorization of such wastes to produce innovative bio-ingredients can open great market opportunities for the food industry and related areas [4]. Hence, to ensure circularity and sustainability of food systems, the food industry must develop approaches that allow for the valorization and reintroduction of these residues in the productive cycle to generate social and economic value. Encouraging a culture of sustainability in the development of industrial processes is one of today's great challenges [5].

Growing worldwide population and recent dietary patterns have driven an increase in the consumption of fruits and vegetables [6]. In addition, changes in daily habits and lifestyle have boosted the appearance in the market of convenient foods, such as IV-range or freshly prepared products, which significantly contribute to food losses or residues in the production stages. Wastes generated in fourth-range production are characterized by their particularly high quality [7]; for this reason, developing processes with the aim of valorizing their constituents are of great importance. Fruit and vegetable by-products are rich and varied in high-value elements, such as bioactive compounds and dietary fiber. These compounds can be classified as primary metabolites (vitamins, minerals) or secondary metabolites, such as phenolics, carotenoids, saponins, and glucosinolates, among others [6]. Many plant metabolites that accumulate in fruits and vegetables are produced as a response to stress or cellular damage, being part of their defense system against pathogens.

Bioactive constituents present in plant-derived wastes are interesting from a nutritional point of view, since they exhibit properties that are beneficial to human health (antioxidant, antiproliferative, antimicrobial, among others) [8,9]. Particularly, glucosinolates (GLs) and isothiocyanates (ITCs) present in Brassicaceae have been proposed as antimicrobial, antioxidant, and anticarcinogenic agents [10,11]. Therefore, the valorization of brassica discards to produce functional ingredients to fortify food products is an approach that may effectively contribute to the concept of more sustainable and healthier diets, as proposed by the FAO [12]. Plant-derived wastes have been proposed as the source of compounds and ingredients for food fortification and food preservation applications [13]. Nonetheless, phytochemicals present in vegetable wastes can also be proposed for other applications beyond the food industry, such as the cosmetic and pharmaceutical manufacturing [6,13], or in agriculture as crop protection products, as some of them could have a potential use in pest management and control [14–16]. The literature evidences an increased interest in GLs and ITCs as bioactive constituents and natural compounds to be used in pest management to avoid or minimize the use of synthetic pesticides [17–19]. Synthetic pesticides are generally highly toxic and persistent, and their residues may contaminate crops and food products and pollute soils and groundwater or negatively affect non-target organisms like pollinators; however, phytochemical biopesticides are less toxic, minimally persistent, environmentally friendly, and safe for humans and non-target organisms. Crop protection plays an essential role in ensuring agricultural productivity to supply enough food to feed the world's growing population [20], but concerns about health, quality, and food

safety, as well as sustainability and environmental issues, have led to an increasing rejection of synthetic pesticides. Therefore, upcycling vegetable wastes as biopesticides not only would contribute to the sustainability of agricultural processes, but also to the whole food production system [14].

Weeds are considered one of the biggest biotic constraints in accomplishing potential yield [21]. These species are better adapted to proliferate in a wide range of soils and climatic conditions. In addition, they are highly competitive for water, nutrients, and energy, depriving crops of these resources. According to Kubiak et al. (2022) [22], 1800 weed species have been reported to produce up to 31.5% of yield losses globally, which is equivalent to USD 32 billion of economic losses every year. Synthetic herbicides have become the most widely used method to control weeds since the discovery of the synthetic auxins after the Second World War [23,24] due to their high efficacy and low cost compared with other weed control methods. They have achieved increased rates of weed control and have reduced farm labor, thereby decreasing both labor demands and the carbon dioxide emissions linked to these activities [23]. However, the overuse of synthetic herbicides has put a very high selective pressure on weeds, promoting the development of weed-resistant biotypes [25]. Consumers' and governments' growing concerns on healthy food, environmental protection, and sustainability have driven the search for new weed control tools, which could replace, or at least complement, chemical ones, following the premises of Integrated Pest Management (IPM), which promote the use of all available pest control techniques rather than the use of one [26] and whose practices are regulated in Europe through the European Union (EU) Directive 2009/128/EC. New strategies such as "From Farm to Fork" in the context of the European Green Deal, which aimed to reduce by 50% the use and risk of chemical pesticides by 2030, demonstrate the increasing interest of the EU in promoting a more resilient agriculture and less dependence on synthetic pesticides in order to preserve biodiversity and ensure food safety and sustainability [27]. Therefore, the discovery of new natural compounds and products that could improve crop production and act as crop protection products is a need for the development of sustainable agriculture and healthy ecosystems.

GLs are secondary metabolites exclusive to plants in the Brassicaceae family [28]. These are present in the whole plant, but their concentration varies depending on the species, variety, environmental factors, age, and type of tissue [29]. In the plant tissue, GLs hydrolyze into ITCs due to the action of myrosinase, an enzyme that is compartmented in the tissue and released when it is disrupted [10,30]. Mechanical disruption releases myrosinase and promotes GLs hydrolysis to ITCs, whereas drying treatments may have a variable impact on ITCs content depending on the technique used, the temperature reached during the process, and the time of exposure [31]. Nevertheless, vegetable residues are highly perishable, and dehydration is a mandatory step for integrally valorizing these wastes. Therefore, producing GLs or ITCs-rich ingredients requires a good knowledge of the effect of processing techniques and conditions on the products' quality, as explored in Bas-Bellver et al. [32]

The development of a circular-economy system requires developing innovative solutions that reintroduce residues into the agri-food chain. However, there is no single innovation that can solve the problem; instead, it must be addressed through different and complementary innovations [33]. In this work, powdered bioproducts obtained from brassica wastes are proposed for two different applications in the agri-food industries: as functional food ingredients and as bioherbicides. These powders were selected based on the results of previous research [32], as they were the more promising for the applications being tested. As functional food ingredients, physicochemical and technological properties acquire relevance, but it is also a key aspect to assess the availability of the beneficial com-

pounds to be absorbed after digestion. In the study, the response of the selected brassica waste bioproducts to simulated in vitro digestion was evaluated as part of their assessment as functional food ingredients. On the other hand, to determine the herbicidal potential of the brassica waste powders, greenhouse trials were carried out applying selected powders as mulching or incorporating them into the soil and the substrate used.

2. Materials and Methods

2.1. Plant Materials and Powders Obtention

White cabbage (*Brassica oleracea* var. *capitata*) wastes consisted of the outer leaves discarded in fresh cabbage manufacturing lines, whereas broccoli (*Brassica oleracea* var. *italica*) wastes consisted of IV-range broccoli residues (mainly stems). Powdered brassica waste products were manufactured by means of a process that combined tissue disruption, drying, and final milling to produce fine powders. Among the different conditions assayed in previous research [32], grinding and freeze-drying (FD) and chopping and hot-air drying (HAD) at 70 °C were chosen for the present study. Brassica powders chopped and HAD at 70 °C were chosen for their better antioxidant properties, including higher phenol and flavonoid contents, as well as for the lower investment and production costs required by this dehydration technique. As for FD powders, they showed a slightly higher sulforaphane content and smaller particle size, which confers them better technological (hydration and emulsifying) properties, parameters that may be relevant for formulation and nutrients release during digestion.

Therefore, freeze-dried broccoli (B_FD) and white cabbage (WC_FD) waste powders were obtained by first grinding fresh wastes to pieces ≤ 5 mm in diameter in a food processor (Thermomix® TM6, Vorwerk, Madrid, Spain) (10,000 rpm, 10 s) and then freeze-drying them in trays containing about 200 g of ground residue. Before being introduced in the freeze-drier, the trays containing the samples were deep-frozen at -40 °C for 24 h (CVN-40/105 freezer, Matek, Barcelona, Spain) and then freeze-dried in a pilot plant equipment (LyoQuest-55 freeze drier, Telstar, Terrasa, Spain) at 0.1 mbar and -45 °C (condenser temperature) for 48 h. As for the hot air-dried broccoli (B_HAD) and white cabbage (WC_HAD) waste powders, fresh tissues were chopped to pieces ≤ 10 mm in diameter (Thermomix® TM6, Vorwerk, Madrid, Spain) (5000 rpm for 5 s), which were spread in 10 mm thick layers on plastic perforated trays (~ 200 g of residue/tray). The trays were then put in a convective transverse flow pilot plant drier with perforated trays (CLW 750 TOP+, Pol-Eko-Aparatura SPJ, Katowice, Poland) using air at 70 °C and with an air velocity of 2 m/s. The drying processes were set to decrease the water activity (a_w) of the samples to 0.3, thus guaranteeing the stability of the dried products, which took 12 h for cabbage wastes and 8 h for broccoli residues. Once dehydrated, the dried wastes were milled (10,000 rpm, 2 min at 30 s intervals) to obtain fine powders (Thermomix® TM6, Vorwerk, Madrid, Spain). The powdered residues obtained were packed into glass containers with an aluminum lid in a light-free environment until use. Figure 1 summarizes the stages and conditions applied to obtain the powdered products from white cabbage and broccoli wastes.

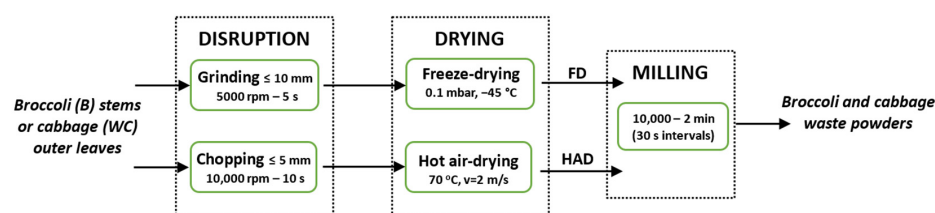


Figure 1. Process diagram with stages and conditions applied to produce the white cabbage and broccoli waste powders for the present study. FD: Freeze-drying. HAD: Hot air-drying.

The yield of transformation of wastes into powders was calculated from the relationship between the mass of residue after (M_f) and before drying (M_0) (Equation (1)).

$$Y(\%) = 100 M_f/M_0 \quad (1)$$

2.2. Physicochemical Characterization of Powders

Cabbage and broccoli waste powders were characterized so that water activity (a_w), moisture content (x_w), and antioxidant properties were analyzed. Water activity was obtained with a dew point hygrometer at 25 °C (Aqualab 4TE, Decagon devices Inc., Pullman, Washington, DC, USA). Moisture content (x_w) was calculated from the weight loss before and after drying in a vacuum oven (Vaciotem-T vacuum oven, JP Selecta, Barcelona, Spain) at 60 °C and $P = 10$ mmHg as described in the official method AOAC 934.06 [34].

As for antioxidant properties, total phenols and total flavonoids were determined by the Folin–Ciocalteu [35] and the aluminum chloride [36] methods, respectively. Regarding total phenols, 0.125 mL of the extract was added to 0.5 mL of bidistilled water and 0.125 mL of Folin–Ciocalteu reagent (Scharlab S.L., Barcelona, Spain) in a cuvette. After 6 min in darkness, 1.25 mL of a 7% w/v sodium carbonate solution and 1 mL of bidistilled water were added. The absorbance was measured at 760 nm after completing 90 min in darkness. Gallic acid was used as standard and results given in mg of Gallic Acid Equivalents (GAE) per g of dry matter. Total flavonoids were obtained by reacting 1.5 mL of the extract and 1.5 mL of a 2% (w/v) aluminum chloride solution, and keeping the mixture in darkness for 10 min. Absorbance measurements at 368 nm were obtained to express total flavonoids as mg of Quercetin Equivalents (QE) per g of dry matter. The antioxidant activity was determined by the DPPH (2,2-diphenyl-1-picryl hydrazyl) and ABTS (2,2′-azino-bis-(3-ethylbenzothiazoline-6-sulphonic acid) radical methods. The DPPH method [37] consisted of mixing 0.1 mL of the extract with 2.9 mL of a 0.1 mM solution of DPPH in methanol (Merck KGaA and affiliates, Darmstadt, Germany). Absorbance was measured at 575 nm after 60 min in darkness. ABTS determination [38] consisted of mixing 0.1 mL of the extract with 2.9 mL of an ABTS+ (VWR International LLC, Radnor, PA, USA) solution in phosphate buffer (0.70 ± 0.02 absorbance at 734 nm). The absorbance of the mixture at 734 nm after 7 min of reaction was obtained and transformed into mg of Trolox Equivalents (TE). Both DPPH and ABTS antioxidant activities were given as mg of Trolox Equivalents (TE) per g of dry matter. All spectrophotometric measurements were made in a Helios Zeta UV/Vis spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA).

2.3. Simulated In Vitro Digestion Experiments for Functional Ingredients Assessment

Simulated in vitro digestion was carried out to evaluate the impact of digestion on the release of total phenol content, total flavonoid content, and antioxidant activity (DPPH and ABTS methods) from selected brassica powders, along simulated digestion.

The in vitro simulation of gastrointestinal digestion (oral, gastric, and intestinal phases) was performed following the standardized INFOGEST static method proposed by Minekus et al. [39], for which Simulated Salivary Fluid (SSF), Simulated Gastric Fluid (SGF), and Simulated Intestinal Fluid (SIF) were prepared. Oral phase simulation consisted in mixing samples with SSF in a 1:1 ratio (w/v) and vortexing (Reax top, Heidolph Instruments GmbH & Co. KG, Schwabach, Germany) for 2 min at 37 °C. Gastric phase simulation consisted of mixing the oral bolus with SGF (1:1 v/v) and stirring for 2 h at 55 rpm (Intell-Mixer RM-2, Elmi Ltd., Riga, Latvia) at 37 °C (JP Selecta SA, Barcelona, Spain). Simulating the intestinal phase consisted of mixing the chyme with SIF (1:1 v/v) and stirring for 2 h at 55 rpm and 37 °C.

Samples were separated for subsequent analysis of antioxidant properties (total phenols, total flavonoids, and DPPH and ABTS antioxidant capacities) at the end of the gastric and intestinal phases. Antioxidant attributes were analyzed in the soluble and insoluble fractions of the digested samples, obtained after centrifugation at 10,000 rpm for 5 min (Eppendorf® Centrifuge 5804R, Hamburg, Germany). Bioaccessibility (BI) and recovery indexes (RIs) of antioxidant attributes were obtained with Equations (2) and (3), respectively. BI is defined as the compounds that remain solubilized in the chyme after the intestinal phase with respect to initial content, in percentage, and refers to the proportion of bioactive compounds that could become available for absorption by the intestinal cells; whereas RI is defined as the percentage of compounds present in the total fraction of the digested sample after the gastric or the intestinal phase of digestion, with respect to the initial ones [40].

$$BI(\%) = \frac{A}{B} \cdot 100 \quad (2)$$

$$RI(\%) = \frac{C}{B} \cdot 100 \quad (3)$$

where A is the amount (μg) of compound in the soluble fraction, after the intestinal phase; B the amount (μg) of compound in the powder before digestion; and C the amount (μg) of compound in the total digest after the corresponding digestion phase.

In the solid fractions, i.e., the non-digested powder or digested precipitate, antioxidant attributes were measured on extracts obtained from 1 g of sample extracted with 10 mL of an 80% (*v/v*) methanol/water solution by applying the methods described above. The extraction procedure was performed in dark conditions for 1 h (horizontal stirrer, Magna Equipments S. L., model ANC10, Barcelona, Spain) [41]. After extraction, the mixture was centrifuged at 10,000 rpm for 5 min (Eppendorf SE, model 5804/5804R, Hamburg, Germany). In the supernatants, antioxidant properties were directly measured with no solvent addition. If required, extracts were diluted with an 80% (*v/v*) methanol/water solvent in a ratio of 1:10 (*v/v*). Bidistilled water was used as the blank.

2.4. Herbicidal Potential of Brassica Waste Powders Tests Under Greenhouse Conditions

2.4.1. Pre-Emergence Herbicidal Trials Against the Weed Seedbank of a Soil Untreated with Herbicides

Powdered bioproducts obtained from brassica wastes were tested in pre-emergence herbicidal trials against the weed seedbank of a soil collected from a citrus orchard untreated with herbicides and located in Puzol, Valencia province, Spain ($39^{\circ}37'10.1''$ N, $0^{\circ}17'25.6''$ W). The soil samples were homogenized and placed in plastic trays of $17 \times 24 \times 4$ cm, adding a total of 1.5 kg of soil per tray. For this application, HAD was selected. This decision was based on the fact that HAD is the drying technique most commonly used in the agri-food sector as it needs lower investments and production costs than FD, and it is also easier to apply and scale-up. Moreover, previous results have demonstrated that the concentration of bioactive constituents depends more significantly on the residue type (cabbage vs. broccoli) than on the drying technique used (HAD vs. FD) [32].

To assess the herbicidal potential of the brassica powders obtained, two different types of applications were implemented (Figure S1): (1) layering the powder onto the surface like a mulching and (2) incorporation of the product into the first two centimeters of soil. Five treatments were tested: control (trays without adding any brassica powder), broccoli mulching (BrM), broccoli incorporated (BrI), white cabbage mulching (WCM), and white cabbage incorporated (WCI). All brassica powder treatments were applied at a dose of 1 kg m^{-2} of soil because it provided good coverage of the soil surface. For each treatment, 3 trays were prepared. Immediately after treatment application, 150 mL of water was

added to stimulate seed germination and to increase the moisture of the waste powder to promote the release of bioactive constituents to the soil. Watering was held every 48 h with a volume of 150 mL per tray manually. The experiment was maintained for 9 weeks. The number of emerged seedlings in trays was recorded weekly. In the last week, all emerged plants were extracted and separated into monocotyledonous and dicotyledonous, and their fresh and dry weights were determined. The dry weight was obtained after drying the plants using a Binder Model ED 23 oven at 60 °C for two weeks. The length of the aerial and root parts of the plants emerging was also measured. For this purpose, images of the whole plants were taken with a Lumix Model No. DC-FZ82 digital camera by Panasonic, which were later processed with Digimizer v.4.6.1 (MedCalc Software, Ostend, Belgium, 2005–2016) software.

2.4.2. Pre-Emergence Herbicidal Trials of Brassica Waste Powders Against Selected Weed Species

The weed species chosen were *Lolium rigidum* Gaudin and *Papaver rhoeas* L. as autumn/winter species, and *Portulaca oleracea* L. and *Echinochloa crus-galli* (L.) Beauv as spring/summer species because of their importance and competitiveness in many crops. Seeds were collected from plants growing wild in crop fields in different locations: *E. crus-galli* seeds were collected in Sollana (Valencia province, Spain) in 2017 and *P. oleracea* seeds were collected in Puzol (Valencia province, Spain) in 2021. *Papaver rhoeas* seeds were purchased from the company Cantueso Natural Seeds (Córdoba, Spain) and *L. rigidum* seeds were purchased from the company Herbiseed (Reading, United Kingdom) in 2019. The treatments applied were the same as described previously for the trials against the weeds from the soil seedbank. For each treatment, 10 square polypropylene pots of 8 × 8 × 7 cm were used. Pots were filled with 200 g of a mixture of ¾ peat and ¼ perlite, previously homogenized and hydrated. In each pot, 5 seeds were placed for *L. rigidum* and *E. crus-galli*, and 10 seeds for *P. rhoeas* and *P. oleracea*. The difference in the number of seeds sown between species was based on previous studies due to the larger size of *L. rigidum* and *E. crus-galli*. For mulching treatments (M), seeds were placed into the substrate before applying the powder on top, whereas in incorporated treatments (I), the powder was first mixed into the first 2 cm of substrate and the seeds were sown afterwards. For 6 weeks, watering was held every 2 days during the winter months and every 3 days during the summer months, adding 50 mL water on top per pot each time to promote weed germination. During the last two weeks of the pre-emergence trial at summertime, 200 mL of water was added at the bottom of the pots to increase water availability. Weed emergence was recorded weekly, and at the end of the experiment, the plants grown in each pot for every treatment were extracted to determine the fresh and dry weights and length of aerial and root parts, in the same way as it is described in the previous section for pre-emergence trials against the weeds present in the soil seedbank. The aerial length of *P. oleracea* and *E. crus-galli* was registered weekly by recording images of all pots using a Lumix Model No. DC-FZ82 digital camera by Panasonic, which were later processed with Digimizer v.4.6.1 (MedCalc Software, Ostend, Belgium, 2005–2016) software.

2.4.3. Starting–End Dates and Climatic Conditions of the Greenhouse Experiments

Experiments were carried out in greenhouse number 8 of Universitat Politècnica de València (UPV) located in Valencia (Spain). The climatic conditions registered during the experiments are reported in Table S1. These data were collected using a HOBO U23 Pro v2 External Temperature Data Logger (Onset Computer Corporation, Bourne, MA, USA).

2.5. Statistical Analysis

Statistical analyses were performed with Statgraphics Centurion XVIII software (Stat-Point Technologies, Inc., Warrenton, VA, USA). Analyses of variance (one-way and multi-factor ANOVAs) were applied after checking data normality. Differences among means were evaluated by using Fisher's Least Significant Difference (LSD) test at a confidence level of 95% ($p < 0.05$).

3. Results and Discussion

3.1. Physicochemical Attributes of Selected Broccoli and White Cabbage Waste Powders

In Table 1, physicochemical attributes of the powders obtained by HAD at 70 °C and FD are presented. The water activity and moisture content values correspond to the powders obtained for the present study and are the average of three different batches. Drying allowed us to reduce the a_w and moisture content of residues to values that significantly reduced their susceptibility to spoilage ($a_w < 0.3$), in contrast to fresh residues, which exhibited a_w values in the range of 0.98–0.99. The moisture content of fresh residues was about 91–93% ($g_{\text{water}}/100 \text{ g}$), whereas dried powders were in the range of 2.5–3.3%. This reduction in the a_w and moisture content values reveals that the process proposed allows us to lengthen the shelf life of residues, transforming them into bioproducts with a more convenient form, ready for storage, transportation, and further use. The average yield of the drying stage was $8.1 \pm 0.1\%$, so that 10 kg of fresh residue gave rise to 810 g of powder, on average.

Table 1. Physicochemical characteristics of broccoli (B) and white cabbage (WC) waste powder ingredients (FD, ground and freeze-dried; HAD, chopped and hot-air-dried at 70 °C). Water activity (a_w), moisture content (% $g_{\text{water}}/100 \text{ g}$), equivalent volume diameter of particles (D[3,4]), total phenols (mg GAE/ g_{dm}), and sulforaphane content ($\mu\text{g}/g_{\text{dm}}$). Particle size * and sulforaphane * content from Bas-Bellver et al. (2022) [32].

Sample	a_w	Moisture Content (%, $g_{\text{water}}/100 \text{ g}$)	Particle Size * D[3,4]	Total Phenols (mgGAE/ g_{dm})	Sulforaphane * ($\mu\text{g}/g_{\text{dm}}$)
WC HAD	0.23 ± 0.02^a	2.5 ± 0.7^a	294 ± 20^a	4.86 ± 0.04^c	73 ± 3^a
WC FD	0.29 ± 0.02^b	3.3 ± 0.2^b	137 ± 3^b	3.42 ± 0.10^a	77 ± 4^a
B HAD	0.29 ± 0.03^a	3.0 ± 0.6^a	445 ± 38^a	5.91 ± 0.15^d	461 ± 9^a
B FD	0.28 ± 0.02^a	2.6 ± 0.2^b	145 ± 4^b	4.52 ± 0.13^b	506 ± 1^b

Different superscript letters in the same column indicate statistically significant differences at the 95% confidence level (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

Values provided for particle size and sulforaphane content in Table 1 correspond to previous own data [32] and are given as considered relevant for the results of the present study. As observed, FD gave rise to powders of smaller particle sizes for both white cabbage and broccoli waste powders. Particle size has an impact on water and oil interaction properties, since larger surface areas facilitate interaction with other compounds. Particle size may also influence the release of bioactive constituents during digestion, since a larger surface area implies a higher exposure to digestive enzymes and other digestion conditions such as pH. Broccoli powders had a higher sulforaphane content compared to white cabbage powders. This difference should be considered when evaluating the nutritional properties of the powders, and it is particularly important when assessing their herbicidal potential. Differences between FD and HAD powders were not as significant as differences attributed to the type of residue.

3.2. Response of Brassica Waste Powders to Simulated In Vitro Digestion

The impact of simulated in vitro digestion on the antioxidant properties of brassica powders was assessed as detailed in the materials and methods section. Antioxidant properties of selected powders, before and after each in vitro digestion phase, in the supernatant (S) and the precipitate (P), are presented in Table 2. The characterization of powders before digestion confirmed that broccoli waste powders exhibited better antioxidant properties than white cabbage ones, and that dehydration conditions had a statistically significant effect on antioxidant properties, being higher for HAD products than for FD ones. It has already been discussed in the literature that HAD may cause biochemical reactions leading to new compounds' formation, such as those of Maillard reactions, the inhibition of pro-oxidant enzymes, and other transformations, which may improve antioxidant properties [32,42,43].

Simulated in vitro digestion showed a positive effect on antioxidant properties, which means that antioxidant compounds were released along digestion. This trend has also been observed in other studies, in which an increase in antioxidant compounds after digestion was attributed to improved extractability and release of matrix-bound antioxidant compounds during digestion, as well as to structural transformations of polyphenol compounds after the intestinal phase [44–47].

Total phenols and flavonoids presented similar values after the gastric and intestinal phases, with slight positive or negative differences. These results are in line with those reported by Bouayed et al. [48], who found that the release of total phenolics and total flavonoids mainly occurred during the gastric phase. Therefore, the potential increase in these compounds after duodenal digestion may be attributed to the extended extraction time and the action of intestinal digestive enzymes, which help release matrix-bound compounds. As opposed to the previous result, Gunathilake et al. [49] reported a decrease in total phenols and flavonoids in leafy vegetables after the intestinal phase, as compared to the gastric one. A change from the acidic gastric conditions to the alkaline intestinal ones due to pancreatic and bile acids might justify the partial degradation of phenols and flavonoids [48,49].

Antioxidant activity after the intestinal phase increased significantly compared to the gastric one, for both DPPH and ABTS antioxidant assays. It is reported in the literature that radical scavenging activity is strongly pH-dependent and might be significantly increased at higher pH values. This phenomenon is attributed to the deprotonation of the hydroxyl moieties present on the aromatic rings of polyphenolic constituents [49–51]. Thus, the small intestine conditions, which imply an alkaline pH and pancreatin action, could promote the solubilization of certain polyphenolic constituents that were previously bound to macromolecules or present in a reduced form [52].

In this study, the soluble compounds that could be easily absorbed were separated from the non-soluble ones by centrifugation, so that the former were collected as a supernatant and the latter as a precipitate. Antioxidants collected in the precipitate were significantly higher than in the supernatant, in both the gastric and the intestinal phases, thus suggesting that most of them remained retained in the structural matrix (Table 2). Nevertheless, the pellets or precipitate also retain a certain amount of liquid phase such that the solubilized compounds in this liquid phase are more accessible than the bound ones [47,53]. The antioxidant components present in the precipitate after the intestinal phase increased in all cases. In the supernatant, the concentration of antioxidant compounds increased in FD powders but decreased in HAD powders, suggesting an increased or reduced solubilization of these compounds, respectively. The increase in FD powders could be explained by the smaller particle size of FD materials, which implies a larger contact surface between the sample and the intestinal fluid, which facilitates extraction and solubilization

along digestion. The rate of antioxidants released from fibrous particles to the surrounding intestinal fluid is inversely related to particle size. In addition, other extractable compounds such as sugars and soluble fiber may exert a protective role on polyphenols during the digestion process, as they establish interactions with certain polyphenol compounds. In contrast, other components such as polysaccharides or insoluble fiber may be responsible for phenol losses during digestion [53]. On the other hand, antioxidant compounds that are solubilized and released to the liquid phase are more available to be absorbed; nevertheless, the compounds retained in the pellets could also be slowly released and further metabolized in the large intestine by the action of colonic microorganisms [45,54,55].

Recovery indexes (RIs) after the gastric and intestinal phases are presented in Table 2. The RI provides information on the release of compounds during digestion with respect to their corresponding initial content. The values obtained for RI were remarkably high in all cases and were generally higher for white cabbage powders than for broccoli ones. However, no clear trend could be identified for the dehydration method used. In most cases, the RI obtained after the intestinal phase was higher than that after the gastric one, indicating the progressive extraction of antioxidant components due to in vitro digestion, rather than a degradation due to the exposure of antioxidant components to digestion conditions.

The bioaccessibility index (BI) for the whole digestion process was also calculated. This parameter provides information on the amount of compound released to the soluble fraction, with respect to the amount present before digestion. BIs are plotted in Figure 2 for the four digested powders. The high values obtained for this parameter suggest that a great proportion of antioxidant compounds become available for absorption into the systemic circulation during digestion. As observed with RI, the values are higher for white cabbage waste powders than for broccoli waste ones. Also, the dehydration technology used does not show a clear impact, as deduced from the results.

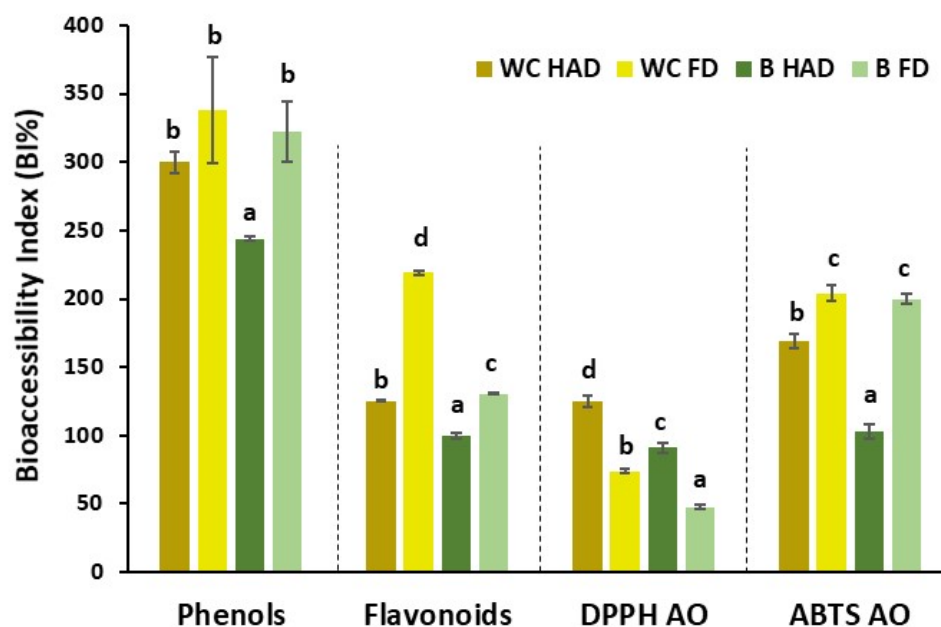


Figure 2. Bioaccessibility index (BI) calculated on total phenols, total flavonoids, DPPH, and ABTS antioxidant activities after the in vitro digestion process. White cabbage (WC) and broccoli (B) waste; HAD: dried with hot air at 70 °C, FD: freeze-dried. Mean $\pm$ standard deviation of three measurements. Different letters above the bars indicate statistically significant differences at the 95% confidence level (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

Table 2. Antioxidant properties along simulated in vitro digestion: total phenols, total flavonoids, and antioxidant capacity by the DPPH and ABTS methods for white cabbage (WC) and broccoli (B) waste powders (BD: before digestion; GP: after the gastric phase; IP: after the intestinal phase; P: precipitate; S: supernatant). Values given as per gram of non-digested sample. %RI: recovery index in percentage. HAD: dried with hot air at 70 °C, FD: freeze-dried. Mean $\pm$ standard deviation of three measurements.

	Sample	BD	Gastric PHASE (GP)				Intestinal PHASE (IP)			
			S	P	Total	%RI	S	P	Total	%RI
Total phenol content (mg GAE/g)	WC_HAD	4.71 $\pm$ 0.04 ^c	9.65 $\pm$ 0.19 ^b	14.9 $\pm$ 0.3 ^b	24.58 $\pm$ 0.18 ^b	521 $\pm$ 4 ^d	4.25 $\pm$ 0.11 ^a	21.7 $\pm$ 0.5 ^d	25.9 $\pm$ 0.5 ^d	550 $\pm$ 10 ^d
	WC_FD	3.32 $\pm$ 0.10 ^a	5.4 $\pm$ 0.3 ^a	9.1 $\pm$ 0.3 ^a	14.5 $\pm$ 0.6 ^a	437 $\pm$ 17 ^b	6.9 $\pm$ 0.8 ^c	8.93 $\pm$ 0.12 ^a	15.8 $\pm$ 0.9 ^a	476 $\pm$ 26 ^c
	B_HAD	5.73 $\pm$ 0.15 ^d	12.10 $\pm$ 0.08 ^c	15.2 $\pm$ 0.9 ^b	27.3 $\pm$ 0.9 ^c	476 $\pm$ 16 ^c	5.48 $\pm$ 0.04 ^b	17.5 $\pm$ 0.2 ^c	22.99 $\pm$ 0.16 ^c	401 $\pm$ 2 ^a
	B_FD	4.38 $\pm$ 0.13 ^b	5.8 $\pm$ 0.2 ^a	8.7 $\pm$ 0.3 ^a	14.5 $\pm$ 0.4 ^a	330 $\pm$ 8 ^a	6.1 $\pm$ 0.4 ^{bc}	13.3 $\pm$ 0.7 ^b	19.4 $\pm$ 0.3 ^b	444 $\pm$ 8 ^b
Total flavonoid content (mg EQ/g)	WC_HAD	5.43 $\pm$ 0.06 ^b	4.036 $\pm$ 0.011 ^c	8.62 $\pm$ 0.08 ^c	12.65 $\pm$ 0.10 ^c	232.9 $\pm$ 1.8 ^b	2.042 $\pm$ 0.005 ^b	13.02 $\pm$ 0.12 ^c	15.06 $\pm$ 0.12 ^d	277 $\pm$ 2 ^d
	WC_FD	2.29 $\pm$ 0.05 ^a	2.87 $\pm$ 0.05 ^b	3.68 $\pm$ 0.05 ^b	6.55 $\pm$ 0.02 ^b	285.7 $\pm$ 0.9 ^c	3.06 $\pm$ 0.03 ^d	3.12 $\pm$ 0.04 ^a	6.18 $\pm$ 0.03 ^b	269.6 $\pm$ 1.3 ^c
	B_HAD	7.48 $\pm$ 0.07 ^c	4.210 $\pm$ 0.004 ^d	10.90 $\pm$ 0.09 ^d	15.11 $\pm$ 0.09 ^d	202.01 $\pm$ 1.17 ^a	2.930 $\pm$ 0.007 ^c	10.45 $\pm$ 0.16 ^b	13.38 $\pm$ 0.15 ^c	179 $\pm$ 2 ^a
	B_FD	2.33 $\pm$ 0.09 ^a	1.665 $\pm$ 0.006 ^a	3.05 $\pm$ 0.07 ^a	4.71 $\pm$ 0.07 ^a	202 $\pm$ 3 ^a	1.319 $\pm$ 0.006 ^a	3.16 $\pm$ 0.04 ^a	4.48 $\pm$ 0.04 ^a	192.3 $\pm$ 1.8 ^b
DPPH (mg TE/g)	WC_HAD	6.7 $\pm$ 1.2 ^a	3.87 $\pm$ 0.07 ^c	32 $\pm$ 2 ^c	36 $\pm$ 2 ^b	541 $\pm$ 37 ^b	2.50 $\pm$ 0.09 ^b	62.7 $\pm$ 0.4 ^d	64.2 $\pm$ 1.7 ^d	961 $\pm$ 2.5 ^b
	WC_FD	5.9 $\pm$ 0.3 ^a	1.61 $\pm$ 0.04 ^b	27.4 $\pm$ 0.6 ^{ab}	29.04 $\pm$ 0.65 ^a	489 $\pm$ 11 ^b	2.70 $\pm$ 0.08 ^c	27.9 $\pm$ 0.3 ^a	30.6 $\pm$ 0.4 ^a	515 $\pm$ 6 ^a
	B_HAD	9.3 $\pm$ 1.4 ^b	4.77 $\pm$ 0.06 ^d	31 $\pm$ 2 ^{bc}	36 $\pm$ 2 ^b	385 $\pm$ 24 ^a	3.4 $\pm$ 0.2 ^d	47 $\pm$ 2 ^c	50 $\pm$ 2 ^c	538 $\pm$ 20 ^a
	B_FD	7.1 $\pm$ 1.4 ^a	0.86 $\pm$ 0.16 ^a	25 $\pm$ 3 ^s	25 $\pm$ 3 ^a	359 $\pm$ 47 ^a	1.47 $\pm$ 0.04 ^a	36.1 $\pm$ 0.5 ^b	37.5 $\pm$ 0.4 ^b	528 $\pm$ 6 ^a
ABTS (mg TE/g)	WC_HAD	56 $\pm$ 4 ^c	49.1 $\pm$ 1.8 ^c	329 $\pm$ 33 ^b	378 $\pm$ 34 ^b	673 $\pm$ 61 ^b	28.4 $\pm$ 0.8 ^a	555 $\pm$ 39 ^b	584 $\pm$ 39 ^c	1040 $\pm$ 69 ^c
	WC_FD	36 $\pm$ 2 ^a	29.6 $\pm$ 1.6 ^b	244 $\pm$ 6 ^a	273 $\pm$ 8 ^a	768 $\pm$ 22 ^c	44.6 $\pm$ 1.3 ^c	295 $\pm$ 5 ^a	340 $\pm$ 6 ^a	954 $\pm$ 16 ^b
	B_HAD	94.7 $\pm$ 1.0 ^d	51.2 $\pm$ 1.8 ^c	414 $\pm$ 32 ^c	465 $\pm$ 33 ^c	491 $\pm$ 35 ^a	38 $\pm$ 2 ^b	550 $\pm$ 40 ^b	588 $\pm$ 42 ^c	621 $\pm$ 44 ^a
	B_FD	45.9 $\pm$ 1.8 ^b	20.8 $\pm$ 0.8 ^a	270 $\pm$ 6 ^a	291 $\pm$ 7 ^a	635 $\pm$ 15 ^b	39.7 $\pm$ 0.8 ^b	474 $\pm$ 4 ^b	512 $\pm$ 4 ^b	1116 $\pm$ 9

Different superscript letters in the same column and for the same property indicate statistically significant differences at the 95% confidence level (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

3.3. Herbicidal Potential of White Cabbage and Broccoli Waste Powders Against the Seed Weeds Present in the Soil Seedbank

In this section, the results of pre-emergence herbicidal trials of brassica waste powders against the weed seedbank of a soil untreated with herbicides are presented. The results evidence significant differences in the number of emerged plants in the trays throughout the trial between the control and treatments with brassica waste powders (Figures S2 and 3). However, according to the one-factor ANOVA performed on weekly results, there were no significant differences among the treatments, neither for the type of powder nor for the application method.

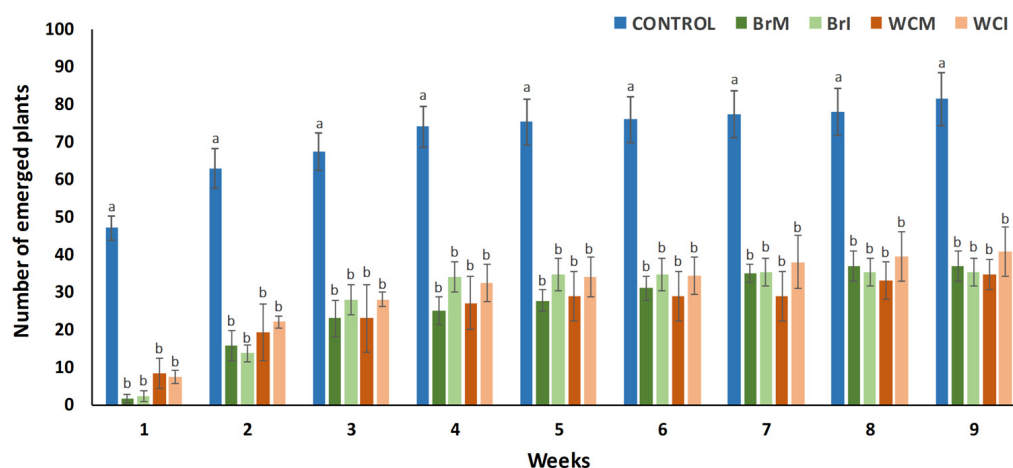


Figure 3. Evolution of the number of plants that emerged weekly in the trays with soil control and treated with broccoli mulching (BrM), broccoli incorporated (BrI), white cabbage mulching (WCM), and white cabbage incorporated (WCI). Different letters in the bars for each week indicate significant differences (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

Emergence in the control trays occurred earlier compared to the trays treated with the brassica waste powders, showing a strong inhibitory effect of the bioproducts applied on the onset of emergence. This inhibitory effect was somewhat stronger for the broccoli powders than for white cabbage powders in the first two weeks. To confirm this trend, a multifactor ANOVA analysis considering the type of residue and application as factors, and the week as a covariable, revealed that broccoli had a stronger inhibitory effect that was statistically significant up to the 5th week of the trial (p -value = 0.0221). This result is coherent with the sulforaphane content of the powders applied, since broccoli waste powders contained higher amounts than cabbage waste powders (Table S1). Sulforaphane is an ITC that results from glucoraphanin hydrolysis by the action of the enzyme myrosinase. While GSLs have limited biological activity, ITCs are the most phytotoxic among glucosinolates hydrolysis products [56], for which reason they are the main factor responsible for the bioherbicidal activity of brassicas. In subsequent weeks, emergence increased for all controls and trays treated with brassica powders, but the differences between control and brassica waste powder treatments remained significant until the end of the trial. Kunz et al. [57] found similar results regarding the germination of spontaneous weeds with brassica-based cover crops such as mustard (*Sinapsis alba*) and radish (*Raphanus sativus* var. niger). On the other hand, Bangarwa et al. [58] used freeze-dried brassica plant samples to control weeds in established crops. Although setting a cover crop has proven to be beneficial for soil quality and local biodiversity, maintaining a living plant cover carries a series of costs and its effectiveness is affected by climatic conditions, plant cycle, and the occurrence of pests [57].

With regard to the application method used, brassica species have been explored to control weeds by various methods such as cover cropping, crop rotations, water extract

application, mulching, intercropping, and crop residue incorporation [59]. Crop residues used as mulch have been shown to suppress weeds via allelopathy, but mulching has also been claimed to reduce weed germination and growth by light exclusion, acting as a physical barrier, and reducing available moisture in the top layer [60]. In the present assay, the mulching application of brassica powders, either broccoli or cabbage, was generally the most successful treatment, although differences in incorporation, considering the data of each week, were not significant, and the main differences were found against the control (Figure 3). Again, the multi-factor ANOVA, considering the 9 weeks of treatments with the type of residue and application factors, and the week as a covariable, revealed that mulching significantly reduced germination as compared to incorporation (p -value = 0.0322), mainly in cabbage waste powders.

Although there were some differences between mulching and incorporation, the slight differences obtained between treatments as compared to the differences with control samples could suggest that brassica powders might be exerting their effect mostly by the liberation of allelopathic compounds. However, given that differences between broccoli and white cabbage powders were less significant than the expected, considering their different sulforaphane contents, it could be postulated that, in addition to isothiocyanates, other bioactive constituents of powders could be exerting some herbicidal activity. According to the literature, simple phenols and polyphenols such as flavonoids are among secondary metabolites identified as allelochemicals [60,61]. All of these are common bioactive constituents of vegetable waste powders and have also been identified in the brassica powders being assayed (Tables 1 and 2).

The fresh and dry weights and length of aerial and radical parts of plantlets that emerged at the end of the trial are shown in Table 3 for monocotyledonous and dicotyledonous weeds. Broccoli waste powders slightly reduced fresh and dry weights in monocots, whereas white cabbage waste powders increased the fresh and dry weights as well as the length. Nevertheless, the effect of brassica waste powder on monocots was not significant compared to the treatment control. In dicotyledonous weeds, fresh and dry weights and the length of aerial parts were not affected by brassica waste powders. In contrast, differences were found in radical parts, so that root development was negatively affected when adding brassica powders. Specifically, significant differences were found in root fresh weight for the plantlets that emerged in the BrM, BrI, and WCI treatments, and a reduced root dry weight was found for plantlets from soil treated with BrM and WCI. Kunz et al. [57] used aqueous extracts of radish and mustard for in vitro tests with *Chenopodium album*, *Matricaria chamomilla*, *Stellaria media*, and *Veronica persica*, all dicotyledonous species. The extracts were able to reduce germination by more than 60% and radicle length by 50%.

Table 3. Effects of brassica waste powders (broccoli mulching (BrM), broccoli incorporated (BrI), white cabbage mulching (WCM), and white cabbage incorporated (WCI) on monocotyledonous and dicotyledonous weeds. Fresh and dry weights and the length of aerial and root parts at the end of the trial.

<i>Monocotyledonous</i>		Aerial Parts			Root Parts	
Treatment	Fresh Weight (g)	Dry Weight (g)	Length (cm)	Fresh Weight (g)	Dry Weight (g)	Length (cm)
control	6.30 ± 0.73 ^{ab}	0.82 ± 0.26 ^{ab}	18.72 ± 2.64 ^a	1.15 ± 0.32 ^{ab}	0.35 ± 0.08 ^{ab}	8.98 ± 1.22 ^a
BrM	3.75 ± 0.21 ^a	0.56 ± 0.05 ^a	20.11 ± 0.17 ^a	0.60 ± 0.06 ^a	0.33 ± 0.17 ^a	8.17 ± 0.64 ^a
BrI	4.19 ± 1.27 ^a	0.64 ± 0.17 ^a	21.62 ± 0.99 ^a	0.95 ± 0.59 ^{ab}	0.27 ± 0.11 ^a	9.58 ± 1.57 ^a
WCM	6.33 ± 3.07 ^{ab}	0.94 ± 0.34 ^{ab}	19.58 ± 1.03 ^a	0.90 ± 0.53 ^{ab}	0.39 ± 0.20 ^{ab}	8.34 ± 0.34 ^a
WCI	9.90 ± 1.50 ^b	1.37 ± 0.22 ^b	22.17 ± 1.53 ^a	2.25 ± 0.47 ^b	1.10 ± 0.35 ^b	10.24 ± 0.96 ^a

Table 3. Cont.

<i>Dicotyledonous</i>		Aerial Parts			Root Parts		
Treatment	Fresh Weight (g)	Dry Weight (g)	Length (cm)	Fresh Weight (g)	Dry Weight (g)	Length (cm)	
control	5.45 ± 2.19 ^a	0.56 ± 0.18 ^{ab}	7.38 ± 0.86 ^a	1.22 ± 0.42 ^a	0.37 ± 0.21 ^a	4.69 ± 1.09 ^a	
BrM	2.02 ± 0.73 ^a	0.24 ± 0.07 ^a	5.90 ± 1.08 ^a	0.31 ± 0.12 ^b	0.07 ± 0.02 ^b	7.65 ± 2.27 ^a	
BrI	2.16 ± 0.28 ^a	0.25 ± 0.01 ^a	5.03 ± 0.75 ^a	0.43 ± 0.11 ^b	0.09 ± 0.01 ^{ab}	6.50 ± 1.22 ^a	
WCM	3.73 ± 1.23 ^a	0.76 ± 0.15 ^b	6.07 ± 1.70 ^a	0.56 ± 0.25 ^{ab}	0.09 ± 0.02 ^{ab}	4.17 ± 1.04 ^a	
WCI	1.82 ± 0.35 ^a	0.24 ± 0.07 ^a	4.70 ± 0.77 ^a	0.26 ± 0.09 ^b	0.06 ± 0.01 ^b	4.75 ± 0.24 ^a	

Different superscript letters in the same column and mono- or dicotyledonous indicate significant differences (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

3.3.1. Herbicidal Potential of White Cabbage and Broccoli Waste Powders Against Selected Weed Species

The germination of selected weed species (*L. rigidum*, *P. rhoeas*, *P. oleracea*, and *E. crus-galli*) in the presence of brassica waste powders (broccoli and white cabbage) applied by incorporation or mulching is shown in Figure 4. On the other hand, the effect of brassica waste powders on fresh and dry weights and the length of aerial and root parts at the end of the trial are presented in Table 4.

Table 4. Effects of brassica waste powders (broccoli mulching (BrM), broccoli incorporated (BrI), white cabbage mulching (WCM), and white cabbage incorporated (WCI)) on *Lolium rigidum*, *Papaver rhoeas*, *Portulaca oleracea*, and *Echinochloa crus-galli* fresh and dry weights and the length of aerial and root parts at the end of the trial.

<i>Lolium rigidum</i>		Aerial Parts			Root Parts		
Treatment	Fresh Weight (g)	Dry Weight (g)	Length (cm)	Fresh Weight (g)	Dry Weight (g)	Length (cm)	
control	0.28 ± 0.04 ^a	0.027 ± 0.001 ^a	18.2 ± 0.7 ^a	0.10 ± 0.02 ^a	0.022 ± 0.003 ^a	15.3 ± 0.8 ^a	
BrM	0.32 ± 0.12 ^a	0.03 ± 0.01 ^a	18 ± 3 ^{abc}	0.03 ± 0.01 ^{bc}	0.007 ± 0.002 ^b	8.4 ± 1.5 ^{bc}	
BrI	0.13 ± 0.03 ^a	0.017 ± 0.001 ^a	17.8 ± 1.4 ^{abc}	0.008 ± 0.001 ^c	0.003 ± 0.001 ^b	5.8 ± 0.6 ^{bc}	
WCM	0.70 ± 0.10 ^b	0.06 ± 0.01 ^b	21.5 ± 1.0 ^b	0.07 ± 0.01 ^{ab}	0.011 ± 0.002 ^b	8.4 ± 0.7 ^b	
WCI	0.19 ± 0.06 ^a	0.02 ± 0.01 ^a	14 ± 2 ^c	0.02 ± 0.01 ^{bc}	0.004 ± 0.001 ^b	5.7 ± 0.9 ^c	
<i>Papaver rhoeas</i>		Aerial Parts			Root Parts		
Treatment	Fresh Weight (g)	Dry Weight (g)	Length (cm)	Fresh Weight (g)	Dry Weight (g)	Length (cm)	
control	0.07 ± 0.01 ^a	0.006 ± 0.001 ^a	3.39 ± 0.32 ^a	0.005 ± 0.001 ^a	0.0008 ± 0.0001 ^a	3.7 ± 0.3 ^a	
BrM	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	
BrI	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	
WCM	0.0075 ± 0.0005 ^a	0.0006 ± 0.0001 ^a	1.05 ± 0.51 ^b	0.0018 ± 0.0003 ^a	0.0003 ± 0.0002 ^a	2.5 ± 0.9 ^a	
WCI	0.01 ^{*,a}	0.0009 ^{*,a}	1.13 ^{*,a,b}	0.004 ^{*,a}	0.0002 ^{*,a}	1.92 ^{*,a}	
<i>Portulaca oleracea</i>		Aerial Parts			Root Parts		
Treatment	Fresh Weight (g)	Dry Weight (g)	Length (cm)	Fresh Weight (g)	Dry Weight (g)	Length (cm)	
control	0.62 ± 0.07 ^a	0.11 ± 0.01 ^a	5.9 ± 0.4 ^a	0.06 ± 0.01 ^a	0.018 ± 0.002 ^a	7.8 ± 0.5 ^a	
BrM	4.2 ± 0.8 ^b	0.51 ± 0.09 ^b	10.4 ± 0.8 ^b	0.28 ± 0.05 ^{bc}	0.08 ± 0.01 ^c	13.1 ± 1.8 ^c	
BrI	7.1 ± 1.4 ^c	0.78 ± 0.16 ^c	11.8 ± 1.6 ^b	0.39 ± 0.08 ^c	0.08 ± 0.02 ^c	9.9 ± 1.5 ^{bc}	
WCM	3.4 ± 0.5 ^b	0.34 ± 0.05 ^b	10.8 ± 0.7 ^b	0.22 ± 0.04 ^b	0.04 ± 0.01 ^b	11.5 ± 1.1 ^{bc}	
WCI	4.3 ± 0.8 ^b	0.36 ± 0.07 ^b	12.5 ± 1.0 ^b	0.21 ± 0.04 ^b	0.05 ± 0.01 ^b	9.8 ± 1.1 ^b	
<i>Echinochloa crus-galli</i>		Aerial Parts			Root Parts		
Treatment	Fresh Weight (g)	Dry Weight (g)	Length (cm)	Fresh Weight (g)	Dry Weight (g)	Length (cm)	
control	2.0 ± 0.4 ^{ab}	0.32 ± 0.05 ^{ab}	43 ± 4 ^a	2.1 ± 0.4 ^a	0.21 ± 0.04 ^{ab}	19.51 ± 1.1 ^a	
BrM	5 ± 3 ^{bc}	0.6 ± 0.4 ^{abc}	40 ± 15 ^a	6 ± 3 ^a	0.3 ± 0.2 ^{ab}	145 ± 7 ^a	
BrI	0.0043 ± 0.002 ^a	0.0022 ± 0.0009 ^a	5.8 ± 1.9 ^b	0.0005 ± 0.0004 ^a	0.0002 ± 0.0002 ^a	0.75 ± 0.03 ^b	
WCM	10.94 ^{*,c}	1.26 ^{*,c}	73.32 ^{*,a}	15.89 ^{*,b}	1.43 ^{*,c}	26.46 ^{*,a}	
WCI	8 ± 2 ^c	0.8 ± 0.3 ^{bc}	67.2 ± 1.5 ^a	7 ± 4 ^{ab}	0.5 ± 0.2 ^b	23 ± 2 ^a	

* No standard error data available. Different letters in the same column indicate significant differences (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

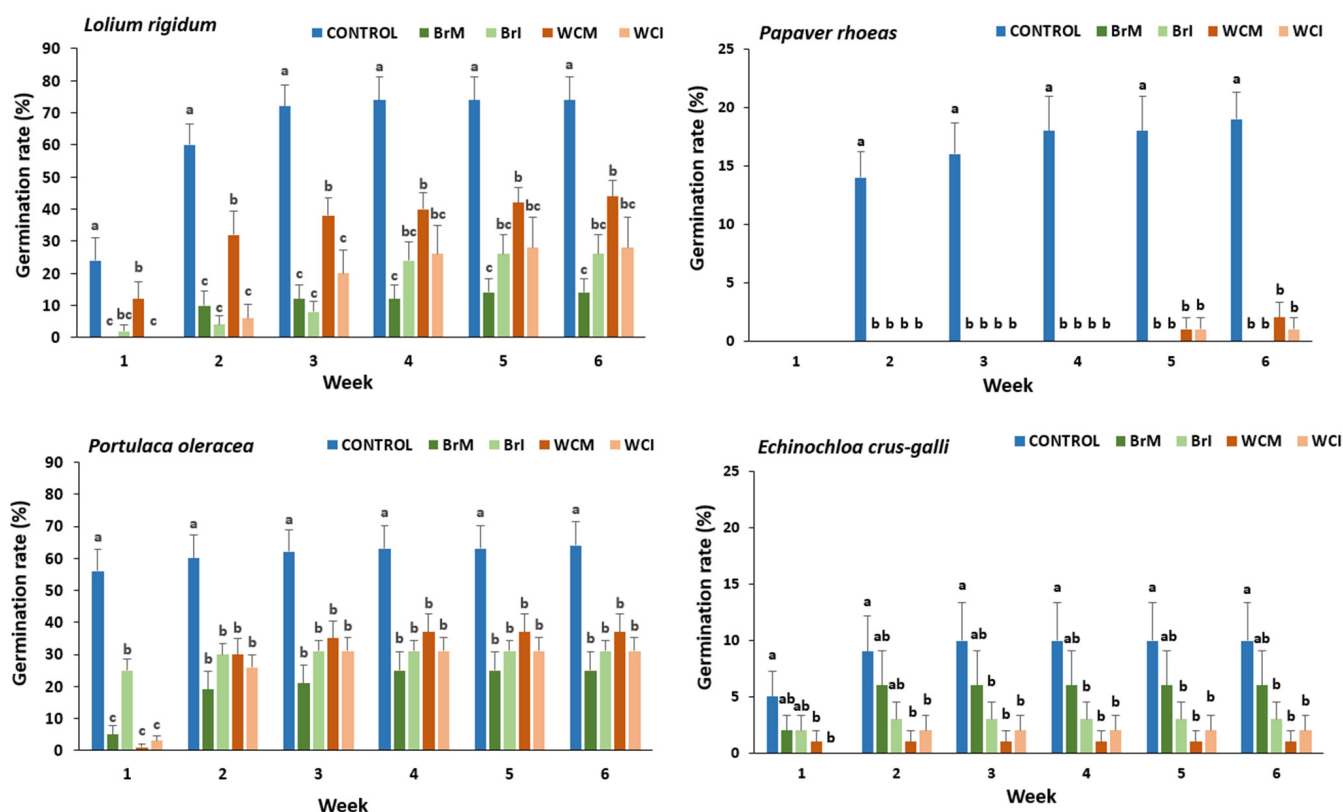


Figure 4. Evolution of weekly germination rate (%) of *Lolium rigidum*, *Papaver rhoeas*, *Portulaca oleracea*, and *Echinochloa crus-galli* treated with broccoli mulching (BrM), broccoli incorporated (BrI), white cabbage mulching (WCM), and white cabbage incorporated (WCI). Different letters in the bars for each week indicate significant differences (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

Lolium Rigidum

The germination of *L. rigidum* was significantly affected by the application of brassica waste powders (Figure 4). This reduction was greater with the BM treatment, followed by BI, WCI, and, lastly, WCM, in which the lowest inhibition was recorded. The BM, BrI, and WCI treatments delayed the onset of emergence more throughout the trial. Brassica waste powder did not affect aerial biomass accumulation (Table 4) but had a reducing effect on root biomass accumulation and growth. Broccoli waste powders did not affect the fresh and dry weights and length of *L. rigidum*'s aerial part. Only WCI treatment significantly affected the aerial length. To the contrary, the radical part of *L. rigidum* was affected by brassica waste powders, having obtained better results with BrI and WCI treatments. WCM treatment produced a stimulatory effect in the aerial part of *L. rigidum* but significantly reduced the root's dry weight and length. However, this was the treatment with the lowest inhibitory effect after the control. Therefore, cabbage seems to be more inhibiting through incorporation rather than mulching for this species. Brassica species showed a very strong inhibitory potential in *L. rigidum*'s root growth, which could be translated into reduced aerial growth. Asaduzzaman et al. [62] demonstrated the potential of high allelopathic canola (*Brassica napus*) in reducing *L. rigidum*'s root length. Seedlings that were sown nearby canola seeds grew shorter than those that were sown away from them and tried to escape from the vicinity of canola seeds, proving that not only canola was capable of reducing the plantlets' length but also altering their growth pattern.

Papaver Rhoeas

The germination of *P. rhoeas* was significantly reduced with white cabbage waste powders and completely inhibited with broccoli waste powders (Figure 4). It should be

noted that *P. rhoeas*'s germination is commonly low due to persistent seed dormancy [63]. Brassica waste powders delayed the onset of emergence up to 5 weeks in WCM and WCI compared to the control. There were no data available of BM's and BI's fresh and dry weights as there was no germination in these treatments. The aerial and root fresh and dry weights of *P. rhoeas* were not significantly affected by WCI and WCM treatments, but WCM treatment significantly reduced aerial length (Table 4).

Portulaca Oleracea

Germination in *P. oleracea* was significantly reduced, overall, by all treatments throughout the trial (Figure 4). The treatment with the final lowest germination rate was BrM. In the first week, differences between treatments could be observed, particularly with BrM, WCM, and WCI treatments, which were the most inhibitory. However, in the second week, emergence in these treatments started to increase rapidly, reaching similar germination rates with the *P. oleracea* seeds treated with all four brassica waste powder treatments at the end of the experiment.

Brassica waste powder had a stimulatory effect on growth and biomass accumulation in both the aerial and radical parts of *P. oleracea*. The fresh and dry weights and length of *P. oleracea* showed a significant increase in contact with brassica waste powder, with the control being significantly lower for both the aerial and root parts (Table 4). The most stimulatory treatment occurred with broccoli waste powder treatments, particularly in the root, given that the results were significantly larger as compared to white cabbage waste powders. Glucosinolates hydrolysis and the persistence of isothiocyanates in the soil might have been negatively affected by the climatologic conditions of these experiments. Glucosinolates molecules rapidly release isothiocyanates into the soil medium in the presence of water, but their persistence is commonly low [64]. The stability of these molecules in different environmental conditions has been studied, with the conclusion that soil moisture is essential for ensuring a proper hydrolysis of glucosinolates into isothiocyanates [58]. The herbicidal effect of brassica waste powder on *P. oleracea* was tested in the summer months when water evaporation in the soil was high. Considering that the product of both mulching and incorporation was on the surface of the substrate, the lack of a maintained moisture level during the trial could have affected the hydrolysis of glucosinolates and the persistence of the isothiocyanates in the soil. This would explain why an inhibitory effect was observed on germination but not on growth. Broccoli treatments (BrM and BrI) had a significant stimulatory effect when compared to white cabbage (WCM and WCI), particularly in the roots (Table 4).

The evolution of the aerial parts' growth of *P. oleracea* is shown in Figure 5, where it can be observed that the control plants exhibited greater growth than those treated with brassica waste powder during the first 4 weeks, being later surpassed by all the treated plants. Growth began to stabilize from the fifth week onwards, coinciding with an increase in the appearance of flowers. These results confirm the hypothesis that the effect of the brassica waste powder affected mainly the germination during the first two weeks of the experiment, which delayed the onset of emergence.

Echinochloa Crus-Galli

The germination of *E. crus-galli* was significantly affected by the presence of brassica waste powder, except for BrM treatment (Figure 4). The plantlets grown on the WCM treatment obtained the lowest final germination rate.

E. crus-galli was not affected negatively by the presence of brassica waste powders, except for BrI. In the plantlets treated with BrI, a toxic effect was observed, which resulted in a decrease in the fresh and dry weights and a significant reduction in length in the aerial

and radical parts (Table 4). As for the rest of the treatments, a stimulatory effect in the growth of the aerial parts was seen in *E. crus-galli*, especially for the WCI treatment. The fresh and dry weights of the radical parts and the length of both the aerial parts and roots were not affected by the presence of brassica waste powders.

Differences in the growth of *E. crus-galli* (Figure 5) could be observed after two weeks of the start of the trial. The growth of plantlets treated with BI was the highest in the first week, but this quickly decreased due to the toxicity observed. During the first three weeks, no differences were detected between plantlets from the different treatments. After that, the growth of plantlets treated with WCM started to speed up, reaching the highest growth at the end of the trial. Plantlets treated with BrM and WCI had a lower growth, but the second increased very rapidly after five weeks. In contrast to *P. oleracea*, no differences were observed between treatments during the first weeks; therefore, brassica waste powders did not influence the emergence of *E. crus-galli*.

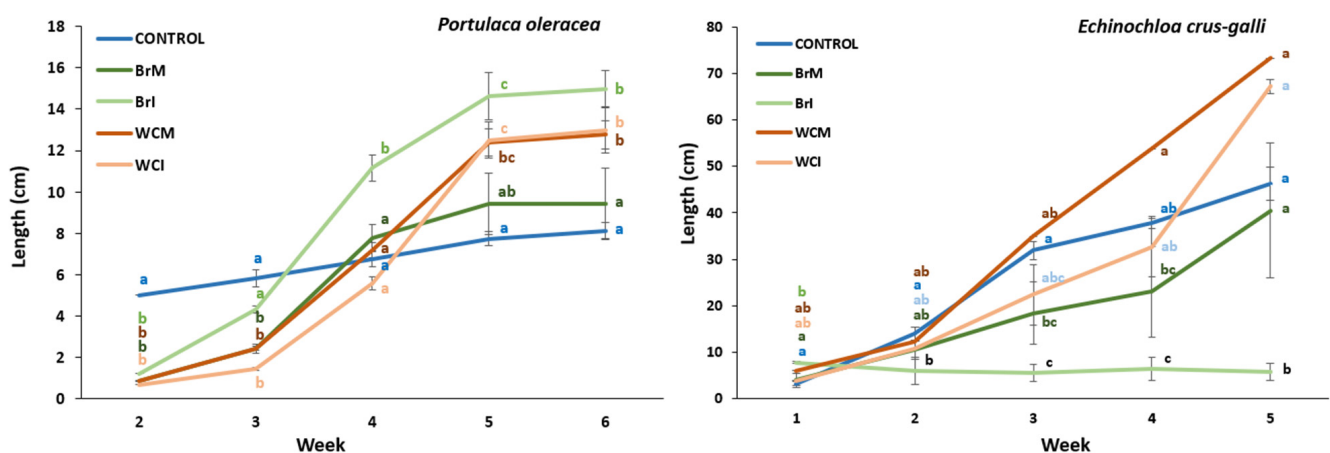


Figure 5. Growth of the aerial part of *Portulaca oleracea* (cm) and *Echinochloa crus-galli* (cm) treated with broccoli mulching (BrM), broccoli incorporated (BrI), white cabbage mulching (WCM), and white cabbage incorporated (WCI). Different letters in the lines for each week indicate significant differences (Fisher's Least Significant Difference (LSD) test, $p < 0.05$).

4. Conclusions

Developing processes that can contribute to the valorization of fourth-range production wastes is of great importance, since these residues are characterized by being of particular high quality, having a huge potential for valorization. However, upcycling vegetable residues and defining specific applications for the value-added products obtained might be a challenge, and they should be tackled by means of different and complementary innovations. In the present study, broccoli and white cabbage powders were transformed into powdered products using selected techniques and conditions, which allowed us to transform these perishable residues into stable bioproducts ready for transportation, storage, and further use.

The present contribution explores two different applications of the upcycled brassica bioproducts in the agri-food sector. On the one hand, powders obtained from brassica discards are presented as innovative bio-ingredients to produce more nutritious foods, thus addressing the concept of more sustainable and healthier diets. The composition of brassica waste powders includes bioactive compounds such as phenolics and isothiocyanates, which may exert a positive impact on human health. Moreover, the *in vitro* simulated digestion of these ingredients demonstrates that the bioactive constituents of brassica powdered ingredients are progressively released during digestion, and, thus, a part of them could be finally absorbed and provide their beneficial effect. Further efforts on this research area

should address the inclusion of these powders into different food matrices and products and assess their benefits and impact on quality and sensory attributes.

On the other hand, upcycled powders also show a potential to be used as a sustainable tool for weed control. Brassica bioproducts significantly reduced germination in weeds in the soil seedbank of a non-treated soil and species such as *L. rigidum*, *P. rhoeas*, *P. oleracea*, and *E. crus-galli*. Brassica waste powders also showed a negative effect on the root weight and length of dicotyledonous weeds from the soil seedbank and *L. rigidum*, whereas a stimulating effect was observed on the spring species, *E. crus-galli* and *P. oleracea*. This opposite result was related to the climatologic conditions during the *P. oleracea* experiments, particularly high temperatures, which may have negatively affected isothiocyanate release and persistence. Although the results obtained are promising, brassica powders did not totally control weeds on their own; nevertheless, they could be used combined with other weed control methods to reduce pesticide use as part of an integrated weed management strategy. Field experiments should be carried out to test the potential of brassica waste powders under real growing conditions.

The results of the present work are of especial relevance since they contribute to extending the range of applications of brassica industrialization wastes. Besides the food sector application, their use as bioherbicides is particularly relevant since, although crop residues have been used to control weeds in the past, the transformation of brassica wastes produced in the food industry to bioproducts for weeds control is an innovative approach and could contribute to a circular economy, which is one of the main priorities of the European Green Deal.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/su17010282/s1>, Figure S1. Application of brassica waste powders to untreated soil: mulching (upper trays) and incorporation to the first layer of soil (lower trays); Figure S2. Images of weed germination in non-treated agricultural soil after 6 weeks of treatment with broccoli and cabbage waste powders, in mulching and incorporated to the top layer (2 cm) of soil; Table S1. Greenhouse temperature and relative humidity conditions (Mean, maximum (Max) and minimum (Min)) during the experiments.

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