

## Research Article

# Influence of Drying Method on the Composition, Physicochemical Properties, and Prebiotic Potential of Dietary Fibre Concentrates from Fruit Peels

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Dietary fibre concentrates (DFC) obtained from fruit and vegetable by-products are powders, mainly obtained by dehydration, used in food formulations to increase nutritional value and to improve functional properties. The modifications of insoluble, soluble, and total dietary fibres (IDF, SDF, and TDF), physicochemical properties (solubility, swelling capacity, water/oil retention capacity, pH, and tapping density), and prebiotic potential of DFC from orange, mango, and prickly pear peels obtained by freeze-drying (FD) and convective hot air-drying (HA) were studied. *In vitro* faecal fermentation was used to evaluate the short-chain fatty acid (SCFA) production as a prebiotic indicator. TDF in FD orange was 5.5 g·100 g<sup>-1</sup> higher than that in the HA sample, whereas HA increased TDF in prickly pear (9.5 g·100 g<sup>-1</sup>). No differences in fibre composition were observed in mango DFC. The physicochemical properties mostly affected by dehydration treatment were solubility and swelling capacity. HA increased SCFA production in orange peel (48 mmol·g<sup>-1</sup> higher) but decreased it in mango and prickly pear (15 and 19 mmol·g<sup>-1</sup> lower). Butyrate production of HA orange DFC was comparable to that obtained with the positive control (4.5 mmol·g<sup>-1</sup>). No production of propionate or butyrate was observed after 6 h fermentation in mango samples, despite the high SDF content (≈20 g·100 g<sup>-1</sup>). A decrease of the SDF:TDF ratio produced by the drying method improved the SCFA production.

## 1. Introduction

Dietary fibre has been widely studied due to the health benefits produced by its consumption. A regular ingest of dietary fibre in the diet decreases the cholesterol and glucose levels in the blood [1, 2] and reduces the risk of haemorrhoids, colorectal polyps, and diverticulitis as a result of its laxative effect [3]. Dietary fibre could be classified, based on its water solubility, into soluble and insoluble dietary fibres (SDF and IDF, resp.), and SDF is composed of both high- and low-molecular weight soluble dietary fibres [4]. The proportion of SDF and IDF, as well as the composition, is

related to the physiological and physicochemical properties of the fibres [5].

Fibre used in the food industry is commonly obtained from cereals, but fruit and vegetable sources contain higher proportions of SDF [6], leading to more diverse physicochemical properties. Fibres obtained from fruits and vegetables used by the food industry are frequently obtained using chemical, mechanical, thermal, and enzymatic processes. Most of these treatments involve the use of high amount of solvents, expensive enzymes, long processing time, or the use of high temperature [7, 8]. For this reason, in the last 20 years, several studies have been focused on the

characterization [5, 9] and application [10, 11] of dietary fibre concentrates (DFC) obtained by the dehydration of fruits and vegetables. DFC are composed of less than 9% lipids and total dietary fibre (TDF) of at least 50% [12], and they are successfully applied in the food industry [5] to increase nutritional value taking advantage of their physicochemical properties for food formulations. In this regard, some fruit and vegetable by-products, such as peels, pomace, and bagasse, with a high content of dietary fibre, have succeeded in obtaining DFC and their inclusion in foods formulations as functional ingredients [13–15].

Drying is the main unit operation to produce DFC. Water removal increases the powder's shelf life and facilitates its use in food applications. Drying processes modify DFC's physical and chemical properties, whereby the selection of the dehydration method should be accompanied by the characterization of the final properties of DFC [16]. Convective hot air-drying (HA) is the most common dehydration method used to obtain DFC [17]; however, the temperature used in HA has shown to have a negative effect on the DF content and composition which potentially may modify its functional and technological properties [12], whereby freeze-drying (FD) might be used as an alternative. During FD, the water is removed by sublimation under high vacuum and freezing temperatures. The food industry prefers FD over HA because the thermal damage of heat-sensitive compounds in foods is avoided, preserving some sensory and structural characteristics. However, FD is an expensive process mainly because of the large energy consumption to maintain the sublimation conditions [18]. Besides, the changes produced in the cell wall during the freezing stage could modify the properties of the fibre powders.

Prebiotics are polysaccharides, mainly fermentable dietary fibres, that resist gastric acid, enzymatic hydrolysis, and gastrointestinal absorption. These polysaccharides reach the colonic microbiota conferring benefits to the consumer's health such as the improvement of host immunity, reduction of pathogenic bacterial population, and the production of SCFAs (primarily butyrate, propionate, and acetate) [19, 20]. Few studies have been reported regarding the prebiotic activity of fruit and/or vegetable by-products. However, these works have been conducted with specific carbohydrates used as prebiotics, chemically or enzymatically extracted. Mandalari et al. [21] studied the *in vitro* prebiotic activity of pectic oligosaccharides from bergamot peel. They demonstrated a higher prebiotic potential of the bergamot peel oligosaccharides than that of the fructooligosaccharides (FOS) used as a control. A similar study was performed by Rubel et al. [22] using inulin-type carbohydrates from Jerusalem artichoke as prebiotics, which showed better prebiotic activity than commercial inulin. For their part, Reis et al. [23] found larger SCFA production and lactate concentrations after a faecal fermentation of arabinoxylans extracted from brewer's spent grain than that in cultures containing FOS. *In vitro* faecal fermentation of agave fructans was conducted by Gomez et al. [24], evaluating changes in the bacterial population and SCFA production. Nevertheless, to our knowledge, only prickly pear and

pineapple peels [25] and tiger nut coproducts [26] have been used to study the prebiotic potential of DFC. The prebiotic potential in these studies was demonstrated by plate microbial growth curves and quantification of SCFAs from culture media, but faecal fermentation of DFC has not been verified. The objective of this research was to evaluate changes in the dietary fibre composition and physicochemical properties of DFC from orange, mango, and prickly pear peels, produced by FD and HA methods. Also, the influence of these drying methods on the DFC's prebiotic potential was evaluated through SCFA production after *in vitro* faecal fermentation.

## 2. Materials and Methods

**2.1. Materials.** Orange (*Citrus sinensis* cv. Valencia) bagasse (peel, seeds, and pulp) was provided by a local juice producer (Central Market, Guadalupe, NL, Mexico) immediately after the juice extraction process. Prickly pear (*Opuntia ficus-indica* cv. Verde Villanueva) was provided by the producer "La Flor de Villanueva" (Acatzingo, Puebla, Mexico). Mango (*Mangifera indica* cv. Ataulfo) peel was provided by a local food-processing company "Genius Foods" (Monterrey, NL, Mexico). Samples of orange juice and mango pulp obtained from the same batch of the by-products were also provided to evaluate the fruit maturity stage. Orange and mango peels were kept at 4°C for no more than 4 h before sample preparation. Enzymatic kit for DF analysis (K-INTDF 06/13) and ion exchange resins (Amberlite FPA53® (OH<sup>-</sup>) and Ambersep 200® (H<sup>+</sup>)) were obtained from Megazyme (Wicklow, LEN, Ireland). All reagents for DF, protein, and fat quantification, chromatographic standards, P<sub>4</sub>O<sub>10</sub>, and SCFA determination were obtained from Sigma-Aldrich (St. Louis, MO, USA).

**2.2. Sample Preparation.** Orange peel (albedo and flavedo) was manually obtained by removing pulp and seed residues. Prickly pear fruits and mango and orange peels were sanitized in a sodium hypochlorite solution for 10 min. Prickly pears were manually peeled, and pulp and seeds were discarded. For maturity stage identification, 500 mL of prickly pear pulp was stored. In the case of DFC from freeze-dried orange, mango, and prickly pear peels, fresh fruit peels were grounded (VM0103; Vitamix, Cleveland, OH, USA) and frozen at -50°C for 24 h. Frozen samples were freeze-dried at -50°C and 2.0 mbar (Labconco, Kansas City, MO, USA), hand-milled, and sieved through 40 mesh. In the case of DFC from orange, mango, and prickly pear peels obtained by convective hot air-drying (HA), fruit peels were dried using a convective hot air dehydrator (model IME-08; EDEL Ingenieros, Monterrey, NL, Mexico) at 55 ± 5°C and air flow rate of 1.5 m·s<sup>-1</sup> for 12 h until constant weight was obtained. Dried samples were grounded in a pilot plant scale hammer mill (model 200; Pulvex, Mexico City, Mexico) and sieved through 40 mesh (425 μm). Powders were stored in desiccators containing P<sub>4</sub>O<sub>10</sub> at 25°C to avoid sample humidification before analysis.

**2.3. Proximate Composition.** Moisture, protein ( $N \times 6.25$ ), ash, and fat content from dried samples were determined following the AOAC Official Methods 920.151, 920.152, 940.26, and 960.39, respectively. All proximate results, except for the moisture content, were reported on dry basis (db). The titratable acidity and soluble solids from fresh juice (orange) and pulp (mango and prickly pear) were evaluated according to the AOAC Methods 942.15 and 932.12 [27], respectively. Titratable acidity was expressed as g of citric acid per kg of juice. The maturity index was calculated as the soluble solids ("Brix"): titratable acidity ( $\text{g}\cdot\text{kg}^{-1}$  (wb)) ratio.

**2.4. Dietary Fibre Evaluation.** IDF and high- and low-molecular weight soluble dietary fibres (referred as SDFP and SDFS, resp., depending on the precipitation or solubilization in the presence of 76% ethanol) were evaluated following the AOAC 2011.25 procedure proposed by McCleary [28] with some modifications previously reported by Tejada-Ortigoza et al. [8], using the Megazyme integrated total dietary fibre kit (Megazyme International Ireland, Wicklow, Ireland). SDF was obtained as the sum of SDFP and SDFS, whereas total dietary fibre (TDF) was obtained as the sum of SDF and IDF. To quantify the SDFS fraction, a Waters 717 plus autosampler operating at  $4^\circ\text{C}$  was used to inject samples from vials to the liquid chromatograph. SDFS was separated through a Bio-Rad Aminex<sup>®</sup> column ( $300 \times 7.8$  mm) at  $90^\circ\text{C}$ , with a flow rate of  $0.5 \text{ mL}\cdot\text{min}^{-1}$  of  $\text{Na}_2\text{Ca-EDTA}$  ( $50 \text{ mg}\cdot\text{L}^{-1}$ ), and analyzed with a Waters refractive index detector 2414 set at  $50^\circ\text{C}$ .

## 2.5. Physicochemical Properties

**2.5.1. Solubility (SOL).** The DFC solubility was obtained with samples (200 mg) suspended in water (30 mL) in 50 mL parafilm-covered beakers and stirred for 3 h at  $25^\circ\text{C}$ . Suspensions were placed in 50 mL centrifuge tubes and centrifuged ( $3000 \times g$ ,  $22^\circ\text{C}$ , and 20 min). The supernatant was carefully discarded, and the pellet was washed with 10 mL of distilled water and filtered using a filter paper (Whatman no. 41). The filter paper with the pellet was air-dried at  $60^\circ\text{C}$  for 24 h. SOL was calculated using the following equation:

$$\text{SOL} = \frac{W_S - W_{\text{DP}}}{W_S} \times 100, \quad (1)$$

where SOL is the solubility of the sample expressed as a percentage and  $W_S$  and  $W_{\text{DP}}$  are the weight of the sample (db) and the weight of the dried pellet, discarding the filter paper weight, respectively, expressed in g.

**2.5.2. Swelling Capacity (SC).** Defined as the resulting volume of the sample after hydration, SC was obtained suspending 200 mg of the DFC with distilled water (10 mL) in a foil-covered graduated test tube. Suspensions were manually stirred with a glass tube to avoid agglomerations. After 24 h at  $25^\circ\text{C}$ , the volume of the precipitate was achieved. SC was calculated using the following equation:

$$\text{SC} = \frac{V_P}{W_S}, \quad (2)$$

where SC is the swelling capacity of the sample expressed as  $\text{mL}\cdot\text{g}^{-1}$  and  $V_P$  is the volume of the precipitate obtained after hydration expressed in mL.

**2.5.3. Water Retention Capacity (WRC).** This parameter was attained using the methodology proposed by Chau and Huang [29] with some modifications. DFC (1 g) were placed in 50 mL foil-covered beakers with 10 mL of distilled water and continuously stirred for 18 h at  $25^\circ\text{C}$ . Suspensions were carefully placed in 15 mL graduated tubes and centrifuged ( $4,500 \times g$ ,  $22^\circ\text{C}$ , and 30 min). The volume of the supernatant and the precipitate was recorded. The WRC was calculated using the following equation:

$$\text{WRC} = \frac{10 - V_S}{W_S}, \quad (3)$$

where WRC is the water retention capacity of the sample expressed as  $\text{mL}\cdot\text{g}^{-1}$  and  $V_S$  is the volume of the supernatant obtained by centrifugation expressed in mL.

**2.5.4. Oil Retention Capacity (ORC).** Oil retention capacity was obtained similarly to WRC but using soybean oil instead of distilled water. ORC is expressed as  $\text{mL}\cdot\text{g}^{-1}$ .

**2.5.5. pH.** Five grams of DFC were suspended in 50 mL of distilled water and stirred for 5 minutes. pH was measured immediately using a pH meter (Orion 3-Star, Thermo Scientific, USA) at  $25^\circ\text{C}$ .

**2.5.6. Tapping Density (TD).** Tapping density was determined by placing the DFC in graduated test tubes until they reached 10 mL and recording the weight (db). The test tube was tapped on a table for 5 minutes. The final volume after tapping was recorded ( $V_T$ ). TD ( $\text{mg}\cdot\text{mL}^{-1}$ ) was calculated as follows:

$$\text{TD} = \frac{V_T}{W_S}. \quad (4)$$

**2.6. In Vitro Fermentation of DFC.** All the experiments of the *in vitro* fermentation of DFC were performed in the facilities of the Whistler Center for Carbohydrate Research at Purdue University, West Lafayette, IN, USA.

**2.6.1. Sample Preparation.** The samples for *in vitro* fermentation were prepared following the methodology adapted by Rose et al. [30]. FD and HA samples (6 g) were suspended in 84 mL of 20 mM phosphate buffer (pH 6.9, 10 mM NaCl) and heated for 20 min in a boiling water bath. Samples were cooled down and placed in a shaking water bath at  $37^\circ\text{C}$  for enzymatic digestions. The first digestion was conducted with salivary  $\alpha$ -amylase for 15 min, followed by a 30 min digestion with porcine pepsin and a 90 min digestion with porcine pancreatin. All enzymatic digestions

were performed at 150 rpm. Digested samples were dialyzed against distilled water for 26 h (Spectra/Por1; MW cutoff 6–8000 Da; Spectrum Labs, Rancho Dominguez, USA)

**2.6.2. Faecal Slurry Preparation.** Fresh human faecal samples were obtained from three healthy adult donors, one woman and two men between 25 and 30 years. Volunteers consumed their normal diet, had no history of gastrointestinal abnormalities in the last 6 months, and had not taken antibiotics for at least 3 months. The samples were collected on the morning of the experiment into sterile vials using a commode specimen collection system. Faeces were kept at 4°C before use, for no more than 2 h after collection. To obtain the faecal slurry, equal weights obtained from the internal part of the faecal samples were pooled and combined with carbonate : phosphate buffer (1 : 10), following by a filtration through 4 layers of cheese cloth. Donors signed an informed consent before the experiment initiation, and all experimental protocols were approved by the Institutional Review Board at Purdue University (IRB protocol #1510016635).

**2.6.3. Inoculation and In Vitro Fermentation.** *In vitro* fermentation of DFC was conducted in an anaerobic chamber (85% N<sub>2</sub>, 5% CO<sub>2</sub>, and 10% H<sub>2</sub>) following the methodology adapted by Tuncil et al. [31]. Cysteine (0.1 g/mL) was added into sterilized carbonate-phosphate buffer (0.25 g·L<sup>-1</sup>), and oxygen was removed bubbling with carbon dioxide. The buffer was conditioned in the anaerobic chamber overnight. 44 ± 0.5 mg of dialyzed dietary fibre concentrate samples was placed in test tubes (4 tubes per sample were considered for each fermentation point, i.e., 0, 6, 12, and 24 h) and located into the anaerobic chamber (FOS were used as a positive control), where 4 mL of the conditioned carbonate-phosphate buffer was added to each tube. Then, each tube was inoculated with 0.4 mL of the faecal slurry, sealed with rubber stoppers, and incubated in a water bath at 37°C and 150 rpm. Blank test tubes per each time point were prepared with no substrate addition.

**2.7. Short-Chain Fatty Acid (SCFA) Quantification.** SCFA quantification was conducted following the methodology described by Rose et al. [30]. Briefly, for each fermentation time point, 0.4 mL of the fermented samples was collected and mixed with 100 µL of a mixture of 4-methylvaleric acid, 85% phosphoric acid, and copper sulphate pentahydrate (157.5 µL, 1.47 mL, and 9 g, resp.), used as an internal standard. 400 µL of copper sulphate (2.75 mg·mL<sup>-1</sup>) was added to stop fermentation, and purified water was used to obtain a final volume of 25 mL. The samples were kept at -80°C until further analysis. Defrosted samples were centrifuged for 10 min at 3000 × *g* (Microfuge 20R; Beckman Coulter, Brea, CA), and 4 µL of the supernatant was injected into the gas chromatograph (5890 Series II, Hewlett Packard, Palo Alto, CA) equipped with a fused silica capillary column (Nukol; Supelco nr 40369-03A, Bellefonte, PA) to separate fatty acids. SCFAs were identified with a flame ionization

TABLE 1: Maturity stage parameters of orange, mango, and prickly pear.

	Orange	Mango	Prickly pear
Soluble solids (°Bx)	15.0 ± 0.0	23.4 ± 0.5	16.8 ± 0.0
Titrateable acidity (g·kg <sup>-1</sup> )*	6.9 ± 0.3	10.7 ± 1.4	1.8 ± 0.3
Maturity index**	2.2 ± 0.1	2.2 ± 0.3	9.3 ± 1.3

Values are mean of three determinations ± SD; \*g of citric acid per kg of fruit pulp (wb); \*\*soluble solids/titrateable acidity.

detector (GC-FID 7890A; Agilent Technologies, Santa Clara, CA). A standard mix (Supelco, Bellefonte, PA) was injected to determine acetate, propionate, and butyrate relative to 4-methylvaleric acid. Concentration of SCFAs is expressed as mmol per 1 g (db) of DFC.

**2.8. Statistical Analysis.** All experiments were performed in quadruplicate, except for SCFA quantification which was conducted in triplicate. Experimental data were analyzed using the Minitab statistical software V. 14.1 (Minitab Ltd., Coventry, UK). Comparison of means was evaluated by one-way ANOVA, followed by Tukey's multiple comparison test to find significant differences between treatments ( $p < 0.05$ ).

### 3. Results and Discussion

**3.1. Proximate Composition of Dehydrated Fruit Peels.** The soluble solids and titrateable acidity and their ratio (maturity index) of the orange, mango, and prickly pear are shown in Table 1. These parameters were used to characterize the ripening stage of the fruits which is related to the content of digestible and nondigestible carbohydrates (i.e., dietary fibre). Maturity index confirmed that all the fruits were at an edible ripening stage [32–34].

The composition of the DFC studied is given in Table 2; the final moisture content obtained after the dehydration process ranged from 1.3 to 3.5 g·100 g<sup>-1</sup> wb. A similar final moisture content of diverse DFC from fruit by-products has been reported previously. Peerajit et al. [35] obtained a final moisture of 0.05 kg·kg<sup>-1</sup> db (i.e., 4.8 g·100 g<sup>-1</sup> wb) in lime residue dietary fibre powder dried at 60°C in a hot air oven. Watermelon rind concentrates obtained by Naknaen et al. [36] using a hot air oven at 60°C showed a final moisture of 5.4 g·100 g<sup>-1</sup> wb. Freeze-dried date fibres obtained by Ahmed [37] showed final moistures from 2.8 to 4.9 g·100 g<sup>-1</sup> wb. The final moisture contents depicted in Table 2 show no significant differences ( $p < 0.05$ ) between drying technologies utilized for mango peel DFC; however, orange and prickly pear samples dried by HA exhibited a significantly higher moisture content ( $p < 0.05$ ) than FD samples (2.1 and 1.3 g·100 g<sup>-1</sup> wb for HA and FD orange, and 3.5 and 2.8 g·100 g<sup>-1</sup> wb for HA and FD prickly pear, resp.). Similar differences in the final moisture content between methodologies were reported by Hsu et al. [38] in yam flours, who obtained a moisture of 0.6 g·100 g<sup>-1</sup> wb in FD samples compared to 5.4 g·100 g<sup>-1</sup> wb in flours attained by HA. These differences could be explained by two different effects: (1) the long drying duration at high temperatures in HA seals the surface capillaries of the samples reducing the water

TABLE 2: Proximate composition (g·100 g<sup>-1</sup> db) of dietary fibre concentrates from orange, mango, and prickly pear peels freeze-dried (FD) and convective hot air-dried (HA).

	Orange		Mango		Prickly pear	
	FD	HA	FD	HA	FD	HA
Final moisture (wb)	1.3 ± 0.2 <sup>b</sup>	2.1 ± 0.1 <sup>a</sup>	2.5 ± 0.3 <sup>a</sup>	1.9 ± 0.4 <sup>a</sup>	2.8 ± 0.3 <sup>a</sup>	3.5 ± 0.3 <sup>b</sup>
Protein (N × 6.25)	3.6 ± 0.2 <sup>b</sup>	4.9 ± 0.1 <sup>a</sup>	4.1 ± 0.1 <sup>a</sup>	3.8 ± 0.1 <sup>a</sup>	6.1 ± 0.2 <sup>a</sup>	4.4 ± 0.3 <sup>b</sup>
Fat	1.1 ± 0.5 <sup>a</sup>	1.5 ± 0.1 <sup>a</sup>	2.1 ± 0.4 <sup>a</sup>	1.4 ± 0.8 <sup>a</sup>	1.6 ± 0.2 <sup>a</sup>	1.3 ± 0.3 <sup>a</sup>
Ash	3.1 ± 0.2 <sup>b</sup>	4.2 ± 0.1 <sup>a</sup>	3.9 ± 0.2 <sup>a</sup>	2.9 ± 0.2 <sup>b</sup>	21.6 ± 0.2 <sup>a</sup>	18.8 ± 1.0 <sup>a</sup>
Carbohydrates*	92.2 ± 0.4	89.4 ± 0.1	89.9 ± 0.3	91.9 ± 0.5	70.7 ± 0.2	75.6 ± 0.6

Values are mean of three determinations ± SD. Values per fruit (freeze-dried or convective hot air-dried) within the same row followed by different letters are significantly different ( $p < 0.05$ ). \* Carbohydrate content includes digestible and nondigestible carbohydrates (i.e., dietary fibre) and was calculated by difference.

TABLE 3: Dietary fibre composition (g·100 g<sup>-1</sup> db) of dietary fibre concentrates from orange, mango, and prickly pear peels freeze-dried (FD) and convective hot air-dried (HA).

	IDF	SDFP	SDFS	SDF	TDF	SDF : TDF
<i>Orange</i>						
FD	46.3 ± 1.2 <sup>a</sup>	5.6 ± 0.3 <sup>a</sup>	1.7 ± 0.2 <sup>a</sup>	7.4 ± 0.4 <sup>a</sup>	54.7 ± 0.9 <sup>a</sup>	0.14
HA	42.7 ± 0.5 <sup>b</sup>	5.6 ± 0.2 <sup>a</sup>	0.9 ± 0.0 <sup>b</sup>	6.4 ± 0.3 <sup>b</sup>	49.2 ± 0.0 <sup>b</sup>	0.13
<i>Mango</i>						
FD	33.4 ± 0.8 <sup>a</sup>	19.5 ± 0.2 <sup>a</sup>	2.2 ± 0.5 <sup>a</sup>	21.8 ± 0.8 <sup>a</sup>	54.8 ± 1.3 <sup>a</sup>	0.40
HA	32.3 ± 1.3 <sup>a</sup>	20.1 ± 0.6 <sup>a</sup>	0.8 ± 0.0 <sup>b</sup>	20.8 ± 0.4 <sup>a</sup>	53.0 ± 1.3 <sup>a</sup>	0.39
<i>Prickly pear</i>						
FD	34.0 ± 0.6 <sup>b</sup>	4.7 ± 0.2 <sup>b</sup>	1.4 ± 0.2 <sup>a</sup>	6.1 ± 0.3 <sup>b</sup>	40.0 ± 0.9 <sup>b</sup>	0.15
HA	40.7 ± 6.0 <sup>a</sup>	12.3 ± 7.5 <sup>a</sup>	1.4 ± 1.0 <sup>a</sup>	13.3 ± 7.8 <sup>a</sup>	49.2 ± 5.6 <sup>a</sup>	0.27

Values are mean of four determinations ± SD. Values per fruit (freeze-dried or convective hot air-dried) within the same column followed by different letters are significantly different ( $p < 0.05$ ). IDF: insoluble dietary fibre; SDFP: high-molecular weight soluble dietary fibre; SDFS: low-molecular weight soluble dietary fibre; SDF: soluble dietary fibre; TDF: total dietary fibre.

releasing from the matrix [39], and (2) the increase of carbohydrate content, mainly digestible carbohydrates, that is, carbohydrates without including dietary fibre, (data not shown) may reduce the moisture content and monolayer water content of the matrixes due to the cross-linking of the carbohydrates with other compounds of the samples limiting the sites to absorb moisture [40].

Low contents of fat and protein were observed in all samples, the maximum fat value was found in FD mango peel DFC (2.1 g·100 g<sup>-1</sup> db), and FD prickly pear peel DFC showed the highest protein content (6.1 g·100 g<sup>-1</sup> db). The ash content of prickly pear, independently of the drying method, was notably higher than that of mango and orange samples. Similar ash content was reported by Tejada-Ortigoza et al. [8] for FD concentrates from prickly pear peels and by Nharingo and Moyo [41] in *Opuntia ficus* cladodes, which have a similar composition as that of the prickly pear peel. On the contrary, FD orange and HA mango DFC exhibited the highest carbohydrate content (92.2 and 91.9 g·100 g<sup>-1</sup> db, resp.), while the FD prickly pear peel sample showed 70.7 g·100 g<sup>-1</sup> db.

Regarding TDF content (Table 3), all concentrates showed values ranging from 40.0 to 54.8 g·100 g<sup>-1</sup> db (FD prickly pear and FD mango peel DFC, resp.), indicating that the concentrates studied might be considered as a source of dietary fibre. Similar TDF contents have been reported in several DFC from fruit by-products (obtained similarly to those studied in this research): for example, pineapple peel (45.2 g·100 g<sup>-1</sup> db) [42], pomegranate pomace (56.3 g·100 g<sup>-1</sup> db) [43], orange peel (49.2 g·100 g<sup>-1</sup> db) [44], grapefruit peel (44.2 g·100 g<sup>-1</sup> db) [5],

melon peel (50.2 g·100 g<sup>-1</sup> db) [45], mango peel (51.2 g·100 g<sup>-1</sup> db) [46], prickly pear peel (38.1 g·100 g<sup>-1</sup> db) [44], and red grape peel (53.2 g·100 g<sup>-1</sup> db) [47], among others. Analyzing the effect of dehydration processes, the TDF, IDF, and SDF (as the sum of SDFP and SDFS) contents in FD and HA mango samples showed no significant differences ( $p < 0.05$ ). Even a slight decrease in SDFS content was observed for the HA concentrate (0.8 g·100 g<sup>-1</sup> db), compared to that of the FD concentrate (2.2 g·100 g<sup>-1</sup> db); no significant effect on the SDF value was observed.

A lower TDF content in orange peel DFC was observed in HA samples (49.2 g·100 g<sup>-1</sup> db) when compared to FD samples (54.7 g·100 g<sup>-1</sup> db). The TDF reduction resulted from a decrease in the IDF and SDF values, both because of the use of the thermal process in the HA process. The same observation was reported by Vega-Gálvez et al. [48] in Cape gooseberry, where the TDF content decreases from 53.5 g·100 g<sup>-1</sup> db in fresh fruit to 37.0 g·100 g<sup>-1</sup> db after convection drying at 60°C, resulted by a decrease in the content of both IDF and SDF. As depicted in Table 3, the IDF content of HA orange peel DFC was lower than that of the FD sample. The main components of orange peel IDF are insoluble pectins [44, 49], which could be modified by de-esterification, via pectin-methyl-esterase enzyme's activity promoted at mild temperatures [50], and potentially turned into digestible carbohydrates. Regarding SDF fraction, HA orange peel showed lower content compared to that of the FD sample, mainly by the reduction of the low-molecular weight fraction, since SDFP fraction remained similar among treatments. This reduction in the SDFS content in

orange peel is due to the effect of the temperature used in HA, since SDFS is composed of FOS, particularly 1-kestose and 1<sup>F</sup>- $\beta$ -fructofuranosylmaltose [44, 51], and both compounds are highly hydrolyzable with the use of temperature [52]. On the contrary, a significantly higher TDF content was observed in HA prickly pear DFC (49.2 g·100 g<sup>-1</sup> db) when compared to FD samples (40.0 g·100 g<sup>-1</sup> db), due to an increment in both IDF and SDFS. The decrease in the fibre content of orange DFC and the increase in that of the prickly pear samples produced by the HA treatment were also observed by Tejada-Ortigoza et al. [8] even with different treatments, such as high hydrostatic pressure. However, more research is needed to understand the effect of temperature on the increase of the DF fractions and carbohydrate modification of prickly pear peel.

**3.2. Physicochemical Properties of DFC.** The results obtained for physicochemical properties are shown in Table 4. SOL of FD and HA concentrates ranged from 34.4 to 53.9% (FD orange peel and FD prickly pear peel concentrates, resp.). SOL in FD mango and prickly pear samples was higher than the one obtained by HA. On the contrary, HA orange peel was more soluble than the FD concentrate. Similar to these, last results were obtained by Que et al. [53], where HA pumpkin powder showed a higher solubility than the FD sample (34.9 and 30.7%, resp.). A negative correlation ( $R^2 = 0.67$ ) between SOL and IDF content of the dehydrated samples was observed (Figure 1(a)). IDF is the fibre fraction that is not soluble in water; therefore, samples with higher original IDF content or increased by the dehydration process exhibited lower SOL. This behaviour is comparable to that reported by Huang and Ma [54], where a reduction in the IDF content of orange pomace from 46.5 to 33.6% (db) increased SOL from 2.8 to 30.6%.

On the contrary, SC values ranging from 6.5 to 11.2 mL·g<sup>-1</sup> (db) were achieved (HA mango and FD prickly pear concentrates, resp.). Similar SC values (from 4.6 to 7.2 mL·g<sup>-1</sup>) were also observed by Martínez et al. [55] in mango, passion fruit, and pineapple DFC and by Figuerola et al. [5] in apple and orange peel concentrates (from 6.1 to 9.2 mL·g<sup>-1</sup>). Higher SC values were obtained in FD prickly pear and mango peel DFC than in HA samples. Nevertheless, in the case of orange peel HA samples, the SC values were significantly higher ( $p < 0.05$ ) than FD concentrates (10.6 and 8.1 mL·g<sup>-1</sup>, resp.). The TDF content of the studied samples seems to have an influence on the decrease of the SC, as it is shown in Figure 1(b) ( $R^2 = 0.61$ ). Comparable behaviour was observed in the results published by O'Shea et al. [56] in orange and apple pomace concentrates, where orange pomace with a TDF content of 40.5 showed an SC of 8.1 mL·g<sup>-1</sup>, whereas apple pomace concentrates with a TDF content of 30.2 g·100 g<sup>-1</sup> exhibited an SC of 12.8 mL·g<sup>-1</sup>.

The studied DFC showed a low capacity to bind water (from 3.2 to 4.4 mL·g<sup>-1</sup>), as it is shown in Table 4, compared to other studies. López-Marcos et al. [43] obtained DFC from lemon, grapefruit, pomegranate, and tiger nut bagasse with WRC up to 8.0 mL·g<sup>-1</sup>. The low WRC values reached in the present research could be due to the DFC particle size

(up to 425  $\mu$ m). Chantaro et al. [57] reported that carrot peel DFC with a particle size of 150–250  $\mu$ m exhibited higher WRC than the one with a particle size of 300–450  $\mu$ m. Raghavendra et al. [58] proved that both WRC and ORC values in coconut residue DFC were higher at the determined particle size and reduced their value at higher and lower particle sizes. They proved that the particle size to obtain the maximum WRC/ORC value depends on the microstructure. When comparing WRC between drying methods, minor but significant differences ( $p < 0.05$ ) were observed. HA samples resulted in lower values than the FD samples in the evaluated fruit peels. A correlation between WRC and SDFS is shown in Figure 1(c) ( $R^2 = 0.70$ ). On the contrary, ORC values ranged from 1.7 to 2.6 mL·g<sup>-1</sup>. No significant differences ( $p < 0.05$ ), related with the effect of the drying method applied, were found in mango DFC. ORC in HA orange peel concentrates was higher than the one obtained by FD (2.5 and 1.8 mL·g<sup>-1</sup>, resp.), contrary to prickly pear DFC samples where the HA sample value (1.8 mL·g<sup>-1</sup>) was lower when compared to the FD sample (2.6 mL·g<sup>-1</sup>). This result agrees with Que et al. [53], who reported a higher ORC value in FD pumpkin flour than in HA samples (2.4 and 1.1 mL·g<sup>-1</sup>, resp.). The amount of TDF seemed to be closely related to the ORC of the studied samples, since a negative correlation ( $R^2 = 0.41$ ) was found, as it is shown in Figure 1(d). Similar behaviour was observed in the results reported by de Moraes Crizel et al. [42], where olive residue DFC, which had the highest TDF content (53.7 g·100 g<sup>-1</sup> db), showed the lowest ORC (2.6 g·g<sup>-1</sup>) when compared to pineapple, papaya, and blueberry residue DFC. Regarding pH, all the comparisons between drying treatments showed significant differences ( $p < 0.05$ ). Mango samples presented lower pH values (4.14 and 4.00 for FD and HA, resp.) than those observed in orange (4.99 and 5.29 for FD and HA, resp.) and prickly pear (5.08 and 5.28 for FD and HA, resp.) powders. pH values could be explained by the high pectin content in the fruit peels. However, there is not a clear relation between the changes in the IDF or SDF composition with the increment or decrement of pH values obtained from the DFC dehydrated by both FD and HA.

Finally, TD values ranged from 354 to 804 mg·mL<sup>-1</sup>; similar density values were reported by Basanta et al. [59] in cherry residue DFC from three different varieties. Higher TD values were found in FD orange and mango peel concentrates (557 and 804 mg·mL<sup>-1</sup>, resp.) when compared to the HA samples (449 and 724 mg·mL<sup>-1</sup>, resp.). However, the prickly pear concentrate TD value observed in HA samples was larger than the one observed in FD samples (628 and 354 mg·mL<sup>-1</sup>, resp.), indicating a most compactable material. Nevertheless, despite the correlations shown in Figure 1, most of the physicochemical properties studied could also depend on the porosity and structure of the fibres produced by the drying process, whereby morphological observations of the fibre structures should be conducted for further studies.

**3.3. SCFA Production during In Vitro Fermentation of DFC.** The total SCFA production of DFC is given in Table 5. Both orange samples, FD and HA, showed higher SCFA content

TABLE 4: Physicochemical properties of dietary fibre concentrates from orange, mango, and prickly pear peels freeze-dried (FD) and convective hot air-dried (HA).

	SOL (%)	SC (mL·g <sup>-1</sup> )	WRC (mL·g <sup>-1</sup> )	ORC (mL·g <sup>-1</sup> )	pH	TD (mg·mL <sup>-1</sup> )
<i>Orange</i>						
FD	34.4 ± 0.4 <sup>b</sup>	8.1 ± 0.2 <sup>b</sup>	3.9 ± 0.1 <sup>a</sup>	1.8 ± 0.1 <sup>b</sup>	4.99 ± 0.01 <sup>b</sup>	557 ± 8 <sup>a</sup>
HA	44.7 ± 1.0 <sup>a</sup>	10.6 ± 0.3 <sup>a</sup>	3.4 ± 0.1 <sup>b</sup>	2.5 ± 0.1 <sup>a</sup>	5.29 ± 0.01 <sup>a</sup>	449 ± 10 <sup>b</sup>
<i>Mango</i>						
FD	51.1 ± 1.6 <sup>a</sup>	8.9 ± 0.2 <sup>a</sup>	3.7 ± 0.1 <sup>a</sup>	1.7 ± 0.0 <sup>a</sup>	4.14 ± 0.01 <sup>a</sup>	804 ± 5 <sup>a</sup>
HA	47.1 ± 0.7 <sup>b</sup>	6.5 ± 0.2 <sup>b</sup>	3.2 ± 0.0 <sup>b</sup>	1.7 ± 0.0 <sup>a</sup>	4.00 ± 0.01 <sup>b</sup>	724 ± 14 <sup>b</sup>
<i>Prickly pear</i>						
FD	53.9 ± 1.2 <sup>a</sup>	11.2 ± 0.2 <sup>a</sup>	4.4 ± 0.1 <sup>a</sup>	2.6 ± 0.0 <sup>a</sup>	5.08 ± 0.09 <sup>b</sup>	354 ± 4 <sup>b</sup>
HA	47.8 ± 1.4 <sup>b</sup>	10.2 ± 0.4 <sup>b</sup>	3.7 ± 0.2 <sup>b</sup>	1.8 ± 0.1 <sup>b</sup>	5.28 ± 0.02 <sup>a</sup>	628 ± 16 <sup>a</sup>

Values are mean of four determinations ± SD. Values per fruit (freeze-dried or convective hot air-dried) within the same column followed by different letters are significantly different ( $p < 0.05$ ). SOL: solubility; SC: swelling capacity; WRC: water retention capacity; ORC: oil retention capacity; TD: tapping density. All physicochemical properties were calculated with dry basis weights.

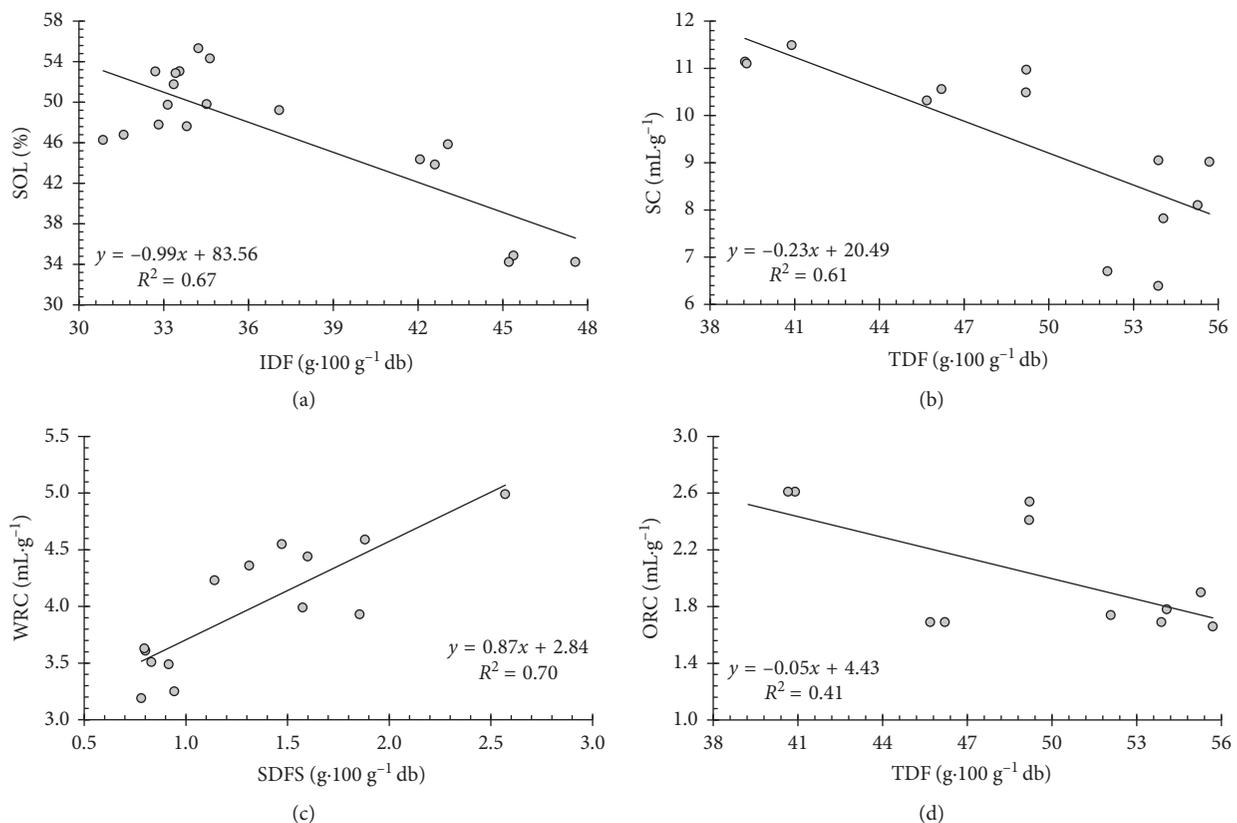


FIGURE 1: Correlation among physicochemical properties and different dietary fibre fractions: (a) solubility (SOL) and IDF, (b) swelling capacity (SC) and TDF, (c) water retention capacity (WRC) and SDFS, and (d) oil retention capacity (ORC) and TDF of DFC from fruit peels obtained by freeze-drying and convective hot air-drying. TDF: total dietary fibre; IDF: insoluble dietary fibre; SDFS: low-molecular weight soluble dietary fibre.

than mango and prickly pear samples at all fermentation times; in fact, SCFA content was even higher than FOS after 6 h fermentation. HA orange peel DFC produced significantly ( $p < 0.05$ ) more SCFA (39.6 mmol·g<sup>-1</sup>) than FD orange samples (34.8 mmol·g<sup>-1</sup>) after 24 h fermentation. For their part, FD mango and prickly pear DFC produced higher SCFA at 24 h than the HA samples. The principal organic acid produced by the fermentation of the DFC was acetate (Figure 2), with more than 70% of the total SCFA composition. The high acetate production during the *in vitro*

fermentation is explained by the large amount of pectin and/or mucilage, present in the studied fruit peels, which are mainly formed by galacturonic acids, and the presence of uronic acids is involved in the production of acetate, while propionate is primarily produced from glucose, xylose, and arabinose, and butyrate mostly from xylose [60].

Orange samples produced higher acetate concentrations than mango and prickly pear concentrates (Figure 2(a)). Significant higher acetate production ( $p < 0.05$ ) was observed in HA orange DFC after 24 h fermentation (30 mmol·g<sup>-1</sup>)

TABLE 5: Total SCFA production ( $\text{mmol}\cdot\text{g}^{-1}\text{ db}$ ) of DFC from orange, mango, and prickly pear peels freeze-dried (FD) and convective hot air-dried (HA), during *in vitro* faecal fermentation.

Fermentation time	Orange		Mango		Prickly pear		FOS
	FD	HA	FD	HA	FD	HA	
6 h	$13.6 \pm 0.5^a$	$15.9 \pm 0.5^b$	$4.3 \pm 0.3^a$	$4.4 \pm 0.2^a$	$10.5 \pm 0.2^a$	$5.7 \pm 0.2^b$	$5.1 \pm 0.3$
12 h	$31.7 \pm 0.6^a$	$31.7 \pm 0.7^a$	$13.0 \pm 0.4^a$	$13.0 \pm 0.3^a$	$21.0 \pm 1.7^a$	$15.1 \pm 0.4^b$	$35.1 \pm 1.1$
24 h	$34.8 \pm 0.3^b$	$39.6 \pm 0.5^a$	$18.3 \pm 0.6^a$	$16.8 \pm 0.2^b$	$28.2 \pm 0.7^a$	$26.3 \pm 1.0^a$	$49.6 \pm 0.9$

Values are mean of three determinations  $\pm$  SD. Values per fruit (freeze-dried or convective hot air-dried) within the same row followed by different letters are significantly different ( $p < 0.05$ ). FOS were used as a positive control.

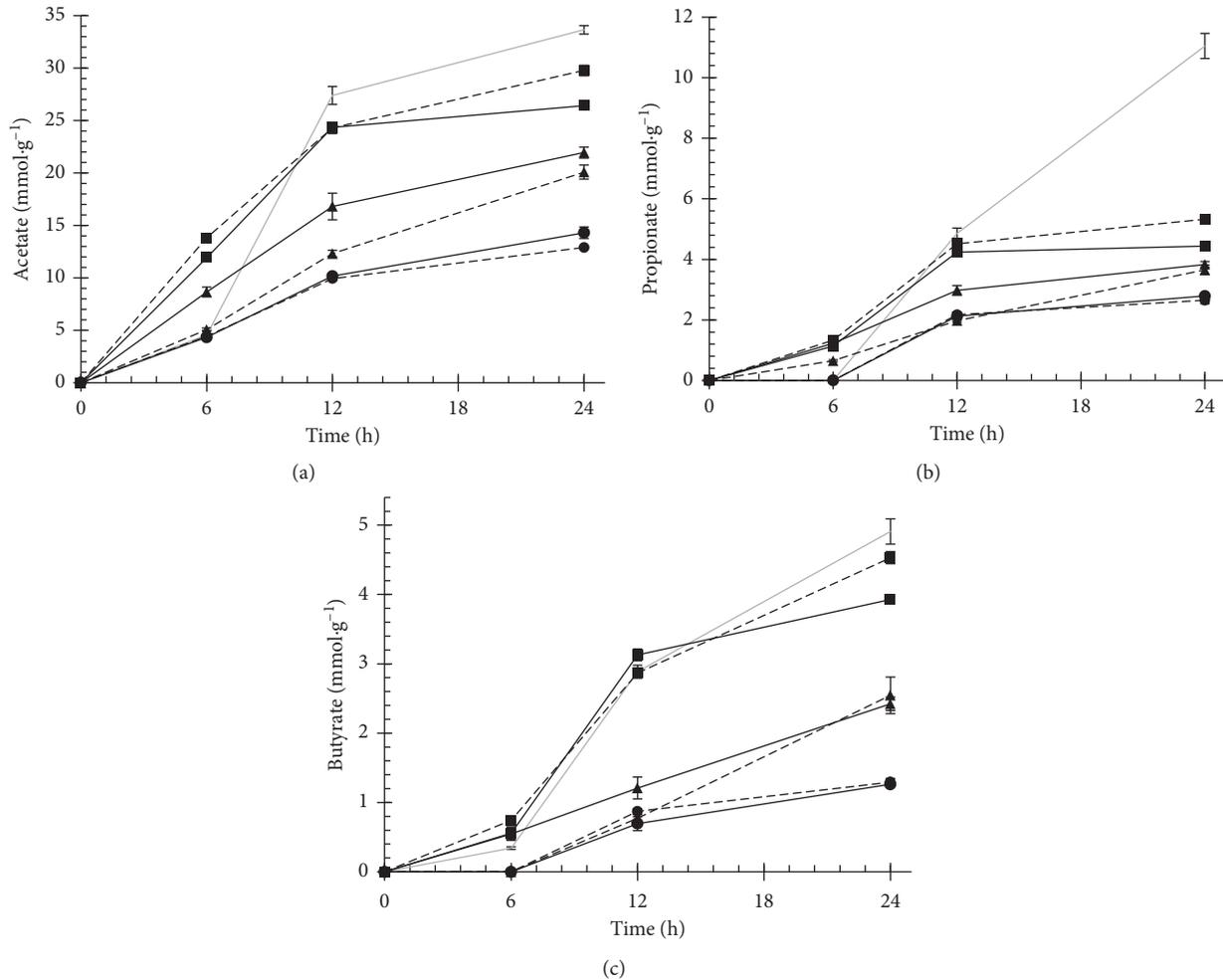


FIGURE 2: Acetate (a), propionate (b), and butyrate (c) production after 24 h of *in vitro* faecal fermentation of orange (■), mango (●), and prickly pear (▲) peel concentrates obtained by freeze-drying (—) and convective hot air-drying (---). Fermentation of FOS (grey line) was used as a positive control.

than that obtained from FD orange samples ( $26 \text{ mmol}\cdot\text{g}^{-1}$ ); however, at the first 6 h, HA orange peel samples produced  $13.8 \text{ mmol}\cdot\text{g}^{-1}$ , compared to  $11.9 \text{ mmol}\cdot\text{g}^{-1}$  obtained from FD samples. Mango peel samples showed the lowest values at the three fermentation times, and significant differences ( $p < 0.05$ ) between FD and HA were only observed after 24 h ( $14.3$  and  $12.9 \text{ mmol}\cdot\text{g}^{-1}$ , resp.). The production of acetate in FD prickly pear was significantly higher at all the evaluated times than that of HA samples.

Propionate production (Figure 2(b)) was the highest in orange ( $4.4$  and  $5.3 \text{ mmol}\cdot\text{g}^{-1}$  for FD and HA, resp., at 24 h)

and the lowest in mango peel samples ( $2.8$  and  $2.6 \text{ mmol}\cdot\text{g}^{-1}$  for FD and HA, resp., at 24 h). Significant differences ( $p < 0.05$ ) were observed due to the drying methods applied in orange, where the HA samples produced more propionate; however, both dried mango samples produced similar propionate concentrations. Prickly pear DFC samples produced significantly more propionate than HA samples after 6 and 12 h.

Regarding butyrate production (Figure 2(c)), orange samples produced  $3.9$  and  $4.5 \text{ mmol}\cdot\text{g}^{-1}$  for FD and HA treatments, respectively. Butyrate produced by HA samples

was significantly higher than that produced by FD. Besides, no significant differences ( $p < 0.05$ ) were found among these values and the value produced by FOS fermentation ( $4.9 \text{ mmol}\cdot\text{g}^{-1}$ ); therefore, HA favoured butyrate production in this fruit DFC. No differences were observed between drying methods in mango peel samples, which reached the maximum value after 24 h of  $1.3 \text{ mmol}\cdot\text{g}^{-1}$ . FD prickly pear samples produced higher values than HA after 12 h, but no significant differences ( $p < 0.05$ ) were observed at 24 h. Butyrate is a desirable end-fermentation product because it is the principal energy source of the colon epithelial cells, and it has antineoplastic properties, which may prevent colorectal cancer [61, 62]. Thus, a high butyrate production, such as in the orange peel DFC case, is of special attention. On the contrary, no propionate and butyrate production was observed in mango peel samples (FD and HA) and FOS after 6 h of fermentation, indicating that high SDF content resulted in a slow fermentation rate, despite the fact that SDF is considered as easy to be consumed by colonic bacteria [63].

It is important to remark that the final production of SCFA of all the samples after 24 h negatively correlates to the SDF:TDF ratio shown in Table 3 ( $R^2 = 0.87$ ; correlation plots are not shown), independently if the SDF:TDF value depends on the fruit or if it is produced by the drying method. High correlation values were also observed, not only between SDF:TDF and acetate production ( $R^2 = 0.89$ ), which is the main organic acid that constitutes the total SCFA value, but also with propionate ( $R^2 = 0.82$ ) and butyrate ( $R^2 = 0.77$ ) contents. Although it is commonly accepted that there is a relationship between SDF and the fermentation produced by the colonic microbiota [64], our results show that a high SDF content inhibited the production of SCFA in *in vitro* fermentation. A possible explanation is that the easily metabolized SDF could already be used in different metabolic pathways by the probiotic bacteria. This is also supported by the lack of propionate and butyrate produced by mango DFC (SDF:TDF  $\approx 0.40$ ) after 6 h. In this regard, further studies on the microbial population and substrate assimilation must be conducted. On the contrary, increases in the IDF content resulted in higher organic acid production. IDF digestion by the colonic bacteria is highly influenced by the accessibility of the microbial enzymes to the fibre [63]; therefore, the prebiotic potential of the DFC depends not only on the composition but also on the physicochemical and morphological properties of the IDF produced during the dehydration process. Finally, a very low correlation was found between the production of total SCFA and TDF content ( $R^2 = 0.04$ ). Therefore, it is not only the fibre content in the concentrates what promotes the production of organic acids but also the dietary fibre composition and SDF:TDF ratio.

#### 4. Conclusions

FD and HA were successfully used to obtain DFC from orange, mango, and prickly pear peels, with high dietary fibre content. The method used to dehydrate mango peels did not modify the fibre content. However, IDF and SDF in

prickly pear sample composition considerably increased with HA ( $6.7$  and  $7.2 \text{ g}\cdot 100 \text{ g}^{-1}$  db, resp.) when compared to FD samples, whereas IDF and SDF in orange peel DFC were decreased by HA ( $3.5$  and  $1.1 \text{ g}\cdot 100 \text{ g}^{-1}$  db, resp.). SOL and SC were the most affected physicochemical properties by the drying treatments, where higher values were obtained for orange dried with HA, while FD improved these properties in mango and prickly pear. All the DFC studied used as a carbon source in *in vitro* faecal fermentation produced an acceptable SCFA content, when compared to the organic acids produced by FOS. HA orange DFC produced more SCFA ( $48 \text{ mmol}\cdot\text{g}^{-1}$  higher) than the FD sample, while FD enhanced organic acid production in mango and prickly pear samples. However, it was observed that the lower SDF:TDF ratio by either the specific content of the fruit or the content modified by the drying method favoured the SCFA production, and differently of what has been previously reported, in these systems, an increase in IDF improves SCFA production. The studied peel DFC have similar physicochemical and functional properties. However, from the point of view of the SCFA production in *in vitro* fermentation, as a prebiotic potential indicator, the orange DFC dried by HA is the most suitable, whereas mango peel obtained by the same drying process is the sample that showed the lowest prebiotic properties.

#### Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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