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Opuntia ficus-indica seed pomace extracts with high UV-screening ability in a circular economy approach for body lotions with solar protection

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ABSTRACT

Opuntia ficus-indica seed pomace, a by-product of seed oil extraction, was investigated due to its richness of phytochemical compounds and high antioxidant capacity. The pomace was subjected to hydrothermal extraction using water under subcritical conditions to solubilize various compounds. Different final heating temperatures 120 to 220 °C were applied, and the resulting filtered extracts were analyzed using HPLC and HPSEC to determine the monosaccharide profile and molecular weight of the oligomers. Notably, extractions performed at temperatures between 120 and 180 °C yielded extracts characterized by elevated levels of glucuronic acid and exhibited higher molecular weights. In contrast, extractions conducted at 200 and 220 °C were distinguished by their enriched xylose content and relatively low molecular weights, falling below 23.6 kDa. The extracts were tested for total phenolic compounds, as well as antioxidant activity using spectrophotometric methods such as TEAC, DPPH, and FRAP. Results showed that increasing temperature led to a corresponding increase in phenolic compounds (from 7.7 to 13.7 g_{GAE}/100 g) and antioxidant activity, with the maximum observed at 200 °C. These phenolic compounds enriched extract were used to produce a body lotion with sun protection factor of 8, with rheological behavior similar to that of commercially available products but without the need for additional UV filters and antioxidants. The lotion was shown to be safe for topical use and did not cause skin irritation. This study highlights the potential of *O. ficus-indica* seed pomace as a valuable source of phytochemical compounds and demonstrates the feasibility of using hydrothermal extraction to produce eco-friendly commercially interesting compounds.

Introduction

Opuntia ficus-indica L., an indigenous cactus species of Mexico, is cultivated and utilized in various regions worldwide, including Africa, Australia, and the Mediterranean region[62], due to its remarkable versatility and substantial economic value in the agricultural industry. The plant's adaptability to diverse climatic conditions, coupled with its capacity to produce nutritious fruits and edible cladodes that can be used as a source of fodder for livestock, makes it an indispensable crop for small-scale farmers and agribusinesses alike[53,70].

The fruit, is constituted mainly by its juicy pulp (28–58 % of fruit mass), seeds (2–10 %), which have a high content of oil, and a thick peel (37–67 %)[9], is rich in biological compounds, possesses numerous beneficial properties, including antioxidant, anti-diabetic, anti-tumoral, anti-hyperlipidemic, and anti-inflammatory effects[29,59], making it a valuable resource for food security. However, the increased cultivation and consumption of prickly pear have led to the generation of large quantities of food waste along the food chain, which poses significant environmental challenges[27]. The generation of large amounts of agricultural by-products and industrial waste has become a crucial problem in many countries around the world, particularly in countries where environmental concerns are raised. In this respect, the scientific community has been increasingly concerned with the treatment of waste through the development of innovative products and materials [44,21][81]. In fact, the use of these remaining wastes may have a positive impact on the reduction of solid residues, on the production of low-cost materials with high added value, and the protection of the environment[49]. Related to this,

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by-products such as grape seed pomace[20]and other fruit and vegetable materials have already been extensively studied in order to obtain high-value products. Nevertheless, the realm of cosmetic applications has remained somewhat underexplored within this domain. While select studies have explored the application of materials like tomato pomace, grape pomace, and grape seed in the context of cosmetics[26,73], there exists a conspicuous absence of research pertaining to resources like prickly pear pomace resultant from seed oil extraction.

As the demand for natural extracts with bioactive compounds for pharmacological and cosmetic applications has increased, there has been a growing interest in using cleaner extraction techniques with greener solvents (L. [45]. Pressurized hot water extraction under subcritical conditions (i.e., autohydrolysis), which uses water as the sole solvent and reagent, has gained attention due to its effectiveness in obtaining extracts rich in bioactive compounds [25,82]. Therefore, the main objective of this study was to valorize prickly pear seed pomace by obtaining an autohydrolysis extract rich in bioactive compounds and further applying it as an active ingredient in a body lotion with high ultraviolet (UV) radiation screening capabilities.

UV radiation (λ = 280–400 nm) from the sun is the primary cause of UV exposure in humans, and excessive exposure without proper protection, like sunscreen and clothing, has been linked to skin cancer due to UV-induced damage to cellular components like proteins and DNA. This damage occurs through two mechanisms: (a) direct absorption of UV light by cellular components, leading to damaging chemical reactions, and (b) photosensitization, wherein light activates sensitizing agents that can detrimentally affect cells through electron transfer and hydrogen abstraction (Type I) or energy transfer with oxygen, creating reactive singlet oxygen (Type II). Notably, the direct absorption of UV radiation by DNA instigates the formation of DNA base dimers and related molecular species[61,65]so, there exists a compelling need for the development of innovative and environmentally sustainable strategies aimed at mitigating solar-induced damage. Within this purview, this research aimed to develop an eco-friendly alternative for industrial compound production by prioritizing the use of natural resources and waste biomass in line with the principles of biorefinery and circular bioeconomy.

Beyond its primary scientific objectives, this study extended its reach to encompass the realms of environmental preservation and public health offering a tangible blueprint for the sustainable valorization of agricultural by-products within the multifaceted landscape of industrial sectors. By effectively reducing waste, and bestowing health-related advantages through the introduction of innovative skincare solutions, thereby instigating innovative products within the green cosmetic industry, this research assumes a role of paramount societal importance. Serving as a steppingstone for more conscientious resource management and an unwavering commitment to technological progress. Moreover, the methods employed in this study, particularly when extrapolated to larger industrial yield substantial quantities of extracts, boasting high extraction yields, and are characterized by relatively low processing times, thus surpassing the efficiency of more conventional extraction techniques. As such, this research unveils a transformative potential that holds immense promise for broad-scale industrial applications.

Material and methods

Material

Prickly pear fruits were harvested in Idanha-a-Nova, Portugal, by the Agricultural Cooperative, Fig d'Idanha. After harvesting, the fruits were processed separately, following internal industrial procedures of Figo d'Idanha, to obtain the pulp + seeds and peels separately. The pulp + seeds set was passed through a filter that retained the seeds, which were collected separately, stored in a polyethylene bag containing and transported in a refrigerated chamber to the laboratory. After arrival, the seeds were dried at 35 °C using a food dehydrator (LACOR MENAJE PROFESIONAL S.L, Spain) until reaching a moisture content of less than 4 %. The dried seeds were ground, and the flour obtained was sieved with a 38-mesh filter, to standardize the particle size. The pomace was obtained after hexane extraction of the seed oils.

Extraction method

Prickly pear seed pomace (PPSP) extracts were obtained using the autohydrolysis technique with conventional heating in a 600 mL extractor (Parr Instrument Company, Moline, IL, USA). Milled PPSP was mixed with distilled water in a liquid-to-solid mass ratio (LSR) (w:w) of 8:1, and the autohydrolysis process was carried out under non-isothermal conditions, with the heating temperature ranging from 120 to 220 °C. The resulting suspension was rapidly cooled with water using a stainless-steel coil. The solid and liquid phases were separated via vacuum filtration, and the liquid phase (i.e., extract) was stored at −20 °C until further use.

To compare the effect of operational conditions in the equipment while minimizing the impact of different heating–cooling profiles, a severity concept was employed. This severity concept takes into account the effects of both time and temperature. Typically, the severity, denoted as S_0, is expressed as the decimal logarithm of R_0:

$$\log R_0 = \log\left(\pi R_{0,heating} + R_{0,cooling}\right) = \log\left\{\left[\int_0^{t_{max}} \exp\left(\frac{T(t)-T_{ref}}{\omega}\right)dt\right] + \left[\int_{t_{max}}^{t_{final}} \exp\left(\frac{T'(t)-T_{ref}}{\omega}\right)dt\right]\right\} \quad (1)$$

where R_0 is the severity factor, t_{max} (min) is the time to reach the target temperature T_{max} (°C), t_{final}(min) is the time for the heating–cooling, and T(t) and T'(t) is the temperature profiles in the heating and cooling stages, respectively, and ω (14.75 °C), T_{ref} (100 °C).

Analytical techniques

The moisture content of the sample was determined through gravimetric analysis using an oven-drying method at 105 °C until a constant weight was obtained within a period of 24–48 h. Ash content was also measured using a gravimetric approach after calcination in a muffle furnace for 6 h at 575 °C. The mineral content was determined using acid digestion with nitric acid (10 mL) and hydrogen peroxide (1 mL) on a Marsxpress (CEM) instrument. Ash (0.3 g) was used for the acid digestion process. The treatment was performed at 1600 W for 15 min, ramping up to 200 °C, and then maintained at this temperature for 10 min. The contents of Na and K were analyzed using Atomic Emission Spectrophotometry (AES), while Zn, Ca, Mg, Fe, and Cu were assessed using Atomic Absorption Spectrophotometry via a 220 Fast Sequence Spectrophotometer (Varian, CA). The levels of Cd, Mn, Se, P, and Pb were determined using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) via an X Series instrument (Thermo Scientific, Waltham, MA, USA). The total content of nitrogen, hydrogen, and carbon in the sample were analyzed using a FlashEA 1112 Elemental Analyzer (Thermo, Waltham, MA, USA) with Helium as the carrier and reference gas. The temperatures of the oxidation and reduction ovens were 900 °C and 680 °C, respectively, and the oxygen flow was 250 mL/min. Protein content was calculated using the univer-

sal conversion factor 6.25 [79]. To determine the carbohydrate content of PPP, acid hydrolysis was carried out using 72 % sulfuric acid (30 °C, 1 h) and 4 % sulfuric acid (121 °C, 60 min), and then analyzed using High-Performance Liquid Chromatography (HPLC) with two different columns: Aminex HPX-87H (50 °C, 0.6 mL/min 0.003 M H_2SO_4) and HPX-87P (80 °C, 0.4 mL/min ultra-pure water). Fatty Acid Methyl Esters (FAMEs) were prepared in accordance with UNE-EN ISO 12966–3:216 and subsequently analyzed via a GC–MS QP 2010 (Shimadzu, Kyoto, Japan). The following temperature profile was applied during the analysis: 50 °C (2 min), gradient increase at 10 °C/min up to 240 °C, which was maintained for 27 min. FAMEs were identified using their mass spectra, which were compared to those of authentic standards and the NIST MS Search 2.0 library. Results obtained from two independent determinations were averaged and expressed as a percentage of total FAME.

Molar mass distribution

The molecular weight profiles of the extracts were assessed by high-performance size exclusion chromatography (HPSEC). The columns used were a pre-guard column PWX guard (40×6 mm^2) and the columns Skel G3000PWXL and G2500PWXL (300×7.8 mm^2), both from Tosoh Bioscience (Stuttgart, Germany). A refractive index (RI) detector was used to obtain the chromatograms, with the operation conditions: 70 °C, Milli-Q water as the mobile phase, and 0.4 mL/min the flow rate. Dextrans from 1 kDa to 80 kDa were used as patterns. As for the higher molecular weight tested- from 23.6 to 786 kDa (Tosoh Corporation, Japan)- an HPLC supplied by SuperMultipore PW-H column (6 mm × 15 cm) with a guard column SuperMP (PW)-H (4.6 mm × 3.5 cm), both from TSKgel by Tosoh Corporation (Japan) was used. The operation conditions were: 40 °C and Milli-Q water as mobile phase (0.4 mL/min), the detector used was a refractive index (RI) detector. The standards used were polyethylene oxide.

Oligosaccharide content

The liquid samples obtained from the tested extraction processes were hydrolyzed with sulfuric acid at 4 % (v/v), with the operation conditions: 20 min, 121 °C and 2 atm. The hydrolyzed samples were filtered through 0.45 μm cellulose acetate cartridges (Sartorius, Germany) and analyzed by high-performance liquid chromatography (HPLC, 1100 Agilent, Germany), coupled to a refractive index detector. The column used was an Aminex HPX-87H (300×7.8 mm^2) from BioRad (Hércules, CA, USA) working at 60 °C, the mobile phase was sulfuric acid (0.003 M), and the flow rate 0.6 mL/min. The standards used were glucose, galactose (Gal), xylose (Xyl), mannose (Man), rhamnose, arabinose, formic acid, acetic acid, galacturonic and galacturonic acid, furfural and hydroxymethylfurfural (HMF) from Sigma-Aldrich (Bratislava, Slovakia). Galactose, xylose, and mannose, eluting at the same retention time, were quantified as Gal + Xyl + Man.

Total phenolic content

Total phenolic content of the extracts was determined by the Folin–Ciocalteu method, using gallic acid as a standard. Briefly, 0.5 mL of extract was mixed with 3.75 mL of water, 0.25 mL of Folin–Ciocalteu's reagent (diluted 1:1 with water), and 0.5 mL of sodium carbonate (10 %, w/v). Samples were incubated for 1 h in absence of light at room temperature before absorbance readings at 765 nm[58]. Three independent assays performed in duplicate.

Antioxidant capacity

The ability of antioxidants/extracts to scavenge free radicals was assessed using two different methods, namely the 2,2-diphenyl-1-picrilhydrazil (DPPH) and 2,2́-azinobis-(3-ethyl-benzo thiazoline-6-sulfonate) (ABTS) assays. In the DPPH assay, 50 μL of antioxidant/extract were added to 2 mL of a methanolic solution of a 3.6×10^{-5} M of DPPH, and the decrease in absorbance at 515 was measured initially (t_0) and after 16 min (t_{16}). The IC_{50} value was calculated for each sample based on at least four dilutions [75]. $ABTS^{\bullet+}$ was prepared by reacting 0. 384 g of ABTS-NH_4 with 0.0662 g potassium persulfate in the dark at room temperature for 12–16 h and then diluted until an absorbance of 0.70 ± 0.05 at 734 nm. Finally, 10 μL of the antioxidant/extract were mixed with 1 mL of the previously prepared solution and the absorbance was measured after 6 min at 30 °C and the results were expressed as Trolox equivalents [66].

In addition, the iron reducing power of the extracts was evaluated using the ferric reducing antioxidant power (FRAP) assay. Briefly, 1 mL of extract/antioxidant (ascorbic acid) was added to 2.5 mL of phosphate buffer (0.2 M and pH 6.6) and 2.5 mL of potassium ferricyanide at 1 %. This was incubated at 50 °C for 30 min and 2.5 mL of trichloroacetic acid at 10 % were added. Afterwards, the resulting mix was centrifuged for 10 min at 4500 rpm and the supernatant was mix with distilled water (1:1, v/v). The absorbance was measured at 700 nm in a spectrometer (Evolution 201, Thermo Scientific, Shanghai, China) after 0.5 mL of ferric chloride (0.1 %, w/v) was added[28,58]. Three independent assays were performed in duplicate to ensure the reliability of the results.

Anti-inflammatory activity

COX-1 and COX-2 inhibitions were evaluated using a fluorometric Cox Activity assay kit (Sigma-Aldrich, Massachusetts, USA) and as described in Casas et al. [14]. Cells and tissues were appropriately washed and prepared in accordance with the kit instructions. To prepare the reaction mixes for samples and those containing the inhibitor, COX Probe, diluted COX cofactor, samples, and COX assay buffer were mixed. For the positive control and the positive control with the inhibitor, the reaction mix was prepared including the COX-1 Positive Control. To determine COX activity, a solution of 2 mL of COX cofactor was diluted in 398 mL of COX assay buffer. An arachidonic acid solution was prepared by adding 5 mL of the acid to 5 mL of NaOH, and then diluting the resulting solution with purified water in a 1:10 ratio. In microplate, one well was filled with 2 mL of DMSO for the total activity assay of the sample, and another was filled with 2 mL of either COX-1 or COX-2 inhibitor for partial activity of sample + inhibitor. For measuring COX-1 activity, COX-1 Inhibitor (Indomethacin) was added, and for COX-2 activity, COX-2 Inhibitor (Celecoxib) was added. 88 mL of the suitable reaction mixture was introduced to the wells and 10 mL of the arachidonic acid/NaOH solution was added to each sample and positive control well, but not to the standard well. The plate was excited at a wavelength of 535 nm (λ_{Ex} = 535 nm), and the resulting emission was recorded at a wavelength of 587 nm (λ_{Em} = 587 nm) every 15 s for a total duration of 30 min. To determine the standard curve, Resorufin Substrate Solution was appropriately diluted in COX Assay Buffer ranging from 0.2 to 0 mM.

Tyrosinase enzymatic inhibition assays

Selected samples were tested for their inhibitory effects on tyrosinase activity, following the general procedure previously described by Chiari et al.[17]. Briefly, a solution of 2 μL mushroom tyrosinase (2500 U/mL) in 50 mM phosphate buffer at pH 6.5 was

mixed with 148 μL of 50 mM phosphate buffer and dissolved in dimethyl sulfoxide. The samples were then incubated for 90 min at 37 °C with continuous agitation. After the incubation, 40 μL of 2.5 mM L-tyrosine or L-Dopa-(phenyl-d3) in phosphate buffer was added to the mixture, and the absorbance at 450 nm was measured directly and after 5 and 15 min. The difference in absorbance values at each time point was used to determine the inhibitory percentage of the samples. Kojic acid was employed as a positive control.

UV-A/B filters

UV wavelength scanning

Selected samples were subject to a UVB and UVA wavelength (200–400 nm). Briefly, the extracts and 2-ethylhexyl 4-(dimethylamino) benzoate were diluted to allow the absorbance for all the wavelength texted to be around the linearity maximum (around 1) and subject to scanning do determine the wavelengths with the maximum absorbance in an (Evolution 201, Thermo Scientific, Shanghai, China) spectrophotometer.

Body lotion with sunscreen characteristics formulation

Body lotions with sunscreen effect formulations were made in duplicate following the altered protocol previously reported by [45]. Four different formulations were prepared using oil cream basis (O/W) consisting of 18 g of oil cream basis, 6 g of dimethicone 350, 3 g of avocado oil, 8 g of sunscreen, 18 g of micronized titanium dioxide, and 0.35 g of fenonip XB. The water phase contained 80 g of water, 1.5 g of carbomer 940, 6 g of propyleneglycol, and 1.5 g of triethanolamine. The first two formulations served as control groups and were prepared following the original protocol. The oil basis was heated to 70 ± 2 °C and when melted the oil phase was then added to the water phase and homogenized until a gel-like matrix was formed. To the control groups, 750 μL of butylhydroxytoluene (BHT) or water was added. The remaining two formulations were prepared in a similar manner. The first formulation, a commercial sunscreen, replaced 8 g of the solid sunscreen with 8 g of 2-ethylhexyl 4-(dimethylamino) benzoate. The second formulation, containing *Opuntia* pomace extract, omitted the 8 g of sunscreen and 72 g of water adding 80 g of extract from autohydrolysis. Commercial BHT was added for comparative purposes with the soluble extracts rich in antioxidants recovered. Bergamot oil (450 μL) and tetramer cyclomethicone (3 mL) were added and mixed with the O/W emulsion at room temperature before storing in flasks and amber glass vials at refrigeration temperature. The four formulations were then evaluated for their stability, rheology, and in vitro sun protection factor (SPF) values.

Sun protection factor (SFP)

Different photoprotection factors (PPFs) of the 200 °C extract of PPSP were estimated by an accredited laboratory in Malaga, Spain (IBYDA, Málaga University). The sun protection factor (SPF) was calculated using the erythematic action spectrum; UV-A radiation protection factor (UVAPF) according to ISO 24442:2022 was calculated with the action spectrum of pigmentation Persistent Pigment Darkening -PPD and factors of protection against other biological effects related to UV radiation (BEPFs- Biological Effective Protection Factors), were performed against the following action spectra: photocarcinogenesis (UV-B related), immunosuppression (UV-B related), elastosis (UV-A related), photoaging (UV-A related) and singlet oxygen formation (related UV-A) according to Coba et al., 2019.

The critical λ value (λλc), length of wave at which 90 % of the received radiation is absorbed, and the SPF were measured based on the ISO:24442, 2022. For BEPFs, PMMA plates (Polymethylmethacrylate; 25 × 25 mm; roughness 6 μm; Schönberg, Hamburg, Germany), whose roughness simulates human skin were used to spread the body lotion. The spread cream at a concentration of 1.3 mg cm^{-2} (=32.5 mg $plate^{-1}$) was incubated in the PMMA for 15 min in the dark at room temperature and measure the transmittance through the plate in a spectrophotometer (UV-2600, Shimadzu, Duisburg, Germany) with integrating sphere (ISR 2600 Plus, Shimadzu, Duisburg, Germany). 2 plates per sample and two measurements of each plate were made (Yéssica A. [80].

$$SPF = \frac{\int_{\lambda=290}^{\lambda=400} BAS(\lambda) x I(\lambda) x d(\lambda)}{\int_{\lambda=290}^{\lambda=400} T(\lambda) x BAS(\lambda) x I(\lambda) x d(\lambda)}$$

BAS (λ) = Biological action spectrum (0–1).
I (λ) = Irradiance of a say day at noon in ($W/m^{-2}(-|-)$).
d(λ) = Wavelength step (1 nm).
T(λ) Transmittance (0–1).

Rheological measurements

Steady-state shear measurements were conducted at least in duplicate to determine the viscous profiles at 25 °C of the proposed sunscreen creams. Time-dependent shear thinning was also evaluated by monitoring the forward and backward flow curves. Then, time measurements were performed to assess the stability of the samples. Rheological tests were carried out on a MCR 302 controlled-stress rheometer (Paar Physica, Austria) using a sand-blasted parallel plate (25 mm diameter, 1 mm gap) to prevent slippage of tested creams equipped with a Peltier system (±0.01 °C). Creams were placed on the measuring geometry, sealed with light paraffin oil to avoid drying during testing, and rested for 5 min before rheological measurements to promote structural and thermal equilibration.

SkinEthic™ skin irritation test

The SkinEthic™ skin irritation test was performed on the produced body lotion using the SkinEthic™ Reconstructed Human Epidermis (RHE) model on a selected system with the best viscoelastic features. The aim of this method was to predict and categorize the skin irritation potential of test chemicals in accordance with the United Nations Globally Harmonized System of Classification and Labelling of Chemicals (UN GHS). Specifically, this method was used to distinguish skin irritant chemicals and mixtures (Category 2) from chemicals Not-Classified for skin irritation (No Category). The test was conducted in accordance with the guidelines provided by the commercial supplier (EPISKIN™) and the SkinEthic™ Skin Irritation Test (DB-ALM Protocol n° 135). The protocol adhered to the OECD Test Guideline No. 439 - *In Vitro* Skin Irritation: Reconstructed Human Epidermis Test Method [56].

Results and discussion

Opuntia ficus-indica seed pomace characterization

Table 1 shows the proximal composition of prickly pear seed pomace. The moisture was found to be consistent with previous reports for prickly pear seeds [24]. The nitrogen, carbon, and hydrogen contents were determined using dynamic flash combustion technology and are presented in Table 1. These values were used to estimate the total calorific value, which corresponded to 20560.71 ± 1.4 kJ/kg, a value comparable to those obtained for other plant materials such as grapes, almonds, and rapeseed seeds[33]. On the other hand, the protein content of PPSP was superior to values reported in the literature for other prickly pear seeds (18.0 ± 0.2 % vs 12–13 %)[23,52]a fact that is possibly due

Table 1
Proximal composition and nutritional composition given by the minerals, monosaccharides, and fatty acids of Opuntia ficus-indica seed pomace.

Prickly pear Pomace		
Proximal composition	Humidity (% dw)	5.08 ± 0.02
	Ash (% dw)	1.48 ± 0.02
	Nitrogen (N)	3.28 ± 0.03
	Carbon (C)	46.5 ± 0.1
	Hydrogen (H)	6.41 ± 0.1
	Alcohol insoluble residue (AIR)	18.51 ± 1.4
	Carbohydrates (% dw)	57.22 ± 3.77
	Lipids (% dw)	4.75 ± 0.1
	Protein (% dw)	18.0 ± 0.3
	Caloric power (kcal/kg)	4913.27 ± 1.5
Macro minerals (g/kg)	Ca	153.7 ± 16.5
	Zn	1.11 ± 0.21
	Cu	0.22 ± 0.09
	Fe	1.83 ± 0.12
	K	160.7 ± 17.1
	Mg	69.17 ± 8.9
	Mn	4.10 ± 0.92
	Na	8.65 ± 1.03
	P	95.0 ± 8.6
Micro minerals (mg/kg)	Se	2.47 ± 0.88
	Pb	4.09 ± 0.74
	Cd	3.75 ± 0.59
Monosaccharides (%)	Glucuronic acid	2.32 ± 0.51
	Galacturonic acid	2.16 ± 0.12
	Glucose	22.64 ± 1.04
	Xylose	26.42 ± 1.15
	Arabinose	1.54 ± 0.21
	Fucose	2.09 ± 0.27
Fatty acids (%)	Palmitic acid	1.38 ± 0.23
	Stearic acid	0.31 ± 0.09
	Oleic acid	0.86 ± 0.07
	Linoleic acid	2.09 ± 0.25

to the oil extraction procedure, and the consequent increase of the percentage levels of the remaining compounds.

Carbohydrates accounted for up to 57.22 ± 3.17 % of the raw material, which is similar to values reported in previous studies of prickly pear seeds by Nassar and Kossori[23,52]. Glucose and xylose deemed to be the most abundant carbohydrates in *O. ficus-indica* seed pomace, accounting for approximately 50 % of the total carbohydrates. In addition, small amounts of glucuronic and galacturonic acids, as well as arabinose and fucose, were also detected, around 2 % each, probably as a results of pectin degradation due to the acid hydrolysis performed [30]. Similar results were reported for other parts of the plants such as peels and cladodes [31,67].

The lipid content of the prickly pear seed pomace was 4.75 %, a lower value compared to those reported in literature for prickly pear seeds, which typically range around 10 %[24,52]This discrepancy may be attributed to the fact that the raw material had been subjected to prior lipid extraction using conventional techniques, such as maceration with hexane for a duration of 24 h. The residual lipid content of 4.75 % represents the fraction that remained unextracted by the conventional methods[2]. These lipids detected in the remaining seed pomace were similar to those reported in literature where linoleic, oleic, and palmitic acids were found in higher concentrations for seeds from different cultivars and cultivated around the world[2].

The mineral content of the raw material was higher than that reported by Mehmet and Fahad for prickly pear seeds from *O. ficus-indica* from Turkey [55]. Potassium (K) and calcium (Ca) were present in higher quantities, with values of 160.7 g/kg and 153.7 g/kg, respectively. In addition, low quantities of cadmium and lead were detected (4.09 ± 0.74 and 3.75 ± 0.59 respectively), but they were within the allowable concentrations set by the FDA (“Lead in Cosmetics | FDA,” n.d.). So, although the seeds were contaminated with these minerals from the soil [60], they can still be used to produce cosmetics.

Opuntia ficus-indica seed pomace extracts

Autohydrolysis

Prickly pear seed pomace was used to obtain extracts using subcritical water extraction. In order to understand the impact of temperature on the extraction of phenolic compounds and on the antioxidant capacity of the extracts, a screening of temperatures was performed (120–220 °C) obtaining the severities between $S_0 = 1.36$ and $S_0 = 4.44$, as presented in Fig. 1 below. As expected, the severity of the process increased for higher temperatures, as presented by Huamán-Leandro et al. for *Lentinus edodes* [38].

Opuntia ficus-indica extracts characterization

The mineral, protein and carbohydrates profile of the different extracts obtained after autohydrolysis are presented in supplementary Table S.1, Table 2 and Fig. 2 below, respectively. As can be observed from Table S.1 the mineral content was very similar for all six extracts produced, which means that temperatures between 120 and 220 °C had little influence on the extraction of minerals from the seeds. As for the protein content, and as expected, it was noticed a decrease in the protein of the extracts with increased temperature. The structure of protein is known to start denaturation at around 40 °C and its stability decreases with temperature [39].

The effect of the autohydrolysis temperature on the sugar composition and its degradation compounds are presented below. The fractions obtained at different temperatures were mostly rich in glucuronic acid, glucose and xylose, being the first higher for lower temperatures (120 to 180 °C) hence its present in more available/free sugars such as pectin and glycosaminoglycans that are extracted more easily. Xylose is a result of the degradation of xylans, a derivate of hemicellulose present in PPS [35]and hence, it is not surprising that this monomer was found as the major compound at 220 °C[76]. In turn, glucose levels were low for all the samples despite being one of the major sugars present in plant materials. This fact indicates that the temperatures used were not high enough to destroy cellulose and that the solubilized glucose in the extracts was non-cellulosic[76]. Similar results were obtained by Habibi et al, for *O. ficus-indica* seeds collected from fruits planted in Marrakech (Morocco), who reported glucuronic acid and xylose as the two major monomers detected after extraction at 100 °C for 2 h and posterior sodium chloride treatment [35]. Formic and acetic acid were detected for all temperatures, since they are sugar degradation compounds produced as a result of high temperatures. Moreover, the furfural detected in the extract obtained at 220 °C is most probably a result of the degradation of xylose, glucuronic and galacturonic acids, among others, as

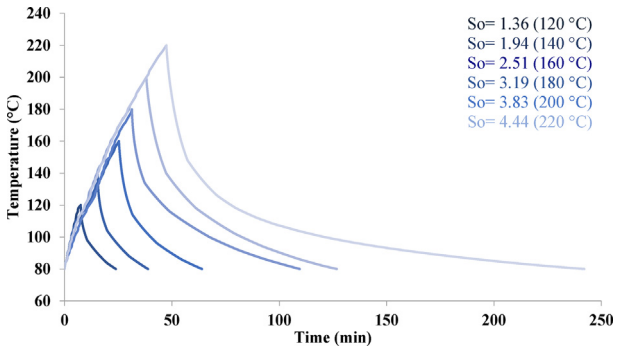

Fig. 1. Effects of temperature on the severity of the autohydrolysis process.

Table 2
Effect of autohydrolysis temperature on the yield of extraction, and the protein and ash content (g/100 $g_{extract}$) of Opuntia ficus-indica extracts obtained by autohydrolysis.

Autohydrolysis Temperature	Yield (%)	Protein (g/100 g extract)	Ash (g/100 g extract)
120 °C	2.57 ± 0.04	6.82 ± 0.03	0.81 ± 0.04
140 °C	3.2 ± 0.1	5.73 ± 0.03	0.83 ± 0.07
160 °C	4.8 ± 0.1	4.51 ± 0.05	0.59 ± 0.02
180 °C	8.7 ± 0.3	2.09 ± 0.01	0.50 ± 0.03
200 °C	9.7 ± 0.2	0.737 ± 0.01	0.49 ± 0.05
220 °C	15.9 ± 0.5	0.240 ± 0.01	0.57 ± 0.06

reported by Gong et al. [32]. Similar results were also observed by Queffelec et al[64].

Molar mass distribution

The results of the molar mass distribution profiles of extracts, obtained through hydrothermal processing at different temperatures, as presented in Fig. 3. Notably, distinctive patterns were observed for the extracts obtained in the range of temperatures from 120 to 180 °C, when compared to those obtained at higher temperatures (i.e., 200 and 220 °C). Specifically, for the extracts obtained at 120 and 140 °C, most of the particles exhibited weights within the range of 23.6 kDa to 786 kDa. Conversely, the extracts obtained at 160 and 180 °C showed additional peaks were observed for molecular weights between 23.6 kDa and 12 kDa, as observed in the Supplementary Fig. S.1. The data obtained for the extracts obtained at 200 and 220 °C were consistent with the sugar content and demonstrated a temperature-dependent degradation of larger molecules, such as polysaccharides. Accordingly, for these two temperatures, only a dominant peak was observed for molecular weights below 23.6 kDa. Notably, as presented in Fig. S.1, the extracts obtained at these temperatures exhibited different size peaks, including molecular weights under 1 kDa. The observed molar weight distribution profiles of the extracts obtained through autohydrolysis can be explained by the severity of the extraction method utilized. In support of these findings, previous studies conducted with hydrolysis extraction of brown seaweed and on plant materials derived from eucalyptus, Miscanthus and wheat straw, also demonstrated a similar behavior, i.e., a temperature-dependent decrease in the molecular weight of the oligomers [64,74].

Total phenolic compounds and antioxidant properties of the extracts

The extracts obtained at varying temperatures (120–220 °C) were evaluated regarding their total phenolic content (TPC) and antioxidant abilities, namely the radical scavenging properties against $ABTS^{+\bullet}$, $DPPH^{\bullet}$, and ferric reducing antioxidant power assay. The TPC of the extracts tended to increase for temperatures greater than 120 °C ($S0 \geq 1.36$), with the exception of 180 °C, where similar values to 160 °C were observed (Fig. 4a). The increased TPC is probably associated with the higher radical scavenging properties against $ABTS^{+\bullet}$, $DPPH^{\bullet}$, and a greater capacity to reduce iron as indicated by the ferric reducing antioxidant power assay, as observed in Fig. 4(b-d). However, a slight decrease in antioxidant capacity was observed at 220 °C when compared to 200 °C. Notably, these results align with previous investigations conducted on other raw materials, where the maximum antioxidant capacity was observed at 200 °C, followed by a decrease [64]. Furthermore, it is well-accepted that the antioxidant capacity obtained after hydrothermal or autohydrolysis processing at different temperatures is dependent on the raw material used, as demonstrated by distinct studies[28,58].

The most extreme condition tested (T_{max} = 220 °C) yielded an extract containing 13.7 ± 0.4 g of gallic acid equivalents (GAE)/100 g (equivalent to 10.9 GAE/100 g dried seeds). The antioxidant capacity of this extract, as determined by TEAC and FRAP assays, was 17.3 ± 0.6 $g_{TroloxE}$/100 $g_{Extract}$ and 14.0 ± 0.18 g_{BHTE}/100 $g_{Extract,}$ respectively (equivalent to 13.8 and 11.2 GAE/100 g dried seeds, respectively). These antioxidant values were approximately 20 times higher than those reported for extracts obtained from three different cultivars of prickly pear seeds, obtained through acid extraction with HCl and methanol for 2 h at room temperature [13], and 8 times higher than those reported by Camarena-Ordonez et al. for *Opuntia rastrera*, where a maximum of 1.7 g gallic acid equivalents/100 g of dried seed was reported[12]. These results demonstrate the potential of the hydrothermal extraction methods. Regarding the use of subcritical water, this work achieved similar results to those reported by M.T. Munir et al., where around 8 $g_{Gallic\ acidE}$/100 $g_{Extract}$ was achieved for onion peels [51]. However, the total phenolic content obtained through hydrothermal processing of *Opuntia* seeds was considerably higher compared to other comparable raw materials such as wood and rice husks, which yielded 5.5 $g_{Gallic\ acidE}$/100 $g_{Extract}$ [68]. These findings corroborate the high amount of biologically active compounds present in *Opuntia* seeds.

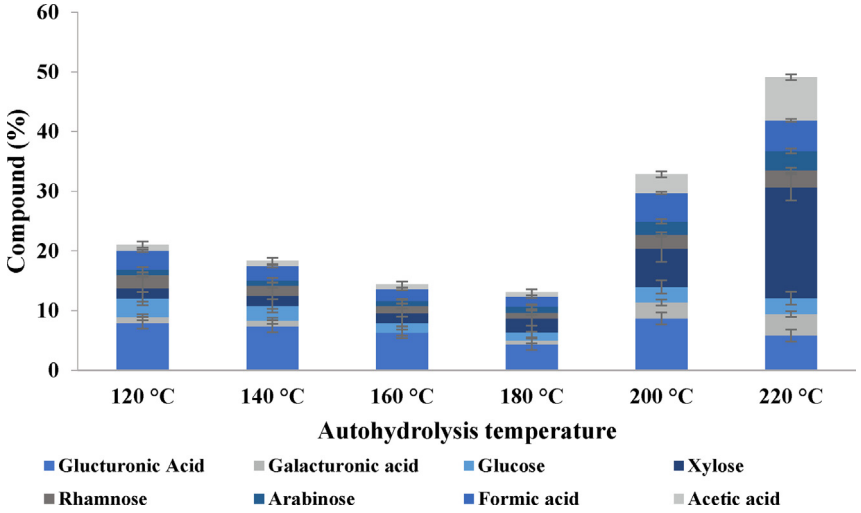

Fig. 2. Influence of autohydrolysis temperature on the saccharide composition and sugar degradation compounds of the soluble extracts of *Opuntia ficus-indica* seed pomace.

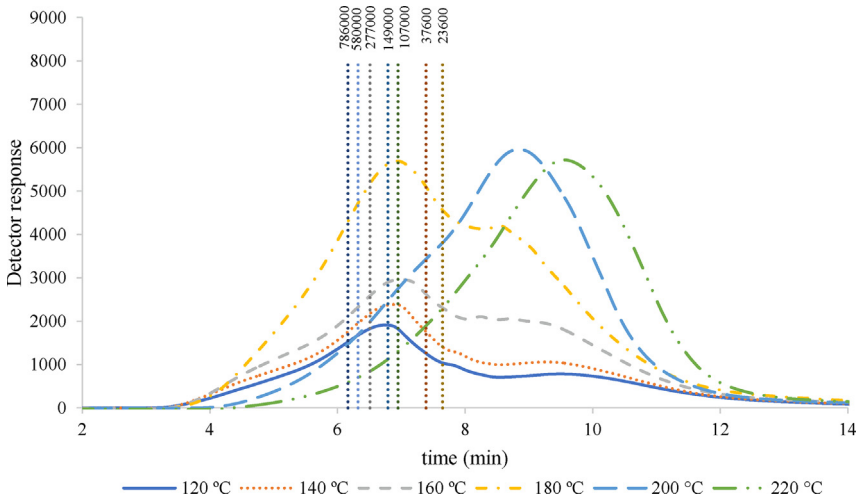

Fig. 3. High pressure size exclusion chromatography (HPSEC) of *O. ficus-indica* extracts obtained by autohydrolysis at different temperatures (120–220 °C).

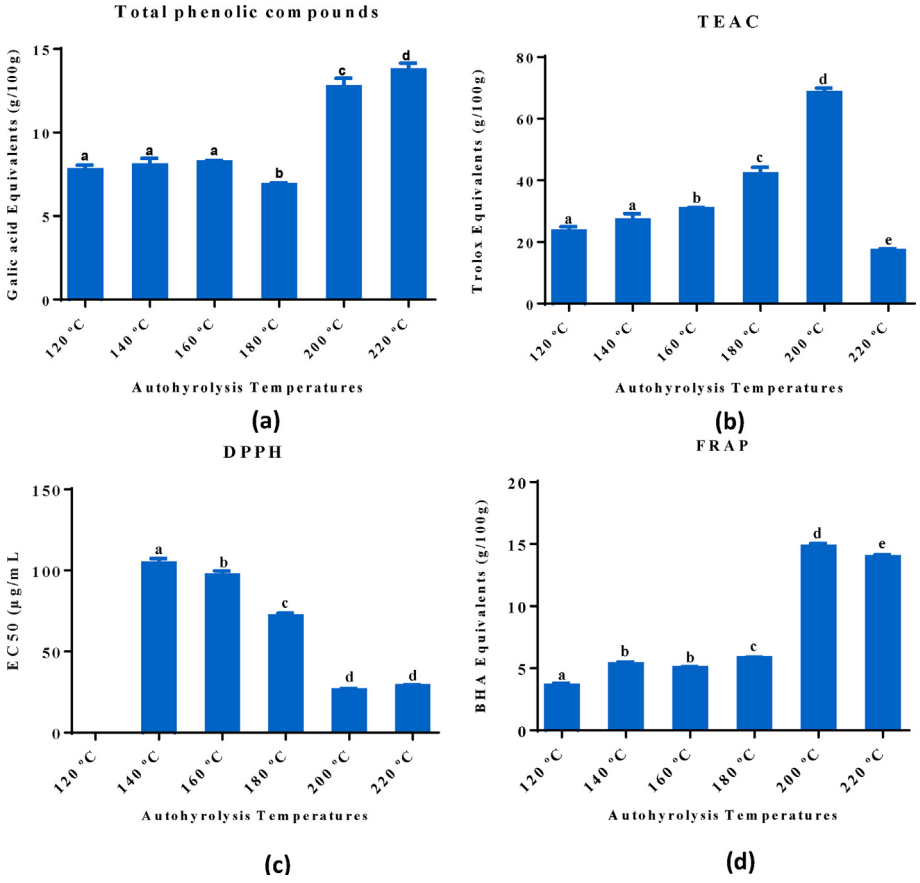

Fig. 4. Influence of autohydrolysis temperature on: (a) the total phenolic compounds of *Opuntia ficus-indica* seed pomace extracts and their antioxidant capacity given by: (b) TEAC in $g_{TroloxE}/100\ g_{Extract}$, (c) DPPH$^{\bullet}$ in IC_{50} (µg/mL) and (d) FRAP in $g_{BHTE}/100\ g_{Extract}$ scavenging assays. Results are presented as mean ± standard deviation and N = 3.

As for the antioxidant capacity given by the TEAC assay, DPPH$^{\bullet}$ scavenging assay and the FRAP assay there was an increase in the activity with temperature until 200 °C, where a maximum activity of 68.5 ± 1.4 $g_{TroloxE}/100\ g_{Extract}$, an IC_{50} of 26.1 µg/mL and 14.8 ± 0.28 $g_{BHTE}/100\ g_{Extract}$, respectively, was achieved. For extracts obtained at 220 °C and higher temperatures, and as reported by other authors using this methodology, there was a decrease in the antioxidant capacity, probably due to the degradation of some compounds with antiradical activity[8,64]. At 200 °C the results obtained for all the assays, were around 10 times higher than those reported for prickly pear seed extracts from three different cultivars and extracted with conventional methods (75 % acetone, 2 h agitation at 37 °C)[16]. The observed rise in total antioxidant capacity following exposure to high levels of heat may be attributed to the production of novel, or neoantioxidants, which are formed as a result of various chemical reactions, such as Maillard reactions, caramelization, and thermoxidation. This effect has been demonstrated in studies by Plaza and colleagues,

who observed changes in the overall antioxidant capacity of different macroalgae and plant materials after undergoing subcritical water extraction [63].

Although the levels of total phenolic compounds were maximum at a temperature of 220 °C, temperatures above that were not tried, as the anti-radical properties were lower at 220 °C than at 200 °C. As such, the increase in TPC would probably not be indefinite, as reported by Seo et al., during the subcritical water extraction of *Inonotus obliquus*. In their work, the authors noted an increase of TPC and antioxidant capacity in the range 50–250 °C for periods up to 60 min, and a reversed tendency for temperatures above that point and up to 300 °C, which corresponded to the maximal tested temperature[69]. This allows to conclude that the results obtained for hydrothermal processing depend on the raw material, as well as on the temperature used[69]. Autohydrolysis, a heat treatment, can convert insoluble to soluble phenolic compounds by cleavage of their covalent bound. So, the increased concentration of these compounds can be due to (i) changes in their extractability due to the disruption of the cell wall and liberation of antioxidant compounds from the insoluble portion of the seeds[69](ii) the formation of novel compounds with antioxidant properties, i.e., non-enzymatic browning reaction products, and (iii) deactivation of endogenous oxidative enzymes as reported for some mushroom species[19]as well as other seed and nuts[42].

Anti-inflammatory activity

In this work, the anti-inflammatory capacity of PPSP extract produced after autohydrolysis at 200 °C (PPSP_200) was measured in means of inhibition of COX-1 and COX-2 (Table 3). The results indicated that the IC_{50} values for COX-1 and COX-2 were 7117 μg/mL and 464 μg/mL, respectively. These values are notably higher compared to those of the control inhibitors Indomethacin (IC_{50} = 5.18 μg/mL) for COX-1 and Celecoxib (IC_{50} = 5.6 μg/mL) for COX-2, thus indicating that the inhibitory ability of PPSP_200 against these inflammatory enzymes is low. Nevertheless, it is noteworthy that the commercial products used as controls were in their pure state, while the extracts obtained in this study were aqueous extracts containing numerous other compounds. Furthermore, the inhibitory effects of *O. ficus-indica* extracts on COX-1 and COX-2 activity have been attributed to the presence of bioactive compounds, including betalains, flavonoids, and alkaloids. These compounds have been found to have potent anti-inflammatory and analgesic activities by modulating various cellular signaling pathways and molecular targets involved in the inflammatory response [4,43]. Previous research has demonstrated that O. *ficus-indica* seed and flowers hydroethanolic and aqueous extracts inhibits COX-1 and COX-2 activity in rats colonic mucosa, leading to a reduction in edema [6,10]. Additionally, a review article by Antunes-Ricardo et al. (2014) summarized the impact of O. *ficus-indica* extracts on the topical inhibition of COX-1 and COX-2 in human skin [15].

Anti-tyrosinase activity

Tyrosinase is an enzyme that plays a pivotal role in melanin production, the pigment responsible for skin coloration. Overproduction of melanin can result in hyperpigmentation, including age spots and melasma. Tyrosinase inhibitors are commonly utilized in skincare products to help reduce hyperpigmentation [50].

In this study, the impact of PPSP_200 on the inhibition of tyrosinase was investigated. The results showed that a concentration of 26500 μg/mL of O. *ficus-indica* extract reduced the enzymatic activity of tyrosinase by 50 % (IC_{50} = 26500 μg/mL). In comparison, the positive control, kojic acid, exhibited an IC_{50} of 55 μg/mL, indicating its superior inhibitory ability against hyperpigmentation enzymes. Therefore, these results suggest that the inhibitory potential of PPSP_200 against hyperpigmentation enzymes is relatively low. Additionally, there is evidence that high levels of tyrosinase activity are implicated in the development of aggressive melanoma. Therefore, reduction of this enzymatic activity has the potential to reduce the risk of skin cancer [72]. Similar results have been reported by other studies investigating *Opuntia dillenii* extracts from Penghu Island, Taiwan. These extracts inhibited melanin production in B16-F10 cells at a concentration of 20000 μg/mL without reducing cell growth or causing cellular death [18]. Other studies have also demonstrated the inhibitory effects of O. *ficus-indica* extracts obtained from different fruit parts and plantation sites [5,7].

Table 3
Anti-inflammatory and anti-tyrosinase activity of prickly pear seed pomace extract obtained after autohydrolysis extraction at 200 °C (PPSP_200).

	Anti-inflammatory activity		Anti-tyrosinase activity (IC_{50} μg/mL)
	COX-1 (IC_{50} μg/mL)	COX-2 (IC_{50} μg/mL)	
PPSP_200	7117	464	26,500
Indomethacin	5.18	–	–
Celecoxib	–	5.6	–
Kojic acid	–	–	55

Production of the body lotion

UVA/B filter and photoprotection factors

In order to understand the capacity of PPSP_200 to be used as a UVA/B filter, this was subjected to a UV scanning in the range 200–400 nm and simultaneously compared with 2-ethylhexyl 4-(dimethylamino) benzoate, i.e., Padimate *O* (Fig. 5). This compound is used as an active sunscreen agent in cosmetics and sunscreen drug products in concentrations up to 8 %, as regulated by the FDA. It absorbs light in the UV-B length (280–320 nm) to prevent photodamage[1]). However, recent investigations concluded that this compound may induce non-ligatable strand breaks on DNA *in vitro* and mutagenic effects on yeast i*n vivo* [34], leading to efforts to reduce the use of this substance.

As demonstrated in Fig. 5, the PPSP_200 extract obtained from O. *ficus-indica* exhibited a higher absorbance curve and peak for the wavelengths corresponding to the UV-B zone, when used at a concentration 10 times higher than the commercial Padimate *O*, resulting in levels of solar protection factors (SPF) measuring at 1.52 ± 0.05 as reported for other plant extracts[71]. Furthermore, it is noteworthy that these SPF levels remain relatively consistent even following a prolonged exposure period of 30 min under UV irradiation, particularly when utilized within the concentration range of 5–10 %. The extract also displays a high absorbance area for the UV-A wavelengths (320–400 nm) and even a slight curve for UV-C (200–280 nm). It is worth noting that the latter wavelength has less importance as it is absorbed by the ozone layer and oxygen present in the atmosphere[41]. In contrast, UV-A and UV-B wavelengths can penetrate the atmosphere, with UV-A accounting for approximately 95 % of the UV radiation reaching the Earth's surface. Due to its ability to penetrate deeper layers of the skin, UV-A is responsible for immediate tanning effects and contributes to skin ageing and wrinkling. Conversely, UV-B cannot penetrate the deeper skin layer (dermis and epidermis) but has higher energy, leading to sunburns and DNA damage, which can ultimately result in the development of skin cancer [57,77]. Therefore, it is crucial to incorporate compounds with high absorbance for both UV-A and UV-B wavelengths in sunscreens, such as the extract obtained in this study.

Considering the above properties, the PPSP_200 was used as an ingredient in a body lotion formulation to examine the potential of the final product as a photoprotective agent. The concentration of PPSP_200 used in the body lotion was 12.0 g per 100 g of body

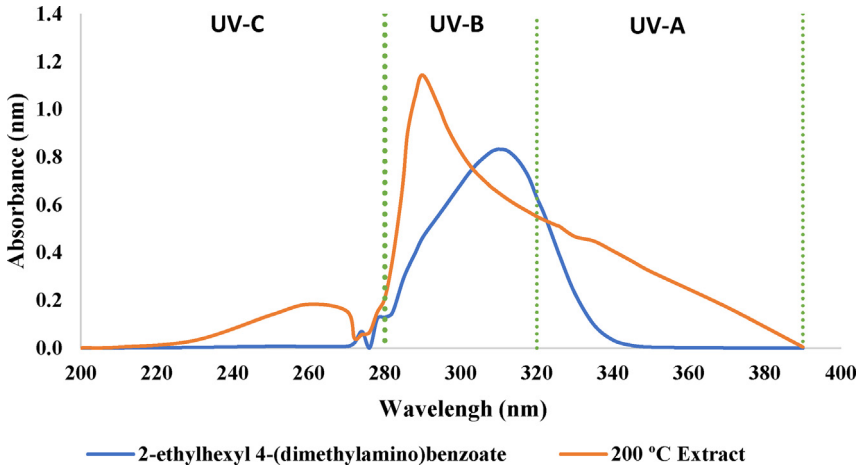

Fig. 5. UV wavelengths scanning (200–400 nm) to compare the absorbance of the 200 °C extract (12.3 $g_{Extrtact}$/ 100 g) with the commercial sunscreen 2-ethylhexyl 4-(dimethylamino) benzoate (1.0 g/100 g).

lotion. The main objective of the investigation was to determine the photoprotective properties of the body lotion, which were assessed and documented in Table 4.

The incorporation of the extract in the formulation resulted in the development of a body lotion manifesting a low sun protection factor (SPF) of around 8, aligning with the reported values of commercially accessible sunscreens with a low SPF, as well as body lotions that encompass sun protection[22,48]. This SPF value was maintained for about one hour, decreasing after two hours to approximately 6 SPF (data not shown). The reduced SPF value, despite the high absorbance curve of the extract, can be attributed to the high viscosity of the final product that may have hindered the cumulative effects of the extract. Nonetheless, the extract's incorporation allowed to produce a body lotion with additional biological characteristics, such as the ability to reduce the harmful effects of solar exposure. Specifically, the extract was found to reduce photocarcinogenesis, which represents the simultaneous and sequential biochemical events that ultimately lead to the occurrence of skin cancer [11]. Additionally, the produced lotion has been found to possess the ability to reduce immunosuppression, elastosis, and singlet oxygen formation. Elastosis is a skin condition that results from sun exposure and leads to the formation of yellowish skin with low elasticity. This reduction in elastosis indicates that the extract can prevent or slow down the skin's aging process[78]. Singlet oxygen formation, on the other hand, corresponds to an oxidative compound that contributes to photoaging. Therefore, the extract's capacity to decrease singlet oxygen formation indicates its potential as an anti-aging agent. Similar values of photoprotection factors were obtained by Monsalve-Bustamante et al., for a holist approach on B*accharis antioquensis* dried methanolic extract and hydrolyzed and purified methanolic extract (Yéssica A. [80]and by Harb et al., for Brazilian Beach–Cast Seaweeds aqueous or ethanolic (50 % w/v) extracts [36]. The extracts can be also use as booster of UV organic and inorganic filters since in addition to the UV screen capacity, antioxidant activity is added as cosmeceutical property.

Table 4
Photoprotection factors (PPF) of the body lotion produced with the addition of *Opuntia ficus-indica* seed pomace extract. Sun protection factor (SPF), protection factor against UV-A radiation (UVAPF) and other protection factors against other biological effects related to UV radiation: Photocarcinogenesis, immunosuppression, elastosis, singlet oxygen formation and photoaging. The values are expressed as mean ± standard deviation (n = 4).

Photoprotection factors (PPF)	
SPF	8.36 ± 0.53
UVAPF	5.61 ± 0.31
Photocarcinogenesis	8.79 ± 0.52
Immunosuppression	8.86 ± 0.53
Elastosis	5.37 ± 0.29
Oxygen singlet	8.08 ± 0.54
Photoaging	6.66 ± 0.41

Rheological properties

Fig. 6 illustrates the viscous characteristics of the body lotions under examination at a temperature of 25 °C. The control sample represents the body lotion produced without the incorporation of an antioxidant. The control + BHT sample corresponds to the body lotion with the inclusion of butylated hydroxytoluene, whereas the Ethylhexyl and the Selected extract samples denote the Control + BHT with the supplementation of 2-ethylhexyl 4-(dimethylamino) benzoate, or of PPSP_200, respectively. All formulated body lotion creams behaved like shear-thinning fluids, where the apparent viscosity decreased notably (more than three decades) with increasing shear rate. Similar viscous profiles were identified for all samples at low and intermediate shear rates (below 4 s^{-1}), which suggests similar consumer perception[37]. At higher shear rates only a power law tendency was maintained for 2-ethylhexyl 4-(dimethylamino) benzoate and the selected soluble extract. At fixed shear rate, the highest apparent viscosity was observed for body lotion incorporated with the selected hydrothermal extract, very close to the values identified for the control added with the BHT standard, followed by the control in the absence of antioxidant extracts and the ethylhexyl system. The values of apparent viscosity at the highest shear rates indicated that the newly formulated body lotion could be easily processed and spread across the skin[37], once the magnitudes and tendencies are consistent with those previously reported for creams enriched with different natural antioxidant extracts and with synthetic commercial counterparts (L. [46,47,54].

The developed body lotion in the presence of the selected extract, i.e., PPSP_200, exhibited the highest stability during storage time at room temperature (Fig. 6b), which can provide an indication of the possible mechanical stability of the sample during storage. It should be also highlighted that this body lotion required a short time (below 4 min) to achieve a steady viscosity, with a quick recovery after application (inset). Moreover, the proposed body lotion creams presented two relevant advantages from the processing, application, and storage point of view since neither the thixotropic effect in terms of apparent viscosity nor water

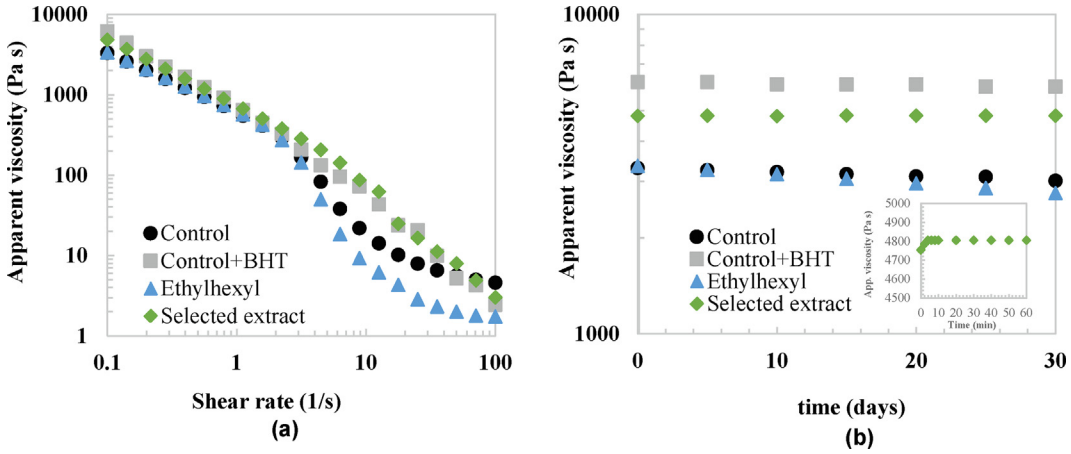

Fig. 6. Rheological properties of developed body lotion in terms of (a) steady-state shear and (b) time measurements. Control- body lotion formulated without the addition of an antioxidant; Control + BHT- body lotion formulated with the addition of butylhydroxytoluene (BHT). Ethylhexyl- body lotion formulated with the substitution of the solid UV filter by the commercial 2-ethylhexyl 4-(dimethylamino) benzoate. Selected Extract- body lotion formulated with the substitution of the solid UV filter by the PPSP_200.

release was identified after one month of storage at room temperature.

Skin irritation tests

O. *ficus-indica* has recently been used in the production of body lotions due to its purported skin benefits. Nevertheless, it is a common occurrence for numerous cosmetic products when containing harsh chemicals and synthetic fragrances to elicit symptoms such as erythema, pruritus, and xerosis [40]. So, in order to ensure the safety of a body lotion formulation containing prickly pear seed pomace (PPSP_200), SkinEthic™ skin irritation testing was performed to evaluate the potential impact of topical application on human skin. The results of the test indicated that the cream did not cause any skin irritation and was classified as UN GHS No category (non-skin irritant) with a 100 % viability rate, thus affirming its safety for topical use. Thus, *O. ficus-indica* body lotion could be considered an excellent option for individuals with sensitive skin or a propensity for allergies, as its phenolic compounds have potent antioxidant capabilities that can help alleviate skin irritation and reduce inflammation. Furthermore, it is free of harmful chemicals, such as parabens, phthalates, and sulfates, which are often present in many cosmetic products and can lead to various health problems, including skin irritation and allergic reactions [3,40].

Conclusion

Opuntia ficus-indica seed pomace (PPSP) was used as a raw material for an autohydrolysis process to extract total phenolic compounds and evaluate their antiradical properties. The optimal conditions for the autohydrolysis process were determined at 200 °C, with a total yield of 1.20 g/100 g. The resulting extracts contained high levels of bioactive compounds, such as phenols, and had a high antioxidant capacity, as well as a low anti-inflammatory and anti-tyrosinase activities. The oligomeric fractions solubilized composed mainly of glucuronic acid and xylose at higher temperatures had lower molecular weight due to thermal degradation of polysaccharides. Additionally, incorporating the PPSP_200 into body lotion models resulted in cosmetics with similar properties and rheology to their counterparts made with commercial antioxidants and with photoprotection. In conclusion, the autohydrolysis technology is an effective method for obtaining high yields of bioactive compounds from PPSP, which can be utilized in cosmetic formulations to provide antioxidant and other beneficial effects.

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CRediT authorship contribution statement

Ricardo M. Ferreira: Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation, Data curation, Writing – original draft. **Noélia Flórez-Fernández:** Data curation, Writing – review & editing. **Artur S. Silva:** Writing – review & editing. **Jorge A. Saraiva:** Writing – review & editing. **F.L. Figueroa:** Writing – review & editing. **J. Vega:** . **M. Dolores Torres:** Conceptualization, Investigation, Writing – review & editing. **Susana M. Cardoso:** Conceptualization, Writing – review & editing. **Herminia Domínguez:** Conceptualization, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jiec.2023.09.052.

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