LWT - Food Science and Technology 124 (2020) 109155

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

Effects of prickly pear (*Opuntia ficus-indica* L.) peel flour as an innovative ingredient in biscuits formulation

Souhir Bouazizi[a], Giuseppe Montevecchi[b,*], Andrea Antonelli[b], Moktar Hamdi[a]

[a] *Laboratory of Microbial Ecology and Technology, Department of Biological and Chemical Engineering, National Institute of Applied Sciences and Technology (INSAT), Centre Urbain Nord, Boulevard de la Terre2, B.P.676, 1080, Tunis, Tunisia*

[b] *Department of Life Sciences (Agro-Food Science Area), BIOGEST - SITEIA Interdepartmental Centre, University of Modena and Reggio Emilia, Piazzale Europa 1, 42124, Reggio Emilia, Italy*

A R T I C L E I N F O

Keywords:
Fruit by-products
Novel biscuit
Food acceptability
Betalains
Antioxidant activity

A B S T R A C T

The peels of prickly pears represent around half of the fruit and are generally discarded, thus becoming an environmental problem. Due to the high content of bioactive compounds, prickly pear peels could be conveniently used as a nutraceutical and functional ingredient in some food preparations, such as bakery products. This study was aimed at assessing the aptitude of prickly pear peel flour to be mixed (10 g, 20 g, and 30 g/100 g) with wheat flour for biscuits preparation through the analysis of the physical and chemical properties of doughs and biscuits and through sensory evaluation.

The composition of prickly pear flour showed a significantly higher concentration of fibre (20.70 g/100 g d.w.), ash (14.57 g/100 g d.w.), and phenolic compounds (2776 mg/100 g d.w.) compared to the control wheat flour, thus improving technological properties such as the aptitude to kneading, the flavour retention, and the antioxidant capacity. The acceptance sensory test showed that biscuits prepared with 20 g/100 g and 30 g/100 g of prickly pear flour were more appreciated for smell, taste, colour, and overall acceptability.

1. Introduction

The prickly pear (PP) cactus (*Opuntia ficus-indica* L. Mill, 1768) is widespread in semi-arid areas. Mexico is the main worldwide producer, while the second most important production countries are located in the Mediterranean basin (Lahsasni, Kouhila, Vighon, & Mahrouz, 2004). The average worldwide yield of PP is about 1 million tons per year, which are mainly consumed as fresh fruit (Inglese, Basile, & Schirra, 2002). In Tunisia, this fruit crop has been domesticated since the 17th century and currently more than 600,000 ha are cultivated (Nefzaoui & Ben Salem, 2006).

PP fruits can be consumed as fresh fruit but can also be used in the manufacturing of juices, alcoholic beverages, and confectionery specialities (FAO et al., 2017), while seeds are utilised for oil extraction (Ennouri, Bourret, Mondolot, & Attia, 2005). However, only the pulps and the seeds are widely used, while the thick peels that represent around 50 g/100 g of the raw material are considered agro-waste (Diaz-Vela, Totosaus, Cruz-Guerrero, & de Lourdes Pérez-Chabela, 2013). Their correct disposal usually represents an environmental problem, which is only partially overcome using them as feed and composting (Larrauri, 1999).

PP peels are promising fruit by-products due to their high content of bioactive compounds and, more in general, for their composition (Ramadan & Mörsel, 2003). The presence of dietary fibre, proteins and antioxidant compounds renders PP peel an interesting ingredient for bakery products.

Several studies describe the use of fruit peels for biscuit production (Ajila, Leelavathi, & Prasada Rao, 2008; Boubaker, El Omri, Blecker, & Bouzouita, 2016; Jeddou et al., 2017), as well as cactus cladodes flour as a source of dietary fibre in bakery products (Ayadi, Abdelmaksoud, Ennouri, & Attia, 2009), though no studies have yet been made on the use of PP peels. The present study was aimed at evaluating the PP peel flour incorporation with wheat flour as regards the performance of physical and chemical properties and the sensory characteristics of doughs and biscuits.

2. Materials and methods

2.1. Plant material

Fully-ripened orange PP fruits (100-kg) were collected from Zelfen (Kasserine, Tunisia), in September 2017 (14.32 °Bx, 6.1 pH, 0.03 g_{citric}

* Corresponding author.
E-mail address: giuseppe.montevecchi@unimore.it (G. Montevecchi).

https://doi.org/10.1016/j.lwt.2020.109155
Received 14 March 2019; Received in revised form 10 February 2020; Accepted 13 February 2020
Available online 18 February 2020
0023-6438/ © 2020 Elsevier Ltd. All rights reserved.

$_{acid}$/100g f.w. titratable acidity, and 370 g/kg dry matter). After thorn removal with water, peels were manually separated from the pulps using a knife. Samples were stored into plastic bags (500 g each) and frozen at −20 °C until further processing.

2.2. Preparation of prickly pear peel flour (PPF)

Defrosted peels were cut into smaller pieces, soaked in water to eliminate the residual juice and left to stand in sodium hypochlorite solution (40 mg/L, 30 min) to reduce the microbial flora (Galla, Pamidighantam, Karakala, Gurusiddaiah, & Akula, 2017). Subsequently, the gross material was oven-dried (45 °C for 48 h; Electrothermal Blast Drying Oven WLG-45B, Tianjin, China) up to 9.0 g/100 g d.w. Dried peel pieces were milled through a grinder (AR1100, Moulinex, Paris, France) and the product was sifted using two sieves in series (400 μm and 100 μm mesh). The PPF flour (yield 10.8 g dried weight/100 g fresh weight) was stored in polyethylene bags at 4 °C for further uses.

2.3. Proximate chemical composition of the flours

PPF proximate composition (moisture, ash, fat, proteins, and carbohydrates) was determined in triplicate, according to the standard AOAC methods (2000). The crude fibre was evaluated through the AACC method 32–10.01 (2000). The carbohydrate content was calculated by difference, while the energy value was determined using the Atwater & Bryant method as the following equation (Atwater, Benedict, Bryant, & Smith, 1899):

Energy (kJ/100 g) = (4 × g/100 g protein +3.75 × g/100 g carbohydrate + 9 × g/100 g) × 4.1868

Glucose and fructose were determined through a HPLC system (Agilent Technologies, Santa Clara, USA), equipped with a refractive index detector (Montevecchi, Masino, Di Pascale, Vasile Simone, & Antonelli, 2017), with minor modifications.

2.4. Determination of total phenolic content, antioxidant capacity, carotenoids, and betalains

The total phenolic content using Folin-Ciocâlteu reagent was carried out with the method described by Swain and Hillis (1959). Results were expressed as mg of gallic acid (GAE) equivalent per 100 g of dry weight.

Free radical scavenging activity (RSA) was used to measure the antioxidant capacity using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay method at 517 nm (Brand-Williams, Cuvelier, & Berset, 1995). The free RSA was expressed as mmol/g eq. Trolox and as inhibition percentage.

As for carotenoids determination, 1 g of each flour was homogenised with 10 mL of a mixture of hexane/acetone/ethanol (5.0 mL:2.5 mL:2.5 mL) before being centrifuged for 5 min at 6956 g at 4 °C. Total carotenoids determination was carried out by measuring the absorbance at 450 nm and using the extinction coefficient of β-carotene (E1% = 2505), specific for the solvent of extraction (Rodriguez-Amaya, 1999, p. 71). Betalains content was calculated according to Castellanos-Santiago and Yahia (2008). The molar extinction coefficients (ε) in water for betacyanins and betaxanthins were 60,000 Absorbance/cm × mol (λ_{max} 535 nm) and 48,000 Absorbance/cm × mol (λ_{max} 483 nm), respectively. Total betalains were calculated as a sum of betacyanins and betaxanthins.

2.5. Functional properties of flours

The ability of flours to bind with water (WAC) and oil (OAC) in specific conditions was determined in the same way described by Kaur, Sandhu, Arora, and Sharma (2015). WAC and OAC were calculated as g of water and oil bound per g of the sample on a dry basis.

The water and oil holding capacity (WHC and OHC, respectively) were evaluated using the method described by Robertson et al. (2000), while bulk density (BD) was calculated as the weight of each sample per volume unit (g/mL) (Kaur et al., 2015). Least gelation concentration (LGC) was evaluated using the method described by Chandra, Singh, and Kumari (2015). Foaming capacity (FC) and foam stability (FS) of flours were tested using the method described by Chandra et al. (2015).

2.6. Alveographic measurements

The alveographic parameters, such as dough tenacity (P), dough extensibility (L), index of swelling (G), dough baking strength (W), configuration of the curve (P/L) were evaluated through a Chopin alveograph MA82 (Tripette et Renaud, Villeneuve-la-Garenne, France), according to the official method (AACC method 54–30.02, 2000), using only flour and water.

2.7. Preparation of biscuits

Biscuits were prepared with the following ingredients: flour (adjusted without altering the total flour content of the recipe), sugar, margarine, baking powder, and an egg. The formulations are outlined in Table 1. Aliquots of 22.5, 45.0, and 67.5 g of PPF were used to prepare samples with 10, 20, and 30 g PPF/100 g, respectively.

Fat and sugar were creamed in a mixer (KM245, Kenwood, Havant, U.K.) using a flat beater for 2 min at slow speed, then the egg was thoroughly added. After 15 min of rest at 4 °C, the dough was reduced to a thickness of 4 mm with the help of a rolling pin and an aluminium foil of a standard height. A circular mould of 50 mm diameter was used to cut the dough. The biscuits were baked at 170 °C for 15 min in an electric convection baking oven (BRIO 43S/X-N, Diamond, Brussels, Belgium). After baking, all the samples were cooled and stored in an airtight plastic bag covered with aluminium foil at room temperature for further analysis.

2.7.1. Rheological properties of the dough used for biscuits production

The texture profile analysis in penetration mode was used for dough used for biscuits production as described by Raymundo, Fradinho, and Nunes (2014). The analysis of hardness and cohesiveness properties were carried out at 20 °C in a temperature-controlled room and an average of five replicates of each dough sample were recorded.

2.7.2. Chemical (proximate composition, phenolics, antioxidant capacity, carotenoids, and betalains) and physical (colour) analysis of biscuits

Biscuit samples were milled with a grinder (AR1100, Moulinex, Paris, France) until obtaining a homogenous and fine powder that was subsequently sieved. Biscuits moisture, total ash, crude fat, total protein content, crude fibre, carbohydrate content, and energy were calculated according to the methods described for the dough.

Table 1
Biscuits formulation with different amount of prickly pear peel flour (PPF).

Ingredients (g)	Level of wheat flour substitution			
	Control (WF)	PPF/WF		
		10 g/100 g	20 g/100 g	30 g/100 g
Wheat Flour (WF)	225	202.5	180	157.5
PPF	0	22.5	45	67.5
Margarine	65	65	65	65
Baking powder	3.5	3.5	3.5	3.5
Number of eggs (≈ 57 g each)	1	1	1	1
Sugar	65	65	65	65

Biscuits total phenolic content, antioxidant capacity, carotenoids, and betalains were calculated in triplicate according to the methods described for flours using 1.0 g of a finely-powdered sample of biscuits. Measurements were made in triplicate.

The surface colour was measured through a tristimulus reflectance colorimeter (Chroma Meter CR-400; Konica Minolta, Milan, Italy) set on the CIE standard illuminant D65. Brightness, redness and yellowness were assessed by CIE tristimulus coordinates (L*, a*, and b*, respectively) (CIE, 1976). The average of six values was calculated by measuring each sample in different positions.

2.7.3. Diameter, thickness, spread ratio, and texture measurements of biscuits

Diameter, thickness and spread ratio were calculated with the methods described by Yadav, Yadav, and Dhull (2012). The average weight of six biscuits belonging to each group was calculated.

Penetration test was performed for biscuits with the method described by Raymundo et al. (2014). The fracture strength of biscuits was measured using a 3-point bending rig and 5000 g load cell of a texture analyser (TA-XT2i, Stable Micro Systems, Haslemere, U.K.). The distance between the two beams was 38 mm, the pre-test speed was 10.0 mm/s and the test speed was 1.0 mm/s. The maximum peak force (N) was reported as the fracture strength (hardness) and the average value of 5 biscuits was calculated (Yadav et al., 2012).

2.8. Sensory analysis of biscuits

The sensory acceptance was evaluated by a panel of 30 trained judges (consisting of staff members and university students, both males and females, aged from 21 to 56, and usual PP consumers) using a 9-point hedonic scale (with ratings ranging from “extremely dislike” to “extremely like”) (Ranganna, 1979, p. 634). The biscuits were evaluated based on their colour, smell, taste, texture, crispness, after-taste, and overall acceptability.

The biscuits samples tested during the three sensory sessions were randomly selected from each box. Each session took place in a sensory lab where isolated booths had been placed beforehand. The four samples were randomly coded and presented on white plastic dishes which had been divided into four equal parts.

2.9. Statistical analysis

For each parameter, average values and confidence intervals ($\alpha = 0.05$) were calculated from replicated measurements. Significant differences were assessed by analysis of variance (one-way ANOVA) based on replicated measurements. When a significant effect (at least $p \leq 0.05$) was shown, comparative analyses were carried out by the post-hoc Tukey's multiple comparison test. All statistical tests were performed using Statistica version 8.0 software (Stat 180 Soft Inc., Tulsa, USA).

3. Results and discussion

Biscuits were selected as a food matrix due to their global diffusion, long shelf-life and the possibility of being easily exported, while the association of wheat flour with PPF was taken into consideration for the characteristics of the latter, such as its “fruity” and sweet taste, its peculiar composition (less prone to oxidation because of its high content of antioxidant compounds) thus making it suitable to satisfy the demand for healthier.

During the preparation of the samples, three temperatures were tested (30 °C, 45 °C, and 60 °C) for drying. The lowest temperature brought about the spoilage while the highest one produced a massive browning of the material. For these reasons, 45 °C was the chosen temperature, also considering the Tunisian temperatures reached during the harvest season. In addition, the use of lower drying temperatures could imply increased costs. Sodium hypochlorite was selected as a disinfectant and sanitising agent since its use is supported by scientific literature (Gil, Selma, López-Gálvez, & Allende, 2009) and it is currently included in small industries’ procedures.

Table 2
Proximate composition, sugar concentration, total phenolic content, free radical scavenging activity, carotenoids, and betalains of wheat flour (WF) and prickly pear peel flour (PPF).

	Units	WF	PPF
Moisture	(g/100 g f.w.)	14.0 ± 0.2 b	9.11 ± 0.07 a
Ash	(g/100 g f.w.)	0.53 ± 0.01 a	14.57 ± 0.02 b
Crude fat	(g/100 g f.w.)	1.16 ± 0.06 a	2.7 ± 0.2 b
Total protein content	(g/100 g f.w.)	12.6 ± 0.2 b	3.3 ± 0.2 a
Crude fibre	(g/100 g f.w.)	0.44 ± 0.01 a	20.70 ± 0.06 b
Total carbohydrates [a]	(g/100 g f.w.)	71.2 ± 0.4 b	49.6 ± 0.5 a
Energy [b]	(kJ/100 g f.w.)	1374 ± 3 b	937 ± 3 b
Glucose	(g/100 g d.w.)	< LOD a	3.0 ± 0.7 b
Fructose	(g/100 g d.w.)	< LOD a	3.1 ± 0.6 b
Total phenolic content	(mg GAE/100 g d.w.)	106.0 ± 0.1 a	2776.0 ± 0.4 b
Free RSA	(mmol/g eq. Trolox)	8.6 ± 0.2 a	274.7 ± 0.5 b
Free RSA	Ratio	n.i.a. a	82.7 ± 0.2 b
Total carotenoids	(mg CAR/ 100 g d.w.)	n.d. a	10.90 ± 0.04 b
Betacyanins	(mg/100 g d.w.)	n.d. a	336.8 ± 0.4 b
Betaxantins	(mg/100 g d.w.)	n.d. a	250.0 ± 0.7 b
Total betalains [c]	(mg/100 g d.w.)	n.d. a	587 ± 1 b

Values are the means and standard deviation ($n = 3$) obtained by descriptive statistics. One-way ANOVA was applied to the data set and *F values* were all higher than 200 with all $p \leq 0.001$. The results of Tukey's test are reported as lowercase letters, where different letters identify samples that are significantly different ($p \leq 0.05$).
d.w. = dry weight; f.w. = fresh weight; CAR = β-carotene equivalents; GAE = gallic acid equivalents; LOD = limit of detection; n.d. = not detected; n.i.a. = no-inhibition activity; RSA = radical scavenging activity.
[a] Calculated by the difference method.
[b] Calculated by the Atwater & Bryant method.
[c] Calculated as the sum of betacyanins and betaxantins.

3.1. Proximate composition of flours

The chemical composition of PPF is shown in Table 2. ANOVA showed significant differences for all parameters. PPF had a moisture content of 9 g/100 g, which represents the highest limit value for handling and preservation of fibrous materials (Larrauri, 1999). The protein content (3.3 g/100 g f.w.) was similar to the one observed in the mango epicarp flour (3.6 g/100 g d.w.) (Ajila et al., 2008) and lower than the values, which had been observed in other by-products, such as dried cladodes (Msaddak et al., 2015), artichokes (Boubaker et al., 2016), and potato peels (Jeddou et al., 2017).

The most relevant result was given by the high content of ash and fibre provided by PPF. As for ash, cations like magnesium and calcium improve the mechanical characteristics of gluten for interactions with amino acid side groups (Sehn, Nogueira, Almeida, Chang, & Steel, 2015).

3.2. Total phenolic content and free radical scavenging activity, carotenoids, and betalains content of PPF

PPF showed values of total phenolic content (Table 2) more than one order of magnitude higher than WF. The phenolic compounds, carotenoids, and betalains were the main classes of antioxidant compounds responsible for the high value of the free RSA showed by PPF. The antioxidant capacity showed by all these substances exerts an active role in preventing auto-oxidation of fats (Ajila et al., 2008).

In PPF, carotenoids showed a content of 10.90 mg/100 g d.w., while

Table 3
Functional properties, alveographic measurements and rheological properties, and colour (CIE coordinates: L*, a*, and b*) of wheat flour (WF) and prickly pear peel flour (PPF).

	Units	Level of WF substitution						Confidence interval (−95% ÷ +95%)
		Control (WF)	*PPF*	g PPF/g WF			ANOVA (F_{value})	
				10 g/100 g	20 g/100 g	30 g/100 g		
FUNCTIONAL PROPERTIES								
WAC	(g_{water}/g_{sample})	0.63 a	*0.95*	0.81 b	0.83 b	0.90 c	80***	0.72 ÷ 0.86
OAC	(g_{oil}/g_{sample})	0.77 a	*0.84*	0.78 a	0.81 ab	0.83 c	9**	0.78 ÷ 0.81
WHC	(g_{water}/g_{sample})	0.63 a	*2.59*	0.97 b	0.92 b	1.21 c	173***	0.80 ÷ 1.07
OHC	(g_{oil}/g_{sample})	0.78 a	*0.94*	0.82 ab	0.87 bc	0.90 c	11**	0.81 ÷ 0.88
BD	(g/mL)	0.80	*0.68*	0.75	0.74	0.71	n.s.	0.72 ÷ 0.78
LCG	(g/100 mL)	12	*4*	12	10	8		
FC	(g/100 mL)	7.67 c	*1*	3.67 b	4.00 b	2.33 a	63***	3.08 ÷ 5.76
FS		96.6 a	*99.0*	96.5 a	96.2 a	97.7 b	7*	96.2 ÷ 97.2
ALVEOGRAPHIC PROPERTIES (using only flour and water)								
P	(mm)	61.0 a		80.4 b	110.2 c	207.1 d	30604***	95.7 ÷ 174.3
L	(mm)	93.0 c		48.1 b	27.0 a	24.0 a	807***	29.7 ÷ 66.4
P/L	ratio	0.66 a		1.98 b	6.56 c	8.62 d	5293***	2.29 ÷ 6.62
G	(mL)	21.6 c		15.4 b	11.6 a	10.9 a	37***	12.0 ÷ 17.8
W	(10^{-4} J)	160.3 a		163.1 a	225.0 b	226.0 b	1738***	172.4 ÷ 214.8
RHEOLOGICAL PROPERTIES (of the dough used for biscuits production)								
Hardness	(g)	464.5 a		429.0 a	814.3 b	919.7 b	83***	512.8 ÷ 801.0
Cohesiveness	ratio	0.29		0.30	0.29	0.27	n.s.	0.27 ÷ 0.30
COLOR PARAMETERS								
L*		97.3 d	*56.8*	87.3 c	85.4 b	81.4 a	12034***	84.0 ÷ 91.7
a*		−0.79 a	*3.51*	0.22 b	0.71 c	0.92 d	2940***	−0.17 ÷ 0.70
b*		6.70 a	*26.40*	9.80 b	9.40 b	12.00 c	131***	8.20 ÷ 10.72

Functional properties, alveographic measurements and rheological properties, and colour (CIE coordinates: L*, a*, and b*) of wheat flour (WF) and prickly pear peel flour (PPF).
Values are the average values ($n = 3$) obtained by descriptive statistics. The interval of confidence is calculated using a confidence level of 95%. Results of the one-way ANOVA and the Tukey's test are reported as F_{values} and lowercase letters, respectively. Different letters identify samples significantly different ($p \leq 0.05$).* = $p \leq 0.05$; ** = $p \leq 0.01$; *** = $p \leq 0.001$.
WAC = water absorption capacity; OAC = oil absorption capacity; WHC = water holding capacity; OHC = oil holding capacity; BD = bulk density; LGC = least gelation concentration; FC = foaming capacity; FS = foam stability; P = dough tenacity; L = dough extensibility; G = index of swelling; W = dough baking strength; P/L = configuration of the curve.

the concentrations of the main classes of betalains were more than one order of magnitude higher. Although a little amount of carotenoids is usually present in wheat flour, the content was lower than the threshold of detectability. Betalains are present in the pulp and the peel of *Opuntia ficus-indica*. Betacyanins and betaxanthins concentrations vary according to the colour of the fruit, whereas betanin and indicaxanthin are found especially in the peel part (Yeddes, Chérif, Guyot, Sotin, & Ayadi, 2013).

3.3. Effects of PPF on flour properties

3.3.1. Functional properties

WAC and OAC significantly augmented with the increase of the PPF amount (Table 3), similarly to WHC and OHC. The increase of WAC and WHC in PPF was likely due to the higher amount of hydrophilic constituents, such as soluble fibre (Akubor & Badifu, 2004). The OAC and OHC can be connected to the higher PPF fat content. Higher OAC and OHC values improve taste sensory properties and help retain the flavour (Noor, Siti, & Mahmad, 2015).

The FC decreased by increasing the PPF rate, probably as a consequence of the reduction of the protein content that allows the formation of the cohesive interfacial film around the air or oil and water in the mixture system (Kaur, Kaushal, & Sandhu, 2011).

The values shown by 10 g PPF/100 g and 20 g PPF/100 g fell in the specific confidence intervals ($\alpha = 0.05$) while in most cases the control figures, as well as 30 g PPF/100 g ones, lay outside those ranges. This statistical behaviour showed that the linear combinations of ingredients resulting from the use of 10 g PPF/100 g and 20 g PPF/100 g are already sufficiently strong to determine a significant difference in the samples.

3.3.2. Alveographic properties

Data on rheological properties are shown in Table 3. ANOVA showed significant differences ($p \leq 0.001$) for most of the parameters. P gradually increased by increasing PPF percentage, while, at the same time, L decreased. As a consequence of this, the configuration of the alveographic curve changed accordingly and, in turn, P/L increased with the increasing amount of PPF. The dough baking strength, W, significantly increased with 20 g PPF/100 g and 30 g PPF/100 g in comparison with 10 g PPF/100 g and the control. Finally, G decreased by increasing the PPF amount.

Some alveographic values found for PPF are very dissimilar to those related to wheat flour. However, Chopin alveograph was designed and optimised for the rheological evaluation of doughs of wheat flour only. On the other hand, the increase of P, P/L, and W, and the contemporary decrease of L have already been observed on cladode flour (Ayadi et al., 2009).

The absence of a centroid sample in the experimental design was reflected in the evaluation of the values that fell within the confidence interval. Indeed, in the case of P and P/L, the sample consisting of 20 g PPF/100 g was the only one that showed its average values in the confidence intervals, while as for L and G, 10 g PPF/100 g was the only sample. In the case of W, no samples fell within.

3.4. Rheological properties of the dough used for biscuits production

Rheological properties were evaluated on the dough used for biscuits production. The hardness of the dough significantly increased with 20 g PPF/100 g and 30 g PPF/100 g, while the cohesiveness did not show any significant difference among the samples. This behaviour was related to the high fibre content, and therefore to hydroxyl groups,

Table 4
Proximate composition, total phenolic content, free radical scavenging activity, carotenoids, betalains, and colour (CIE coordinates: L*, a*, and b*) of the biscuits obtained with wheat flour (control, WF) and different amount (10 g/100 g, 20 g/100 g, and 30 g/100 g) of prickly pear peel flour (PPF).

	Units	Level of WF substitution					Confidence interval (−95% ÷ +95%)
		Control (WF)	g PPF/g WF			ANOVA (F_{value})	
			10 g/100 g	20 g/100 g	30 g/100 g		
Moisture	(g/100 g f.w.)	9.44 c	8.00 b	7.71 b	7.23 a	206***	7.54 ÷ 8.64
Ash	(g/100 g f.w.)	0.86 a	1.94 b	2.52 c	3.62 d	277***	1.57 ÷ 2.90
Crude fat	(g/100 g f.w.)	16.4 c	16.3 c	15.7 b	15.3 a	n.s.	15.6 ÷ 16.2
Total protein content	(g/100 g f.w.)	8.98 b	9.15 b	8.75 ab	8.29 a	12**	8.56 ÷ 9.03
Crude fibre	(g/100 g f.w.)	0.77 a	2.12 b	2.45 c	3.14 d	10646***	1.55 ÷ 2.69
Total carbohydrate content [a]	(g/100 g f.w.)	63.5 b	62.5 a	62.8 a	62.5 a	6*	62.5 ÷ 63.2
Energy [b]	(kJ/100 g f.w.)	1766 d	1748 c	1726 b	1695 a	2898***	1716 ÷ 1752
Total phenolic content	(mg GAE/100 g d.w.)	34 a	360 b	477 c	575 d	11279***	226 ÷ 497
Free RSA	(mmol/g eq. Trolox)	141 a	198 b	236 c	236 c	56177***	177 ÷ 229
Free RSA		n.i.a. a	25.0 b	55.2 c	56.5 d	55253***	33.7 ÷ 57.4
Total carotenoids	(mg CAR/100 g d.w.)	0.90 a	1.77 b	2.57 c	3.54 d	3782***	1.54 ÷ 2.84
Betacyanins	(mg/100 g d.w.)	n.d. a	29.3 b	57.0 c	106.9 d	268821***	22.2 ÷ 74.4
Betaxantins	(mg/100 g d.w.)	n.d. a	20.4 b	61.7 c	69.8 d	112646***	18.8 ÷ 57.1
Total betalains [c]	(mg/100 g d.w.)	n.d. a	49.7 b	118.7 c	176.7 d	409435***	41.7 ÷ 130.8
COLOUR							
L*		57.8 d	46.4 c	40.9 b	38.2 a	240***	40.8 ÷ 50.8
a*		9.56 b	9.07 b	7.95 a	7.26 a	25***	7.83 ÷ 9.09
b*		26.2 d	25.2 c	23.7 b	22.3 a	124***	23.4 ÷ 25.4

Values are the average values ($n = 3$) obtained by descriptive statistics. The interval of confidence is calculated using a confidence level of 95%. Results of the one-way ANOVA and the Tukey's test are reported as F_{values} and lowercase letters, respectively. Different letters identify samples significantly different ($p \leq 0.05$). * = $p \leq 0.05$; ** = $p \leq 0.01$; *** = $p \leq 0.001$.
d.w. = dry weight; CAR = β-carotene equivalents; GAE = gallic acid equivalents; n.d. = not detected; n.i.a. = no-inhibition activity; RSA = radical scavenging activity.
[a] Calculated by the difference method.
[b] Calculated by the Atwater & Bryant method.
[c] Calculated as the sum of betacyanins and betaxantins.

which establish strong interactions with gluten proteins via water interactions through hydrogen bonds (Rosell, Rojas, & Benedito De Barber, 2001). The absence of hardness values falling within the confidence interval was indicative of a significant difference in the behaviours of samples with 10 g PPF/100 g and 20 g PPF/100 g.

3.5. Proximate composition of biscuits

Statistical treatment showed significant differences among the different compositions of biscuits for all the parameters assessed, except for crude fat (Table 4). 10 g PPF/100 g and 20 g PPF/100 g average values fell in most of cases within the confidence intervals calculated for each parameter. These two samples showed a clear deviation from the control being significantly distant from 30 g PPF/100 g.

The moisture significantly decreased ($p \leq 0.001$) with the increase of the PPF, as did total protein content ($p \leq 0.01$), total carbohydrates ($p \leq 0.05$), and energy ($p \leq 0.001$). However, some constituents significantly increased ($p \leq 0.001$) as the effect of the presence of PPF in the biscuits, in particular ash, crude fibre, and phenolic compounds.

Total phenolic content increased ($p \leq 0.001$) in values of 10.7; 14.7; 17.1 folds together with the increase of PPF in the biscuits, although this content was lower than the concentrations found in the flour, due to the partial thermal degradation of these compounds. Similar increase was shown by the free RSA ($p \leq 0.001$), because of the increase of antioxidant phenolic compounds.

The concentrations of carotenoid and betalain pigments followed the same trend of the total phenolic compounds and antioxidant capacity. Statistical treatment among these parameters confirmed their high positive correlation ($p \leq 0.001$) with r values included between 0.868 and 0.997. Carotenoids and betalains show good resistance to heat treatment so their concentration is not significantly affected during the oven cooking of biscuits.

Other studies have been recently conducted for the preparation of bakery goods using *Opuntia* cladodes (Msaddak et al., 2015), as well as fruit peels from other plants. The presence of pigments and, in turn, their antioxidant activity is one of the major advantages of PPF in comparison with cladodes. Mango peels have a composition similar to PP peels and their use was evaluated for the production of biscuits. However, the content of fibre was higher in mango peels, while, the concentrations of ashes, total phenolic compounds, and carotenoids were much lower and betalains were totally absent (Ajila et al., 2008). Banana presents a thick and meaty peel, however, the amount of different kinds of anti-nutrients is very high (Romelle, Dani, & Manohar, 2016).

3.6. Effect of PPF on the colour of biscuits

ANOVA showed a significant progressive decrease in brightness (L*) and yellowing index b* for all samples by increasing the PPF content, while the redness index a* showed a significant reduction solely in the samples containing 20 g/100 g and 30 g/100 g PPF (Table 4). The evaluation of the confidence intervals confirmed that 10 g PPF/100 g and 20 g PPF/100 g were able to determine a significant difference in the samples.

The most evident effect was the reduction of brightness, as PPF substitution level increased (Fig. 1). This could be the effect of enzymatic browning due to the presence of polyphenols oxidases and non-enzymatic browning due to the higher presence of reducing sugars (Ajila et al., 2008). The reduction of redness and yellowness was a minor effect.

3.7. Effect of PPF on physical and textural properties of biscuits

The average diameter of the biscuits significantly decreased ($p \leq 0.001$) due to the PPF content, whereas the thickness and the spread ratio did not show significant differences (Table 5). The

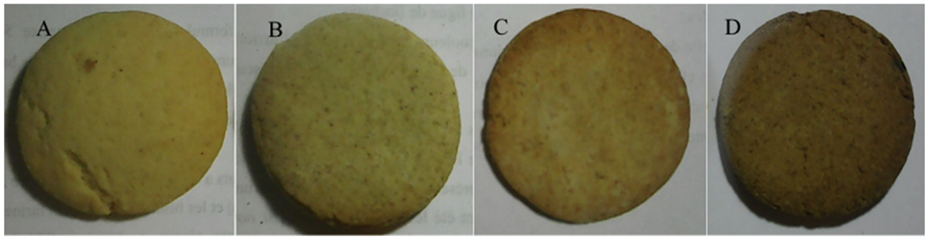

Fig. 1. Control biscuit (A, 54.3 mm diameter) without prickly pear peel flour, and biscuits prepared with 10 g PPF/100 g (B, 52.9 mm diameter), 20 g PPF/100 g (C, 52.3 mm diameter), and 30 g PPF/100 g (D, 51.4 mm diameter) of prickly pear peel flour.

contraction of the diameter was associated with the reduction of the gluten structure with increased levels of wheat flour replacement (Choudhury, Badwaik, Borah, Sit, & Deka, 2015).

The textural parameters analysed through the penetration test were the maximum force associated with the strength of the sample and the area under the curve, which represents the work carried out on the break (Table 5). ANOVA showed significant differences ($p \leq 0.05$) for both of the textural parameters considered. The area under the curve and the maximum force critically rose when 30 g/100 g PPF was used to prepare biscuits. Likewise, only samples with 30 g/100 g PPF showed critical higher values ($p \leq 0.001$) for fracture strength.

Cookie textural properties, which are affected by the water-–starch–protein complex, are the result of the interactions among components of the different flours (Fustier, Castaigne, Turgeon, & Biliaderis, 2008). The progressive reduction of gluten proteins and increase of fibre content did not show significant differences up to 20 g PPF/100 g. Conversely, samples with 30 g PPF/100 g showed a sudden increase in the textural values. Indeed, foods rich in dietary fibre are difficult to chew and take longer to be ingested (McCance, Prior, & Widdowson, 1953). Actually, 50 g PPF/100 g was also used in a preliminary test. The product appeared stiffer and very hygroscopic and, after some hours, it got excessively chewy. 30 g PPF/100 g showed similar properties to a lesser but decisive extent. The combination of egg proteins with peels fibre was, therefore, conducive to obtain a better texture and, in turn, increased acceptability of the novel biscuit formulation (Mridula, 2011).

3.8. Sensory evaluation

All the acceptance descriptors showed significant differences among the samples considered (Fig. 2). Colour, smell, and taste acceptance scores mostly increased with the highest amounts of PPF, probably due to the presence of some reducing sugars in PPF that can undergo non-enzymatic browning reactions, along with a higher aptitude to retain

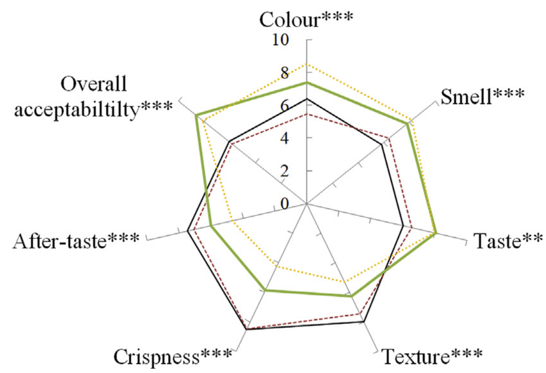

Fig. 2. Radar graph of sensory acceptance analysis of the biscuits obtained with wheat flour (—— control, WF) and different amount (- - - - 10 g PPF/100 g, —— 20 g PPF/100 g, and ······ 30 g PPF/100 g) of prickly pear peel flour (PPF). The results are expressed as mean values of 30 replicates. Results of the one-way ANOVA are reported. ** = $p \leq 0.01$; *** = $p \leq 0.001$.

the smell. Conversely, after-taste, texture, and crispness showed a drastic liking reduction using 20 g/100 g and 30 g/100 g of PPF. However, the biscuits prepared with 20 g/100 g and 30 g/100 g PPF were judged more acceptable in comparison with those containing a lower or no amount of PPF.

4. Conclusions

The use of prickly pear peels as raw materials is of paramount importance both for their interesting composition and in a wider perspective of waste reduction.

The initial hypothesis of using PP peels of partial sugar and gluten replacer was ascertained by biscuit acceptability and textural properties. Aside from the richness of active and functional biomolecules, the presence of fibre and polyphenols favours technological properties such

Table 5
Dimensional measurements and texture analysis of the biscuits obtained with wheat flour (control, WF) and different portions of prickly pear peel flour (PPF): 10 g, 20 g and 30 g/100 g.

	Units	Level of WF substitution					Confidence interval (95% ÷ +95%)
		Control (WF)	g PPF/g WF			ANOVA (F_{value})	
			10 g/100 g	20 g/100 g	30 g/100 g		
Diameter (D)	(mm)	54.3 c	52.9 b	52.3 b	51.4 a	61***	52.0 ÷ 53.4
Thickness (T)	(mm)	1.11	1.10	1.09	1.07	n.s.	1.07 ÷ 1.11
Spread ratio	(D/T)	48.9	48.1	48.2	48.0	n.s.	47.4 ÷ 49.2
PENETRATION TEST							
Area under the curve N	(mm)	1478 a	1224 a	1513 a	2887 b	5*	1359 ÷ 2192
Maximum force	(N)	1375 a	1157 a	1463 a	2564 b	6*	1296 ÷ 1984
HDP 3-POINT BENDING FLEXURAL TEST							
Fracture strength	(g)	1148 a	903 a	829 a	1664 b	12***	978 ÷ 1385

Values are the average values (n = 5) obtained by descriptive statistics. The interval of confidence is calculated using a confidence level of 95%. Results of the one-way ANOVA and the Tukey's test are reported as F_{values} and lowercase letters, respectively. Different letters identify samples significantly different ($p \leq 0.05$). * = $p \leq 0.05$; *** = $p \leq 0.001$.

as the aptitude to kneading, the flavour retention, and the antioxidant capacity.

The partial substitution of wheat flour with PP peel flour to produce biscuits is a promising strategy in the manufacturing of bakery products. The possibility of testing the PP peel flour for cake preparation could be another interesting field of application.

Author contributions section

Souhir Bouazizi: Conceptualization, Methodology, Formal analysis, Investigation, Writing - Original Draft.

Giuseppe Montevecchi: Software, Validation, Data Curation, Writing - Review & Editing.

Andrea Antonelli: Validation, Writing - Review & Editing.

Moktar Hamdi: Conceptualization, Resources, Supervision, Project administration, Funding acquisition.

Acknowledgements

The authors wish to thank Adem Patisserie, Mrs Maroua Hamdi, and Dr Sara Ronconi (English-language reviewer) for their valuable contribution in the present study. The authors gratefully acknowledge the Tunisian Ministry of Higher Education and Scientific Research.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2020.109155.

References

AACC (2000a). *Method 32-10.01 (Crude fiber in flours, feeds, and feedstuffs). American Association of Cereal Chemists approved methods* (10th ed.). St. Paul, USA: AACC. http://methods.aaccnet.org/methods/32-10.pdf.

AACC (2000b). *Method 54-30.02 (Alveograph method for soft and hard wheat flour). American Association of Cereal Chemists approved methods* (10th ed.). St. Paul, USA: AACC. http://methods.aaccnet.org/methods/54-30.pdf.

Ajila, C. M., Leelavathi, K. U. J. S., & Prasada Rao, U. (2008). Improvement of dietary fiber content and antioxidant properties in soft dough biscuits with the incorporation of mango peel powder. *Journal of Cereal Science, 48*(2), 319–326.

Akubor, P. I., & Badifu, G. I. O. (2004). Chemical composition, functional properties and baking potential of African bread fruit kernel and wheat flour blends. *International Journal of Food Science and Technology, 39*, 223–239.

AOAC (2000). *Official methods of analysis of AOAC. International*(17th ed.). Gaithersburg, MD: USA Association of Analytical Communities Methods 920.87; 925.10; 923.03.

Atwater, W. O., Benedict, F., Bryant, A., & Smith, A. (1899). *Experiments on the metabolism of matter and energy in the human body.* Washington, USA: Office of Experiment Stations Bulletin.

Ayadi, M. A., Abdelmaksoud, W., Ennouri, M., & Attia, H. (2009). Cladodes from *Opuntia ficus-indica* as a source of dietary fiber: Effect on dough characteristics and cake making. *Industrial Crops and Products, 30*(1), 40–47.

Boubaker, M., El Omri, A., Blecker, C., & Bouzouita, N. (2016). Fibre concentrate from artichoke (*Cynara scolymus* L.) stem by-products: Characterization and application as a bakery product ingredient. *Food Science and Technology International, 22*(8), 759–768.

Brand-Williams, W., Cuvelier, M. E., & Berset, C. L. W. T. (1995). Use of a free radical method to evaluate antioxidant activity. *LWT-Food Science and Technology, 28*(1), 25–30.

Castellanos-Santiago, E., & Yahia, E. M. (2008). Identification and quantification of betalains from the fruits of 10 Mexican prickly pear cultivars by high-performance liquid chromatography and electrospray ionization mass spectrometry. *Journal of Agricultural and Food Chemistry, 56*(14), 5758–5764.

Chandra, S., Singh, S., & Kumari, D. (2015). Evaluation of functional properties of composite flours and sensorial attributes of composite flour biscuits. *Journal of Food Science & Technology, 52*(6), 3681–3688.

Choudhury, M., Badwaik, L. S., Borah, P. K., Sit, N., & Deka, S. C. (2015). Influence of bamboo shoot powder fortification on physico-chemical, textural and organoleptic characteristics of biscuits. *Journal of Food Science & Technology, 52*(10), 6742–6748.

CIE (1976). *Colourimetry: Official recommendation of the international commission on illumination.* Paris, France: Bureau Central de la CIE. Publication CIE No. (E-1.31).

Diaz Vela, J., Totosaus, A., Cruz Guerrero, A. E., & de Lourdes Pérez Chabela, M. (2013). In vitro evaluation of the fermentation of added-value agroindustrial by-products: Cactus pear (*Opuntia ficus-indica* L.) peel and pineapple (*Ananas comosus*) peel as functional ingredients. *International Journal of Food Science and Technology, 48*(7), 1460–1467.

Ennouri, M., Bouret, E., Mondolot, L., & Attia, H. (2005). Fatty acid composition and rheological behaviour of prickly pear seed oils. *Food Chemistry, 93*, 431–437.

FAO (2017). In P. Inglese, C. Mondragon, A. Nefzaoui, & C. Saenz (Eds.). *Crop, ecology, cultivation and uses of cactus pear* (pp. 244). Rome: FAO. Year of upload 2017 http://www.fao.org/3/a-i7012e.pdf.

Fustier, P., Castaigne, F., Turgeon, S. L., & Biliaderis, C. G. (2008). Flour constituent interactions and their influence on dough rheology and quality of semi-sweet biscuits: A mixture design approach with reconstituted blends of gluten, water-solubles and starch fractions. *Journal of Cereal Science, 48*(1), 144–158.

Galla, N. R., Pamidighantam, P. R., Karakala, B., Gurusiddaiah, M. R., & Akula, S. (2017). Nutritional, textural and sensory quality of biscuits supplemented with spinach (*Spinacia oleracea* L.). *International Journal of Gastronomy and Food Science, 7*, 20–26.

Gil, M. I., Selma, M. V., López-Gálvez, F., & Allende, A. (2009). Fresh-cut product sanitation and wash water disinfection: Problems and solutions. *International Journal of Food Microbiology, 134*(1–2), 37–45.

Inglese, P., Basile, F., & Schirra, M. (2002). Cactus pear fruit production. In P. S. Nobel (Ed.). *Cacti biology and uses* (pp. 163–183). Los Angeles: University of California Press (Chapter 10).

Jeddou, K. B., Bouaziz, F., Zouari-Ellouzi, S., Chaari, F., Ellouz-Chaabouni, S., Ellouz-Ghorbel, R., et al. (2017). Improvement of texture and sensory properties of cakes by addition of potato peel powder with high level of dietary fiber and protein. *Food Chemistry, 217*, 668–677.

Kaur, M., Kaushal, P., & Sandhu, S. K. (2011). Studies on physicochemical and pasting properties of Taro (*Colocasia esculenta* L.) flour in comparison with a cereal, tuber and legume flour. *Journal of Food Science & Technology, 50*(1), 94–100.

Kaur, M., Sandhu, K. S., Arora, A., & Sharma, A. (2015). Gluten free biscuits prepared from buckwheat flour by incorporation of various gums: Physicochemical and sensory properties. *LWT-Food Science and Technology, 62*(1), 628–632.

Lahsasni, S., Kouhila, M., Vighon, M., & Mahrouz, M. (2004). Impact of convective solar drying on the color and the composition out of free sugars of the cladode, the peel and the fruit of a cactus inerme of *Opuntia ficus indica*. *Physical and Chemical News, 18* 89–89.

Larrauri, J. A. (1999). New approaches in the preparation of high dietary fibre powders from fruit by-products. *Trends in Food Science & Technology, 10*(1), 3–8.

McCance, R. A., Prior, K. M., & Widdowson, E. M. (1953). A radiological study of rate of passage of brown and white bread through the digestive track of man. *British Journal of Nutrition, 7*(1–2), 98–104.

Montevecchi, G., Masino, F., Di Pascale, N., Vasile Simone, G., & Antonelli, A. (2017). Study of the repartition of phthalate esters during distillation of wine for spirit production. *Food Chemistry, 237*, 46–52.

Mridula, D. (2011). Physico-chemical and sensory characteristics of β-carotene rich defatted soy fortified biscuits. *African Journal of Food Science, 5*(5), 305–312.

Msaddak, L., Siala, R., Fakhfakh, N., Ayadi, M. A., Nasri, M., & Zouari, N. (2015). Cladodes from prickly pear as a functional ingredient: Effect on fat retention, oxidative stability, nutritional and sensory properties of cookies. *International Journal of Food Sciences & Nutrition, 66*(8), 851–857.

Nefzaoui, A., & Ben Salem, H. (2006). Cactus: Un banco de alimentos para ganado en entornos áridos y semiáridos. Cactus: A food bank for cattle in arid and semi-arid environments [original language]. *Cactusnet Newsletter, 10*, 41–57 [Número Especial].

Noor, S. A. A., Siti, N. M., & Mahmad, N. J. (2015). Chemical composition, antioxidant activity and functional properties of mango (*Mangifera indica* L. var. Perlis Sunshine) peel flour (MPF). *Applied Mechanics and Materials, 754*, 1065–1070.

Ramadan, M. F., & Mörsel, J. T. (2003). Recovered lipids from prickly pear [*Opuntia ficus-indica* (L.) Mill] peel: A good source of polyunsaturated fatty acids, natural antioxidant vitamins and sterols. *Food Chemistry, 83*(3), 447–456.

Ranganna, S. (1979). *Manual of analysis of fruit and vegetables products.* New York: Tata McGraw-Hill Publishing Company.

Raymundo, A., Fradinho, P., & Nunes, M. C. (2014). Effect of Psyllium fibre content on the textural and rheological characteristics of biscuit and biscuit dough. *Bioactive Carbohydrates and Dietary Fibre, 3*(2), 96–105.

Robertson, J. A., de Monredon, F. D., Dysseler, P., Guillon, F., Amado, R., & Thibault, J. F. (2000). Hydration properties of dietary fibre and resistant starch: A European collaborative study. *LWT-Food Science and Technology, 33*(2), 72–79.

Rodriguez-Amaya, D. B. (1999). *A guide to carotenoid analysis.* Washington DC: ILSI Press.

Romelle, F. D., Rani, P. A., & Manohar, R. S. (2016). Chemical composition of some selected fruit peels. *European Journal of Food Science and Technology, 4*(4), 12–21.

Rosell, C. M., Rojas, J. A., & Benedito De Barber, C. (2001). Influence of hydrocolloids on dough rheology and bread quality. *Food Hydrocolloids, 15*, 75–81.

Sehn, G. A. R., Nogueira, A. D. C., Almeida, E. L., Chang, Y. K., & Steel, C. J. (2015). Fortification of wheat dough with calcium and magnesium ions affects empirical rheological properties. *Cereal Chemistry, 92*(4), 405–410.

Swain, T., & Hillis, W. E. (1959). The phenolic constituents of *Prunus domestica*. I.—the quantitative analysis of phenolic constituents. *Journal of the Science of Food and Agriculture, 10*(1), 63–68.

Yadav, R. B., Yadav, B. S., & Dhull, N. (2012). Effect of incorporation of plantain and chickpea flours on the quality characteristics of biscuits. *Journal of Food Science & Technology, 49*(2), 207–213.

Yeddes, N., Chérif, J. K., Guyot, S., Sotin, H., & Ayadi, M. T. (2013). Comparative study of antioxidant power, polyphenols, flavonoids and betacyanins of the peel and pulp of three Tunisian *Opuntia* forms. *Antioxidants, 2*, 37–51.