DEVELOPMENT OF NEW POWDERED SMOOTHIES AGGLOMERATED WITH POLYPHENOL EXTRACTS

Desarrollo de nuevos smoothies en polvo aglomerados con extractos polifenólicos

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ABSTRACT

Consuming fruits and vegetables through smoothies can contribute to improving current dietary imbalances in the population. Fruit and vegetables have high nutritional value, although they are not stable and can quickly lose their properties. Spray Drying can be used to stabilise these products although entails some loss of quality, involving antioxidants and other oxidizable compounds. By applying an appropriate addition technique, powdered smoothies can be enhanced with functional compounds, such as polyphenol antioxidants from plant extracts. This research explores the technological feasibility of developing fruit and vegetable-based powdered smoothies, enriched with a sustainable rosemary extract obtained from distillation by-products, which is rich in polyphenols (e.g., rosmarinic acid). The objective was to test a novel agglomeration method using a fluidised bed to obtain powdered smoothies enriched with rosemary polyphenols. Two formulations were assessed: a fruit-based (apple, banana, orange and whey protein) and a vegetable-based (apple, carrot, pumpkin and whey protein) to which aqueous rosemary extract (RE146) (145.6 mg polyphenols/g) was added at 800 mg/kg powder. The results showed that the agglomeration with RE has a little impact on the physical traits of powders (granulometry, colour, pH, sorption and solubility properties) and does not degrade rosemary polyphenols, maintaining the recovery level of rosmarinic acid around 100%. Addition of RE enhances the response of phenolic antioxidants 100% in these powdered smoothies, however, rosemary off-flavour is detected in the rehydrated smoothies, being needed reduce RE dose in preventing acceptability problems. The study concludes that agglomeration with RE represents a viable, innovative and sustainable technological strategy for the development of functional and value-added smoothies, contributing to reduced fruit and vegetable wastage and reaching their recommended daily intake.

Keywords: Powder agglomeration, fruit, vegetable, rosemary, antioxidants.

RESUMEN

El consumo de frutas y verduras a través de smoothies puede contribuir a mejorar los desequilibrios dietéticos actuales en la población. Las frutas y verduras tienen un alto valor nutricional, aunque no son estables y pueden perder rápidamente sus propiedades. El secado por pulverización puede utilizarse para estabilizar estos productos, aunque conlleva cierta pérdida de calidad, involucrando a antioxidantes y otros compuestos oxidables. Mediante la aplicación de una técnica de adición adecuada, los smoothies en polvo pueden ser mejorados con compuestos funcionales, como antioxidantes polifenólicos de extractos de plantas. Esta investigación explora la viabilidad tecnológica de desarrollar smoothies en polvo a base de frutas y verduras, enriquecidos con un extracto de romero sostenible obtenido a partir de subproductos de destilación, que es rico en polifenoles (p. ej., ácido rosmarínico). El objetivo fue probar un nuevo método de aglomeración en lecho fluidizado para obtener smoothies en polvo enriquecidos con polifenoles de romero. Se evaluaron dos formulaciones: una a base de fruta (manzana, plátano, naranja y proteína de suero) y una a base de vegetales (manzana, zanahoria, calabaza y proteína de suero) a las que se añadió extracto acuoso de romero (RE146) (145,6 mg polifenoles/g) a 800 mg/kg de polvo. Los resultados mostraron que la aglomeración con RE apenas afecta a las características físicas de los polvos (granulometría, color, pH, propiedades de sorción y solubilidad) y no degrada los polifenoles de romero, manteniendo el nivel de recuperación de ácido rosmarínico en torno al 100%. La adición de RE mejora la respuesta de los antioxidantes fenólicos en estos smoothies en polvo; sin embargo, se detecta un sabor a romero en los smoothies rehidratados, siendo necesario reducir la dosis de RE para prevenir problemas de aceptabilidad. El estudio concluye que la aglomeración con RE representa una estrategia tecnológica viable, innovadora y sostenible para el desarrollo de smoothies funcionales y de valor añadido, contribuyendo a reducir el desperdicio de frutas y verduras y alcanzar su ingesta diaria recomendada.

Palabras clave: Aglomeración de polvos, fruta, verdura, romero, antioxidantes.

INTRODUCTION

Fruits and vegetables are widely recognised as nutrient-dense foods, characterised by their high content of carbohydrates, dietary fibre, minerals, vitamins, and diverse bioactive compounds with potential health-promoting properties. Their intake, as long as it is in the recommended amounts (400 g/day), is associated with a multitude of health benefits, such as reducing the risk of cardiovascular and non-communicable diseases (cancer, obesity and diabetes); improving the immune system and mental health, and contributing to growth, development and life expectancy (FAO, 2020). Therefore, the World Health Organization (WHO) and the Food and Agriculture Organization of the United Nations (FAO) seek to raise population awareness about the importance of daily fruit and vegetable consumption, highlighting their nutritional benefits for health and disease prevention, while also promoting the reduction of food waste (FAO, 2021). An increasingly popular way to consume fruits and vegetables is through smoothies. A smoothie is a blended drink made from a combination of ingredients such as fruits, vegetables, yoghurt, milk, or ice cream. The ingredients are typically blended until smooth and creamy, creating a thick and refreshing beverage. Smoothies can be customised to suit various tastes and dietary needs and are often consumed as a quick and nutritious snack or meal replacement. Smoothies are an easy and convenient way to ensure the recommended intake of fruit and vegetables, considering consumers' current lifestyles and their interest driven by health and wellness trends. As a result, their demand is constantly growing and its market is becoming increasingly important for the food industry, especially in North America, although it is estimated to reach a global value of $17 billion by 2025 (Cano-Lamadrid et al., 2020).

There is no legal definition of "smoothie", the European Union (EU) legislation regulating these products falls under the same framework as that relating to fruit juices (Council Directive 2001/112/EC, relating to fruit juices and certain similar products intended for human consumption). Smoothie preparation is often based on the use of the whole fruit or vegetable, removing only the seeds and peel (Cano-Lamadrid et al., 2020). Fruits and vegetables are seasonal products that generate surpluses, and the development of these smoothies can contribute to the revalorisation of discarded fruit and vegetables, addressing the food waste we are currently facing (Jiménez-Monreal et al., 2025). Industrial smoothies are unstable products that require preservation treatments, such as pasteurisation, sterilisation or chilling. A lesser-used technology to date is Spray Drying (SD). SD involves smoothie dehydration by spraying with hot air, which prevents an excessive thermal damage to the fruit and vegetable components. This technology produces highly stable powdered products with good rehydration properties, although vitamin C and other fruit antioxidants and nutrients can be degraded under pro-oxidant conditions, resulting in some detriment of the powder's nutritional value. For this reason, incorporating ingredients such as rosemary extracts that are rich in other polyphenols, may improve the nutritional and functional properties of smoothies by increasing the intake of diverse phenolic compounds.

For centuries, rosemary products (*Salvia officinalis*, L.) have been used as medicinal and therapeutic herbs for their antioxidant, antimicrobial, antidiabetic, antimutagenic, hepatoprotective and antiangiogenic properties, among others (Nieto et al., 2018). These beneficial effects, attributed to the presence of bioactive compounds, have led to their increased use in recent years in other industries, such as pharmaceuticals and food. This antioxidant activity is mostly related to the presence of non-volatile phenolic compounds including phenolic acids (rosmarinic acid, caffeic acid), flavonoids (hesperidin 7-o-glucoside, hispidulin, diosmin, genkwanin) and terpenoids (carnosol, carnosic acid, epirosmanol, rosmanol, methylcarnosate, isorosmanol), while the major phenolic volatiles present in essential oil are eucalyptol, α-pinene and camphor (Jordán et al., 2013). Several studies have demonstrated their ability to scavenge free radicals (Karadağ et al., 2019), such as reactive oxygen species (ROS), and to induce metal chelation (Nieto et al., 2018). Given that the efficacy of natural antioxidants may be higher than that of synthetic antioxidants (Nieto et al., 2018), and in view of the consumer's opposition to using synthetic antioxidants in food, due to various drawbacks presented, such as the alteration of organoleptic properties or potential long-term health risks; rosemary presents a promising natural alternative for the food industry, as its incorporation can increase their functional and nutritional value, as well as their shelf life. In fact, several studies have shown that the addition of rosemary extract has been able to slow down the oxidation in different food products such as meat (Liu et al., 2009; Naveena et al., 2013; Teruel et al., 2015), bakery (Ou et al., 2018), candies (Cedeño-Pinos et al., 2020) and yoghurt-based sauces (Martínez-Tomé et al., 2022) where it can act as a bio-preservative and improve their quality parameters. These improvements are partly due to rosemary's distinctive sensory attributes (herbal, bitter, astringent, pungent and spicy), linked to the presence of phenolic compounds (Tannins), plant pigments (Chlorophylls), and essential oils (Eucalyptol and camphor) (Cedeño-Pinos et al., 2020).

Once harvested, rosemary is processed in the industry as follows: briefly, the crushed fresh leaves are steam-distilled to obtain essential oil. The distilled leaf by-product is then extracted with water or organic solvents to obtain the hydrophilic and lipophilic extracts. The extraction of bioactive compounds from rosemary distilled leaf is a critical step, and its effectiveness largely depends on the solvent used (Cedeño-Pinos, 2023). This can be performed by two main strategies: a whole extraction, using a water/acetone mixture, or a selective extraction, aimed at obtaining specific fractions. The fat-soluble fraction is obtained using acetone as solvent, while the water-soluble fraction is isolated by aqueous extraction. The extraction method influences the phytochemical profile and functional properties of the final extract. In our case, we selected water-soluble extracts to obtain non-volatile phenolic compounds. In any case, rosemary extract (E392) is a food additive authorised by Regulation (EC) No 1333/2008 of the European Parliament and of the Council of 16 December 2008 on food additives (EC Regulation No 1333/2008) and it is recognised by the FDA as safe (GRAS) for human consumption (21 CFR 182.2) (FDA, 1977). Although the regulation does not specify a maximum dose for this additive in powdered smoothies, the limit for similar products, such as dehydrated milk, is 200 mg/kg (Regulation (EC) No 1333/2008).

Powder agglomeration is a process that involves joining fine powder particles together to form larger particles, called agglomerates. This is achieved by applying mechanical, thermal, or chemical forces that allow the particles to adhere to each other. Agglomeration treatment can be conducted by fluidisation, mixing and compression. Each method has its own advantages and is selected based on the characteristics of the powder and the desired product. This technique is being used in the food industry to improve powder properties such as flowability, solubility, and stability, although it has not yet been used to incorporate small amounts of extracts into powdery food. Turning a food powder into an instant powder, easy to use and soluble, can sometimes be a significant technological challenge (Szulc & Lenart, 2013). To improve the dissolution or reconstitution properties of these fruit and vegetable powdered smoothies, we could apply the fluidised bed agglomeration technique, with the aim of achieving the adhesion of small particles to each other, forming a larger structure composed of conglomerates (Cardona, 2014), thus improving the flowability, dispersibility, and uniformity of the ingredients (Srivastava & Mishra, 2010). This technique has already been used in the food industry in a wide variety of products designed to dissolve quickly in liquids, such as dairy powders (milk or cocoa powder), beverages (coffee or tea), and starch-based foods (soups, sauces or baby products) (Szulc & Lenart, 2013). However, this process also opens up the possibility of producing powdered products with improved organoleptic characteristics and greater preservative and functional capacity, thanks to the addition of rosemary extracts (RE) during the instantaneisation process. When a fluidisation system is used, fruit and vegetable powdered smoothies are introduced into the feed zone, where the particles are kept suspended by a gas (air) circulating from below, creating a state called a fluidised bed. These moving particles come into contact with a binding agent (water with rosemary extract), which adheres to the surface of the particles. Thanks to the turbulent environment, the particles collide with each other and agglomerate, forming small clumps or agglomerates, resulting in a more soluble powder (Cardona, 2014).

Agglomeration results and some properties of interest such as particle size, porosity, flowability, solubility, stability, shape and density will depend on the type of agglomeration and the operating conditions during the process (Dacanal & Menegalli, 2009): feeding, spraying liquid concentration, atomisation pressure and droplet size, working temperature and air convection. Working temperature is divided into three main ones: inlet air temperature, product temperature and outlet air temperature. Inlet air temperature is considered a key parameter for the evaporation of the agglomeration liquid and powder drying product. Furthermore, product temperature is also important, as it can influence the agglomeration process, facilitating or hindering adhesion with the binding agent, as well as its final properties. Although the feeding rate and working temperature directly affect the processing time, the main parameter to control is the droplet size, which is influenced by other parameters such as air convection (flow rate) and atomisation pressure (Kramm et al., 2023). The higher the air pressure and flow rate, the smaller the droplet diameter (Chaussonnet et al., 2020), which leads to a more uniform suspension, better fluidisation and ultimately, a more homogeneous mixture. In addition, the concentration must be adequate to ensure strong bonds between the powders and the binding agent. Agglomeration is a process that may improve the dispersion and solubility of fine powders (Dacanal & Menegalli, 2009), facilitating the preparation of fruit and vegetable-enriched smoothies. Furthermore, the inclusion of a rosemary extract through the atomisation liquid provides the antioxidant properties of smoothies.

For these reasons, the research objectives were:

- To incorporate a water-soluble rosemary extract into the powdered smoothie through agglomeration or instantization to facilitate dispersion and dissolution.
- To validate the final product and the agglomeration process using physicochemical and sensory assessments.

MATERIAL AND METHODS

Experimental design.

A randomised design was performed with two different smoothies: (i) fruit and (ii) vegetable, both enriched with rosemary extract (RE). For both cases, two experimental smoothies were compared: (i) untreated and (ii) agglomerated (800 mg RE/kg smoothie powder). CIE-Lab colour, water activity, pH, water absorption and water solubility index, rosmarinic acid content and total phenolics antioxidant activity were studied in the smoothies.

Smoothies manufacturing

Fruit smoothies were manufactured at the Pilot Plant of IRTA, Monells, Girona, Spain. Smoothie ingredients are shown in Table 1.

Table 1. Smoothie ingredients (g/kg).

	Fruit Smoothie	Vegetable Smoothie
Apple	427	290
Orange	341	--
Banana	85	--
Pumpkin	--	375
Carrot	--	188
Whey protein concentrates WPC 80%	147	147

Fruits were purchased in a local market. A commercial whey protein WPC80 (DMV, Campinas, SP, Brazil) was used. To obtain the smoothies, fruits were first washed with tap water. Apples carrot and pumpkin were juiced using a J100 cutter (Robot Coupe, Mataró, Barcelona, Spain), oranges were juiced using an Easy-Pro juicer (Mizumo Elche. Alicante, Spain), and bananas were pureed together with some of the orange juice using a TR 550 industrial hand cutter (Sammic, Azkoitia, Gipuzkoa, Spain).

Rosemary Extract 146.

An aqueous rosemary extract (R146) of high polyphenol content (145.6 mg/g) was used. RE146 consisted in a yellow-brownish powder (Figure 1) with an intense flavour described as herbal, bitter and astringent (Martínez-Tomé et al., 2022). This extract was obtained by previous steam distillation of fresh rosemary leaves to obtain essential oil and further extraction of distilled leaves with water. For more information, see Cedeño-Pinos et al., (2020). As expected, the most abundant polyphenols present in rosemary extract were non-volatile polyphenolic acids, while the concentration of lipophilic polyphenols such as carnosol and carnosic acid was less relevant (Table 2).

Figure 1. Rosemary aqueous extract (RE146) used for the agglomeration of powdered smoothies.

Table 2. Polyphenols identified in rosemary aqueous extract (RE146) (Cedeño-Pinos et al., 2020).

Polyphenols	Classification	mg/g
Rosmarinic acid	Phenolic acid	76.77
Salvianolic acid	Phenolic acid	16.89
Luteolin-glucoside derivative	Flavonoid	11.88
Luteolin-7-O-glucuronide	Flavonoid	10.77
Hesperidin	Flavonoid	11.25
Luteolin	Flavonoid	7.62
Carnosic acid	Terpenoid	4.13
Genkwanin	Flavonoid	3.33
Cirsimaritin	Flavonoid	2.64
Carnosol	Terpenoid	0.38
Total phenolic acids		93.66
Total flavonoids		47.49
Total terpenoids		4.51
Total polyphenols		145.66

Agglomeration conditions at the Pilot Plant.

Coarse smoothie powders (collected from the dehydration chamber) obtained by Spray Drying were agglomerated in a GEA STREA-1 Pro fluid bed equipment (GEA Pharma Systems AG, Switzerland) (Figure 2). 20 g aspersion liquid containing rosemary extract dissolved in distilled water at room temperature was sprayed over 4 min (5 g/min) under the conditions described in Table 3. Freshly-agglomerated smoothies (Figure 3) were collected in a plastic bag and frozen at -80 ºC until their assessment.

Figure 2. Aeromatic AG Strea 1 Pro Fluid Bed Agglomerator (generated by IA).

Table 3. Agglomeration conditions used for powdered smoothies.

Powder feeding	0.3 kg
Liquid sprayed	20 g
RE146 concentration in sprayed liquid	0.6 g/100 g
Spraying time	4 min
Inlet air temperature	80 ºC
Product temperature	45 ºC
Drying time	8 min
Stop temperature	60 ºC

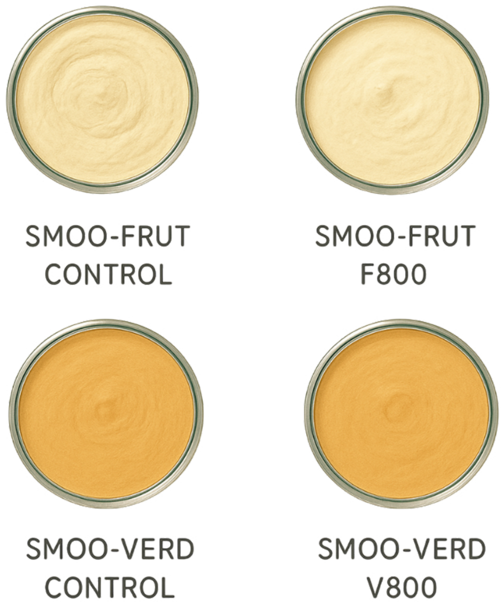

Figure 3. Fruit (above) and vegetable (bellow) powdered smoothies with (right) and without (left) rosemary extract.

Moisture content.

Moisture content was determined with an infrared thermobalance MA 50.R (Radwag, Poland) at 5 °C/min up to 120 °C.

Particle size analysis.

Particle size distribution was determined by laser light scattering using a Mastersizer 3000 (Malvern, UK). The refractive index and absorption coefficient were set at 1.52 and 0.01, respectively. The cumulative particle size distributions were presented graphically. The mean diameters of the particle size based on volume-weighted was calculated as D [4,3] value with the following equation:

$$D[4,3] = \frac{\sum_1^n {D^4}_{i\ v_i}}{\sum_1^n {D^3}_{i\ v_i}}$$

Where the Di value was the geometric mean, the square root of upper x lower diameters. For the numerator, the geometric Di was taken to the fourth power x the percent in that channel, summed over all channels; for the denominator, the same calculation was done, but with Di taken to the third power.

CIELab colour.

Colour was measured in triplicate on the powders and rehydrated liquid, using a CR-400 colorimeter (Konica Minolta, Japan). Results were expressed as CIELab values: lightness (L*), redness (a*) and yellowness (b*). These data were used to calculate the saturation (Chroma, $C^* = (b^{*2} + a^{*2})^{1/2}$) and hue angle ($H^* = \arctan(b^*/a^*)$).

pH.

pH was measured by dissolving approximately 12.5 g powder (depending on the relative humidity of the liquid smoothie) in 50 mL distilled water, and the result was recorded in triplicate, using a pH meter (Crison, Barcelona, Spain) with a combined electrode Cat. No. 52-22 (Ingold Electrodes with a Inc., Wilmington, USA) and a magnetic stirrer.

Water activity.

Water activity (a_w) was measured in triplicate by weighing approximately 5 g powder in a plastic container using a LabMaster-a_w (Novasina AG, Switzerland).

Water Solubility Index (WSI) and Water Absorption Index (WAI).

2 g powder and 25 mL MiliQ water were added to 50 mL centrifuge tubes (previously weighed), in triplicate, and agitated in a vortex mixer for 1 min. The tubes were kept in a water bath with agitation (37 ºC; 30 min) and centrifuged for 10 min at 4560 g in a refrigerated (4 ºC) centrifuge Dicigen 21 R (Ortoalresa, Spain). The supernatant was transferred to pre-weighed Petri dishes and dried in a stove at 105 ºC for 24 h. The weight of the dried residue was recorded to calculate the WSI = (Weight of dried supernatant residue / Initial sample weight) × 100; and the weight of tubes with the residue to calculate the WAI = Weight of residue after centrifugation / Initial sample weight.

Rosmarinic acid.

The identification and quantification of rosmarinic acid was carried out using a UV absorption spectrum at 330 nm and the retention time (5.360 min) of a reference standard of rosmarinic acid (Cedeño-Pinos et al., 2020). To determine rosmarinic acid by HPLC-DAD, 2 g rehydrated smoothie samples were used, in triplicate. For this, approximately 0.5 g powder (depending on the initial relative humidity) was brought to 2 g with Mili-Q water. Then, they were made up to volume with methanol in a 10 mL volumetric flask. They were stirred at room temperature for 5 min, placed in an ultrasound bath for 5 min and centrifuged at 5000 rpm (4 ºC; 10 min). The supernatant was filtered at 0.22 μm, collected in 2 mL opaque glass vials and stored at -80 ºC until next use. 20 μL samples were injected into an Infinity II HPLC-DAD-1260 Series system (Agilent Technologies, USA). A Brisa LC2 C18 column (25 cm x 0.46 cm x 5 μm) (Teknokroma, Spain) with a flow rate of 1 mL/min was used. As a mobile phase, 0.05% acidified water was used in channel A with formic acid, and acetonitrile at 100% in channel B.

Total phenolic content.

Total phenolic content (TPC) was determined using a Folin-Ciocalteu modified method (Cedeño-Pinos et al., 2020), in which the oxidising reagent reacted with reducing agents or antioxidants, such as polyphenols, forming a soluble complex with an intense blue colour (Pérez et al., 2023). Sample extraction was carried out as previously mentioned, but the supernatant was collected in Eppendorf tubes and stored at -80 ºC. For the reaction, 250 μL sample extract, 800 μL Folin-Ciocalteu reagent and 5 mL Mili-Q water were added to a 10 mL volumetric flask and shaken. After 8 min, 1.2 mL sodium carbonate (20%) was added and made up to the meniscus with water. The solutions were kept in a water bath (20 ºC; 2 h). Absorbance was measured at 760 nm using a UV-Visible spectrophotometer GENESYS 180 (Thermo Fisher, USA) and water as a blank. Gallic acid (GA) was used as standard for quantification, using a calibration line ($R^2 = 0.9981$) from 1.5 to 5.5 μg GA/mL. Results were expressed as mg gallic acid equivalents (GAE)/100 g.

Sensory triangular test.

A sensory triangular test (UNE-87-006-92) was performed with trained panellists at the University of Murcia to discriminate rosemary flavour in the rehydrated smoothies (3:1 *w:w*, water: powder). The panel was composed of 6 internal tasters, who claimed they consumed smoothies from several times a year to several times a month. Tasters were trained with water solutions of RE at concentrations around those used in smoothies. Each taster performed a total of 12 triangular tests per each smoothie formulation (fruit and vegetable) and RE dose (800 and 400 mg RE/kg powder) on randomly coded samples (untreated and treated).

Statistical analysis.

A one-way ANOVA test was used to determine the effects of RE addition by agglomeration on the fruit and vegetable smoothies. Sample size per each treatment level was n = 6 (2 formulations × 3 replicates). The Tukey's rank test was used ($p < 0.05$) for pairwise mean comparisons. Data were analysed with Statistix 8.0 software for Windows (Analytical Software, Tallahassee, Fl, USA).

RESULTS

The results of physicochemical assessment of fruit and vegetable powdered smoothies (FPS and VPS) are shown in Table 4. After agglomeration, the increase in the average particle size was higher for FPS than for VPS. Moisture content increased in the FPS while, in contrast, decreased in the VPS, which dehydrated under the drying conditions existing in the agglomerator. Agglomeration slightly decreased L* value in both powdered smoothies, decreased a* value in VPS and increased b* value in FPS. Thus, the agglomeration of fruit rehydrated smoothie, resulted in a slightly darker powders, while chromatic differences were less clear. The value of pH slightly increased in both agglomerated smoothies. Agglomeration decreased water activity in the VPS but not in the FPS and did not affect the water absorption ratio and only increased the while the water solubility index in the VPS.

Table 4. Physical characterization of fruit and vegetable powdered smoothies.

		Untreated		RE		
		M		M		SEM
Moisture content (g/100g)	Fruit	3.95	b	4.70	a	0.206
	Vegetable	6,00	a	5.15	b	0.901
Average particle size ($D_{[4,3]}$)	Fruit	55.30	b	61.65	a	0.050
	Vegetable	70.20	b	72.75	a	0.254
CIELab colour (CIE units)						
L*	Fruit	87.37	a	86.78	b	0.041
	Vegetable	81.75	a	81.59	b	0.046
a*	Fruit	2.10		2.17		0.050
	Vegetable	10.31	a	10.07	b	0.034
b*	Fruit	21.06	b	21.63	a	0.047
	Vegetable	32.34		32.20		0.100
Chroma	Fruit	21.16	b	21.74	a	0.047
	Vegetable	33.94		33.74		0.088
Hue	Fruit	84.31		84.26		0.132
	Vegetable	72.31	b	72.63	a	0.100
pH	Fruit	5.77	b	5.81	a	0.005
	Vegetable	5.56	b	5.89	a	0.007
Water activity	Fruit	0.22		0.22		0.002
	Vegetable	0.31	a	0.22	b	0.001
Water absorption	Fruit	0.18		0.16		0.008
	Vegetable	0.43		0.41		0.015
Water solubility index (%)	Fruit	85.82		86.38		0.306
	Vegetable	84.84	b	86.35	a	0.106

Treatments: untreated and RE (agglomerated with 800 mg RE/kg powder). M: Mean; SEM: Standard error of the mean. ^{a,b}: Agglomeration effects ($p < 0.05$; Tukey test).

Phenolic total contents are shown in Figure 4. Addition of RE clearly increased the concentrations of total phenolics in both powdered smoothies. The percentage of rosmarinic acid, the most abundant polyphenol from RE, recovered after agglomeration was around of 100% in both powders (Figure 5). Thus, agglomeration treatment did not seem to affect the recovery of rosmarinic acid, as no losses occurred during the process. Moreover, the standard error of the mean was low (<0.8) for an addition level of 15.4 mg rosmarinic acid /kg powder, which revealed a good homogenisation of this acid in the powders. The results obtained in the triangular test (Figure 6) with rehydrated samples confirmed that rosemary off-flavour was clearly detected by the trained panellists at concentrations of 800 mg RE per kg powder. By contrary, rosemary off-flavour was not detected when RE dose was reduced by half.

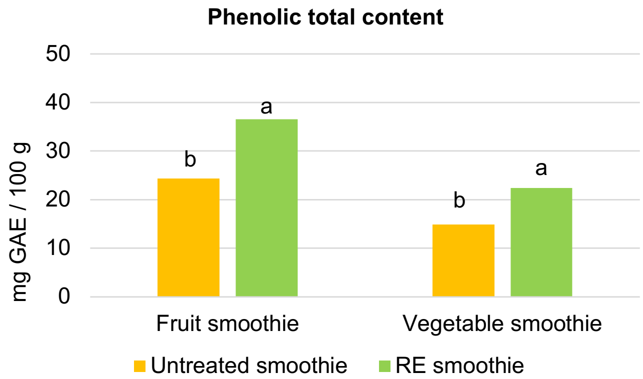

Figure 4. Phenolics total content of fruit and vegetable powdered smoothies. Treatments: untreated and RE (agglomerated with 800 mg RE/kg powder). Standard error of the mean: Fruit: 0.392; Vegetable: 2.656. [a,b]: Agglomeration effects ($p < 0.05$; Tukey test).

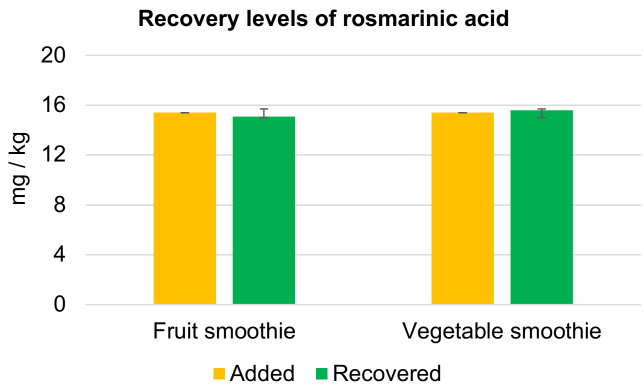

Figure 5. Recovery levels of rosmarinic acid in fruit and vegetable smoothies. Addition level: 15.4 mg/kg powder; Standard error of the mean: Fruit: 0.5; Vegetable: 0.8.

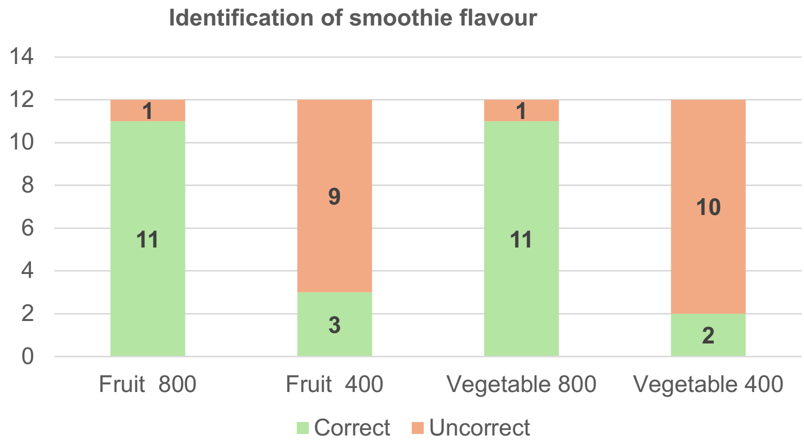

Figure 6. Identification of smoothie flavour by a trained sensory panel. At least 10 correct responses are required for a positive identification ($p < 0.05$) according to the ISO 11136:2014 (International Organization for Standardization, 2014).

DISCUSSION

The agglomeration treatment used, based on spraying an extract solution in a fluidized bed, has proven effective in incorporating rosemary polyphenols to smoothie powders. However, this treatment required some adjustments to ensure that adequately sized agglomerates were formed without collapsing the equipment's filters, while also preventing excessive overheating. A series of preliminary process set-up tests were firstly carried out under different conditions. Because their suitable flowability, coarse powders collected in the dehydration chamber from SD resulted more suitable for agglomeration. Some key aspects were the selection of the grids to avoid powder losses and the duration of the agglomeration process, which should not exceed 8 minutes with the equipment used. The quality control of powders before and after agglomeration verified that the average particle size of the powders increased slightly, which should facilitate their rehydration, and that their moisture content changed slightly by around 4-5%, which should stabilize their stability for a long time.

Agglomeration treatment modified some quality traits of the powdered smoothies. These changes might be due to the addition of rosemary pigments, morphological changes in agglomerates, and moisture readjustment. Fruit powdered smoothie tended to yellow hue, while vegetable powdered smoothie tended to orange hue. RE146 used is a yellow-brownish and dark powder able to modify the original colour of these smoothies despite it was present at very low concentrations. A slight darkening can be quantified by reflectometry, being more evident in the product with fruit. Whatever the case, it cannot be stated clearly that agglomeration with RE146 enhances or decreases yellowness or orangeness in these powders, as can be seen in Figure 3. Agglomeration with RE slightly increased the pH because this extract had a higher pH value than both powdered smoothies. This effect was more pronounced for the product with vegetable because its pH was somewhat lower. Martínez-Tomé et al. (2022) reported that RE146 does not affect the pH value in a yogurt sauce, and more acidic product than smoothies. Water activity only decreased in the vegetable powdered smoothie because agglomeration process involved some additional dehydration in these samples. Agglomeration was not significant in terms of water solubility and water absorption index in fruit smoothies, as the selected conditions (working temperature, spraying and drying time) for our main objective, which was to add the rosemary extract, were not optimal to achieve the conventional objective or use of this technology, which initially was to increase powder solubility. Even so, water solubility index increased in the vegetable smoothies. Finally, agglomeration with RE146 hardly affect the water solubility indexes because these smoothies are highly water-soluble matrixes that came from a previous Spray Drying process.

A relevant finding of this study was that the addition by agglomeration of a little quantity of rosemary polyphenols (117 mg polyphenols/ kg powder) resulted in a considerable increase in the response of phenolic antioxidants assessed as gallic acid equivalents. This fact was confirmed in both powdered smoothies, despite they are often considered as a rich source of phenolic compounds, given their formulation and ingredient composition (Flavones, flavonols, flavan-3-ols, anthocyanins, phenolic acids and tannins) (Kidoń & Uwineza, 2022; Singh et al., 2016; Starowicz et al., 2020). These results agree with those obtained by other authors on apple juice enriched with aromatic plants, which significantly increased total polyphenol and flavonoid content (Ivanišová et al., 2015). Other products, such as soy milk, also showed a higher phenolic content in samples containing rosemary extract (Qiu et al., 2018). The presence of these phenolic compounds enhances antioxidant capacity, due to their structural properties. Depending on the position and amount of the hydroxyl and methoxy groups, the ability to donate hydrogen atoms and scavenge free radicals will vary, being higher in phenolic acids, flavanols and flavan-3-ols, which could improve the shelf life of smoothies and preserve their nutritional value (Pérez et al., 2023). This improvement in the antioxidant capacity of the rehydrated smoothies seems to be related to the fact that rosmarinic acid, and probably other rosemary polyphenols, resist the agglomeration process without degrading. Furthermore, rosmarinic acid is generally considered to be stable to the heating treatments used in food products, although its stability may depend on other factors such as handling, interaction with ingredients or storage conditions (Cedeño-Pinos et al., 2020).

A possible limitation of using RE would be that smoothie flavour may change due to the herbal, spicy, bitter and astringent tones provided by rosemary. As seen, the trained panellists detected RE off-flavour relatively easily in both rehydrated smoothies made with a high dose of RE, although they did not detect this off-flavour when the dose was reduced by half. Consumers are often less accurate at detecting off-flavours than the trained panellists, so a sensory study with consumers would be necessary to establish the dose at which this RE can be added to both powdered smoothies. In other study on orange and apple juices, panellists accepted an addition of 10 mL/L rosemary extract without any detriment of flavour scoring (Waley et al., 2020). In any case, the incorporation by agglomeration of polyphenolic rosemary extracts from industrial by-products improves the antioxidant status of powdered smoothies, which might enhance their potential functional and their oxidative stability.

CONCLUSIONS

Agglomeration of fruit and vegetable powdered smoothies with rosemary extract provides added polyphenols that may improve their functional and nutritional value. Agglomeration process does not degrade rosemary polyphenols and improves powder water solubility and water activity, although can change the colour depending on the powder. The agglomeration with rosemary extract does not affect the sensory quality of smoothies in the testing conditions. Agglomeration with functional ingredients can be offered as a technological strategy to improve nutritional quality in powdered smoothies. The food industry has a chance to ensure the recommended daily intake of fruits and vegetables by providing value-added and sustainable products.

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