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Research article

Valorization of agri-food waste through the extraction of bioactive molecules. Prediction of their sunscreen action

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ABSTRACT

The aim of this work was to identify the phenolic composition of 18 different vegetable residues and to determine the relationship between their phenolic compounds, antioxidant capacity and sun protection factor. For this purpose, samples of agri-food residues were analyzed to quantify their antioxidant capacity, total polyphenol and flavonoid content, sun protection factor and individual phenolic compounds through HPLC-DAD-FLD. Among the different phenolic compounds found in the extracts, the phenolic acids, especially caffeic acid, chlorogenic acid, *p*-coumaric acid and protocatechuic acid were the ones that have been most frequently identified, and, therefore, are present in a wide range of extracts. Black chai tea, lemon ginger tea and peanut extracts were the most antioxidant and photoprotective extracts. Phenolic compounds in the extracts have been found to contribute to their antioxidant activity and are closely correlated to their photoprotective capacity. A regression model that allows predicting the photoprotective capacity of any extract based on its total phenol content has been developed as a tool to determine the most suitable industrial application for each vegetable extract.

1. Introduction

The demand for fruits and vegetables has grown significantly in recent years due to dietary recommendations and dietary changes. In fact, in the period 2015–2019 the production of fruits and vegetables grew by 6.6% and the losses of this type of food also increased to the same extent, by 6.8% (FAOSTAT, 2022). Fruit and vegetable waste generated by food industry comprises the inedible parts of food that are discarded during collection, handling, transportation and processing. So, depending on the industry, the waste generated can be of a very different nature, being the most important ones stems, husks, seeds, peel, pomace, pulp, bagasse, etc. (Šelo et al., 2021). Vegetable processing industries annually generate about 600 million tons of fruit waste globally (Banerjee et al., 2017). In Europe, this sector generates around 90 million tons of residues per year, and it is expected that this production increases in the next years (Montenegro-Landívar et al., 2021a). One of the main concerns about these residues is their contribution to environmental pollution due to their high biodegradability. The anaerobic biological degradation of vegetable residues is the third largest anthropogenic source of methane atmospheric emissions (Breeze, 2018). Another very important issue related to the generation of food waste is the economic loss that it entails. According to FAO estimates, these losses can reach approximately $ 940 billion per year (Food Loss and Waste Protocol, 2016). Food waste is also of great concern in terms of social effects. In contrast to this global context of increasing food demand and waste, close to one billion people are chronically undernourished (Porat et al., 2018). Therefore, a decisive change in the agri-food system is necessary for the proper management of these by-products. In this context, the circular economy promises to be an efficient option in the medium and long term to avoid, reuse or recover natural resources and by-products derived from this industry (Del Rio Osorio et al., 2021).

Due to their high content of bioactive compounds, plant-derived foods have beneficial effects on health. Among the different bioactives, phenolic compounds stand out for their high antioxidant potential (Montenegro-Landívar et al., 2021b). In fact, epidemiological studies have shown that diets rich in these compounds can help cancer prevention, as well as neurodegenerative and cardiovascular diseases,

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among others (Montenegro-Landívar et al., 2021a). Moreover, these biomolecules generally absorb in the UV region and are able to act as UV filters protecting photosynthetic tissues from damage (Harborne and Williams, 2000). Research on the photoprotective capacity of plant extracts is very interesting since it can be oriented to very different fields of application. It is increasingly important to protect the skin against the action of sunlight, not only to prevent the spot appearance or skin aging, but also to prevent more serious issues, such as skin cancer. If the cosmetic products developed for this purpose are prepared with natural ingredients, they will be better received by consumers who are increasingly looking for this type of "natural products" (Ibrahim et al., 2022). Likewise, in recent years, there has been a growing interest in antioxidant additives of natural origin in order to both replace the synthetic ones, such as butylated hydroxyanisole - BHA or butylated hydroxytoluene – BHT (Olszewska et al., 2020), and to protect food against light effects, delaying the initiation of oxidative reactions that cause food rancidity (Tamkutė et al., 2021). In addition, in recent years, there has been a growing interest in the incorporation of agri-food waste extracts in active packaging films (Azman et al., 2022). Such innovative application could become very important for the food sector as it can prevent both microbial contamination and oxidation of food, thus prolonging its shelf life (Esposito et al., 2020; Kanatt, 2020; Han and Song, 2021). Finally, some drugs or other active principle, dietary or nutraceutical products … could also be target products for the incorporation of this type of sun shield compounds.

Due to the large number of beneficial properties associated with phenolic compounds, the recovery of those substances from agri-food waste has become a primary target in the higher-value biorefinery options, and a large number of investigations is being conducted in this regard (Makris and Şahin, 2019). However, the high heterogeneity of the different raw materials makes necessary to conduct a comprehensive characterization of each by-product in order to determine its composition and identify their potential uses. In view of this, the aim of this work is to identify and compare the phenolic composition of extracts from 18 different vegetable residues and to determine the relationship between their phenolic content, antioxidant capacity and sun protection factor. Therefore, the novelty of this work is to contribute to the knowledge of the phenolic composition of different agri-food residues and its influence on their antioxidant and photoprotective capacity, which has been little studied so far. In this way, the potential application of each of the extracts in a specific industrial sector (nutraceutics, pharmaceutics, cosmetics, functional food) could be determined.

2. Material and methods

2.1. Obtaining extracts from plant by-products

Different agri-food by-products were collected from household wastes. On one hand, by-products that are generated in considerable quantities in Spain were selected for this study: tomato (peel), potato (peel), cabbage, eggplant (peel and stem), strawberry (leaves), pear (peel), kiwi (peel), garlic (white peels), orange (peel), tangerine (peel), beet (leaves, peel and stems). On the other hand, widely generated by-products whose consumption is widespread in Europe were also considered for this study: avocado (peel and pit), peanut (shell) and commercial herbal teas, previously used to make infusions: Hornimans® black chai tea (tea, cinnamon, cardamom, ginger, aromas, cloves, chicory aniseed, pepper), and Pompadour® lemon ginger tea sachets (ginger, lemongrass, lemon peels, lemon myrtle, licorice). Finally, residues of a less common product, but with recognized beneficial properties for health, was also selected, as its phenolic composition has been little explored so far: leaves of the *Moringa oleifera* tree, marketed in form of powder by the herbalist Sanct Bernhard (Barcelona, Spain) as food supplement.

All these the residues were dried in an oven at 30 °C on filter paper. Once dried, they were ground and sieved through a 300 μm sieve to obtain a homogeneous particle size. In all cases, a mixture of ethanol: water (96:4 v/v) was used as extraction solvent with a solid:liquid ratio of 1:100 (w/v). The extraction was carried out in a stove at 40 °C for 24 h under orbital stirring (250 rpm). The resulting mixture was centrifugated (8000 rpm, 15 min) using a Sorvall ST 8 centrifuge (Thermo Scientific, Waltham, MA, USA) and filtered. The solvent was removed from the extract by rotatory evaporation. Then, the extract was resuspended in a small volume of water, frozen and finally freeze-dried. The resulting dry extracts were kept refrigerated until analysis.

2.2. Characterization of extracts

2.2.1. Antioxidant capacity

Three methods were used to determine the antioxidant capacity of the obtained extracts: the FRAP (Ferric Reducing Ability of Plasma) assay, proposed by Benzie and Strain (1996); the DPPH (2,2-diphenyl-1-pycrilhydracyl) radical scavenging assay, outlined by Brand-Williams et al. (1995); and the ABTS (2,2′-azinobis (3-ethylbenzothiazoline-6-sulphonic acid)) method, based on the description made by Re et al. (1999). These procedures have also been previously adapted and optimized by our research group for the analyses of grape stem extracts (Esparza et al., 2021). Briefly, for the calibration curve, Trolox was used as standard in a concentration range of 0.05–1.19 mM, 0.05–0.67 mM and 0.05–2.07 mM for FRAP, DPPH and ABTS methods, respectively. The wavelengths used to measure the absorbance by the three methods were 595, 517 and 734 for FRAP, DPPH and ABTS, respectively. All samples were measured with a UV/Vis spectrophotometer (Jenway 7315, Staffordshire, UK).

All extracts were processed and analyzed in triplicate by the different spectrophotometric methods. The determination coefficient obtained for the calibration curve was $R^2 > 0.996$ in all cases (see calibration parameters in Table S1 of the Supplementary Material). The results of antioxidant capacity were expressed as mmol Trolox equivalents/g dry extract.

2.2.2. Total polyphenol and flavonoid content

The total polyphenol content (TPC) of the extracts was determined by the Folin Ciocalteu method described by Singleton et al. (1999). The calibration curve was prepared from gallic acid in concentrations ranging from 0.21 to 4.15 mM ($R^2 > 0.997$, see calibration parameters in Table S1 of the Supplementary Material). For sample measurement, 0.1 mL of the gallic acid standard or sample were mixed with 0.5 L of the Folin-Ciocalteu reagent, 7.9 mL of deionized water and 1.5 mL of Na_2CO_3 (20% w/w). After 2 h in darkness, the absorbance of the resulting solutions was measured at 765 nm. All extracts were processed and analyzed in triplicate, and results were expressed as mmol of gallic acid equivalents/g dry extract.

The total flavonoid content (TFC) was determined through a colorimetric method proposed by Chandra et al. (2014). For the calibration curve, quercetin standards in concentrations ranged between 3.03 and 30.30 μg/mL ($R^2 > 0.998$, see calibration parameters in Table S1 of the Supplementary Material) were prepared. For sample analysis, 1.5 mL of the standard or sample (prepared in the same way than for TPC analysis) were mixed with 1.5 mL of a 2% $AlCl_3$ solution (prepared in 5% acetic acid). After 30 min in darkness, absorbance was measured at 420 nm. All extracts were processed and analyzed in triplicate, and results were expressed as mg quercetin equivalents/g dry extract.

2.2.3. Sun protection factor (SPF)

The photoprotective properties of the extracts were determined by calculating the sun protection factor (SPF) and by measuring the absorbance in the ultraviolet range. The FPS was determined spectrophotometrically using an *in vitro* method developed by Mansur et al. (1986). This method allows obtaining the SPF value by means of a simplified formula [1] that takes into account the erythemogenic effect of radiation, the intensity of sunlight and the absorbance of the sample

at wavelengths corresponding to the UVB-UVA range (290–320 nm). All extracts were prepared in triplicate at a concentration of 0.07 mg/mL in methanol before analysis.

$$spectrophotometric\ SPF = CF \cdot \sum_{290}^{320} EE(\lambda) \cdot I(\lambda) \cdot abs\ (\lambda) \quad [1]$$

CF: Correction factor (10).

EE (λ): erythemal efficiency spectrum.

I (λ): solar simulator intensity spectrum

abs (λ): absorbance value of the sample at the defined wavelength.

The values of the product EE(λ) x I(λ) used in the present work for the calculation of the SPF data where the normalized values defined by Sayre et al. (1979).

In addition, in order to determine the SPF of the extracts, a wavelength scan was registered for each extract sample in the entire ultraviolet region (200–400 nm).

2.2.4. Identification and quantification of polyphenolic compounds using HPLC-DAD-FLD

Identification and quantification of the polyphenolic compounds present in the extracts were carried out with a Waters chromatograph (Milford, MA, USA) equipped with two 510 pups, a 717 Plus autosampler, a 996-photodiode array detector and a 474-fluorescence detector. The column used for the analysis was a reversed-phase column (Zorbax Eclipse Plus C18, 250 × 4.6 mm, particle size of 5 μm, Agilent, Santa Clara, CA, USA). Prior to the analysis of the extracts, between 26.6 ± 0.1 and 32.2 ± 0.1 mg of each sample were weighted and dissolved in 350 μL of methanol with the aid of an ultrasonic bath (Ultrasons-HD, Selecta, Barcelona, Spain). Subsequently, samples were filtered through 0.45 μm PTFE syringe filters. Each extract was processed and analyzed in triplicate.

The chromatographic separation of the different polyphenols was conducted according to a modified method of Barros et al. (2014). HPLC quality solvents were used to prepare the two mobile phases required in this method: A (water:formic acid 85%, 99.9:0.1 v/v) and B (acetonitrile:formic acid 85%, 99.9:0.1 v/v), with the following gradient: (time in min, % A): (0, 95%), (15, 85%), (22, 80%), (25, 80%) (35, 70%), (45, 50%), (50, 5%), (55, 95%) y (60, 95%). Acetonitrile was from PanReac AppliChem (Barcelona, Spain) and formic acid from Scharlab (Barcelona, Spain). The flow rate was 1 mL/min and the injection volume 10 μL. The column temperature was set at 30 °C. The identification of each phenolic compound in the extracts was carried out by double comparison of the UV–Visible spectrum at the characteristic wavelength of the compound and the retention time of the standard. For compounds with similar retention time and UV–Visible spectrum, standard additions were conducted in order to unambiguously identify the compound in a specific sample. In those cases, where compounds with similar retention time and maximum absorption wavelength are present in the same extract and overlap in the chromatogram, only identification, not quantification, could be performed. Data were processed using Empower 2.0. For the quantification of the compounds, calibration curves were prepared for each standard. Considering the great variability of samples analyzed, in some cases it was necessary to prepare two different calibration ranges for a compound, so that samples with low concentrations were quantified in the low range one and those with high concentrations in the high range one. The determination coefficients of the calibration curves used were $R^2 > 0.99$ in all cases. The detection limit (LLOD) was calculated by the following expression: $LLOD = 3.3 \cdot \sigma / S$, were S is the slope of the calibration curve and σ is the standard deviation of the regression line. The quantification limit (LLOQ) was the lowest concentration included in the calibration range. The calibration parameters obtained for each specific compound can be found in Table S2 of the Supplementary Material.

2.3. Statistical analysis

Data are expressed as the mean ± standard deviation (SD). Kruskal-Wallis test and the Nemenyi post-hoc procedure were applied to study differences in the variables of interest among extracts. Beside this, a regression model was fitted to explain the behavior of SPF by means of the antioxidant capacity and TPC. In all cases, $p < 0.05$ was considered to be statistically significant. All data processing was conducted by using the statistical package R (R Foundation, Vienna, Austria).

3. Results and discussion

3.1. Extraction yield

It is known that the extraction yield and the antioxidant capacity of the extracts depend to a great extent on the extraction solvent (Moure et al., 2001), and on the extraction method. For this reason, and in order to obtain comparable results, the same solvent and extraction protocol were used to process all the food by-products analyzed. In this way, the differences found in yield and bioactive content were due exclusively to the different nature and composition of the residues used. Considering the wide variety of vegetable by-products used in this work as source of bioactive compounds, the resulting extraction yields were very different among extracts, ranging from 4.54 ± 1.35% in the case of garlic peel extract to 60.37 ± 2.26% for kiwi peel extract (see Fig. S1 of the Supplementary Material). The comparison of the results obtained in the present study with other works is very difficult as each study use different extraction protocol. Despite of this, the yield obtained by Kallel et al. (2014) in the extraction of garlic husk by conventional solid-liquid extraction method and using ethanol as solvent was 4.0 ± 0.25%, which agrees with the yield obtained from the same source in the present work.

3.2. Antioxidant capacity, TPC and TFC

Table 1 shows the results of the spectrophotometric analysis performed on all samples. Due to the fact that the estimation of the antioxidant capacity is strongly dependent of the reactive oxygen species (ROS) or reactive nitrogen species (RNS) employed in the assay, this parameter has been measured by three different assays: DPPH, ABTS and FRAP. The three methods used revealed that, among the extracts obtained in the present study, the one with the highest antioxidant potential was the black chai tea extract, followed by lemon ginger tea and peanut extracts, while kiwi, cabbage, avocado and pear extracts depicted the lowest antioxidant capacity values.

On the other hand, in most of the extracts, the highest values of antioxidant capacity correspond to those obtained by the ABTS method, while values obtained by FRAP were of the same order than the ones obtained by DPPH. This agrees with previous studies conducted on other plant extracts (Barros et al., 2014; Jiménez-Moreno et al., 2019). Considering that ABTS and DPPH methods share the same mechanism of action, the limited steric accessibility to the radical site of the DPPH molecule could explain the lowest values observed when using this method (Xie and Schain, 2004). These differences could also be attributed to the absorption spectrum of the extracts themselves, which could overlap with that of DPPH at 517 nm and, therefore, interfere with the results, leading to default errors. Nevertheless, when the estimation of the antioxidant capacity is calculated as percentage of radical scavenging activity or as ascorbic acid equivalents, the ABTS method does not always depict the maximum values of antioxidant capacity of plant extracts (Smuda et al., 2018). This makes comparisons of the antioxidant potential of extracts obtained by different authors even more difficult, and highlights the importance not only of the appropriate selection of the antioxidant capacity method to be used, but also of the way it is carried out, as this has a significant impact on the final results.

It is well known that the antioxidant assays used in this work estimate the overall antioxidant activity of the extracts, which includes all

Table 1
Antioxidant activity, total phenolic content (TPC) and total flavonoid content (TFC) of extracts from different agri-food by products (Mean ± SD).

Extracts	FRAP[a]	DPPH[a]	ABTS[a]	TPC[b]	TFC[c]
Cabbage	<LLOQ a	0.03 ± 0.00 ab	0.13 ± 0.01abcd	0.09 ± 0.01abc	0.93 ± 0.04abc
Eggplant	0.26 ± 0.01abcd	0.28 ± 0.01abcd	0.32 ± 0.01abcd	0.30 ± 0.01abc	2.11 ± 0.24abcd
Orange	0.09 ± 0.01 abcd	<LLOQ a	0.19 ± 0.01abcd	0.18 ± 0.01abc	3.00 ± 0.14abcd
Potato	0.15 ± 0.01abcd	0.12 ± 0.00abcd	0.25 ± 0.02abcd	0.19 ± 0.00abc	0.64 ± 0.01 a
Kiwi	0.03 ± 0.00 a	0.02 ± 0.00 a	0.04 ± 0.00 a	<LLOQ a	0.93 ± 0.06 ab
Peanut	0.73 ± 0.04bcd	0.73 ± 0.03bcd	1.06 ± 0.06bcd	1.08 ± 0.09bc	38.50 ± 1.85abcd
Garlic	0.33 ± 0.01abcd	0.26 ± 0.01abcd	0.74 ± 0.02abcd	0.50 ± 0.04abc	1.42 ± 0.01abcd
Tangerine	0.20 ± 0.00abcd	0.08 ± 0.00abcd	0.39 ± 0.01abcd	0.34 ± 0.03abc	5.29 ± 0.12abcd
Pear	0.06 ± 0.00 abc	0.05 ± 0.00abcd	0.09 ± 0.00 ab	0.07 ± 0.00 ab	1.21 ± 0.02abcd
Lemon ginger tea	0.97 ± 0.02 cd	0.80 ± 0.05 cd	1.56 ± 0.13 cd	0.95 ± 0.07bc	29.09 ± 1.71abcd
Black chai tea	1.22 ± 0.02 d	1.45 ± 0.06 d	2.54 ± 0.03 d	1.43 ± 0.01 c	70.79 ± 1.21 cd
Avocado	0.12 ± 0.00abcd	0.06 ± 0.00abcd	0.12 ± 0.01abc	<LLOQ a	12.19 ± 0.35abcd
Moringa oleifera	0.29 ± 0.00abcd	0.21 ± 0.01abcd	0.32 ± 0.01abcd	0.37 ± 0.00abc	72.26 ± 3.57 d
Beet leaves	0.19 ± 0.01abcd	0.13 ± 0.00abcd	0.30 ± 0.01abcd	0.41 ± 0.02abc	47.54 ± 0.98bcd
Beet peel	0.11 ± 0.00 abcd	0.10 ± 0.00abcd	0.14 ± 0.00abcd	0.10 ± 0.01abc	1.35 ± 0.06abcd
Beet leaves, peel and stems	0.14 ± 0.01abcd	0.13 ± 0.01abcd	0.22 ± 0.01abcd	0.20 ± 0.01abc	15.43 ± 1.09abcd
Strawberry	0.55 ± 0.04abcd	0.52 ± 0.03abcd	0.78 ± 0.08abcd	0.47 ± 0.04abc	18.07 ± 1.81abcd
Tomato	0.08 ± 0.00abcd	0.07 ± 0.00abcd	0.16 ± 0.02abcd	0.12 ± 0.01abc	3.68 ± 0.17abcd

Different letters in the same column indicate significant different results among extracts (significance level: 0.05). In all cases, n = 3.
[a] mmol Trolox equivalents/g of dry extract.
[b] expressed as mmol gallic acid equivalents/g dry extract.
[c] Expressed as mg quercetin equivalents/g dry extract.

the sample constituents (polyphenols, ascorbates, reducing sugars, carotenoids, pigments, terpenes, tocopherols and others) that can act as antioxidants. However, when analyzing the total phenolic content (TPC) of the different extracts, it was found that the extracts with the highest TPC coincided with those with the highest antioxidant capacity (black chai tea, lemon ginger tea and peanut extracts), and the extracts with the lowest TPC were also the ones with the lowest antioxidant capacity (kiwi, pear, cabbage and avocado extracts). Moreover, significant Pearson's correlation coefficients were obtained between the ABTS, DPPH and FRAP values and the TPC results of the extracts (0.951, 0.952 and 0.966, respectively; see Table S3 of the Supplementary Material). This good correlation indicates that polyphenols could be the main responsible for the antioxidant capacity of the different extracts studied in this work. The strongest correlation was found when the antioxidant activity was determined by FRAP method, what means that this method is more closely related with total phenolic content of the extracts. These results agree with Yu et al. (2021), who also found strong relationships between antioxidant capacities and total phenols, being the FRAP method the best correlated with TPC.

Fig. 1 represents the correlation heatmap between the results obtained through the different spectrophotometric methods used in this work. Fig. 1a shows the raw correlations between all the spectrophotometric variables analyzed in all the extracts. While Fig. 1b depicts the partial correlogram, which reflect the quality of the previously mentioned correlation: when sharp color changes are observed from raw correlations (correlogram 1a) to partial correlations (correlogram 1b), it means that the correlation is highly dependent on the effects of other variables, so there is not a real direct correlation. Again, it can be confirmed that there exist good correlations between antioxidant activity and TPC values (Fig. 1). On the other hand, lower correlations were found for the antioxidant activities and TFC values (Table S3). Moreover, there is a striking change in sign in the partial correlogram in the case of the correlation found between the TFC values and those of antioxidant capacity, especially when the FRAP and ABTS methods were used (Fig. 1b).

This lack of correlation of TFC with the antioxidant capacity was also described by other authors (Suleria et al., 2020; Yu et al., 2021). In addition, previous studies performed on grape stem extracts (Jiménez-Moreno et al., 2019) showed that there is a low correlation between the total flavonoid content and the concentration of the different flavonoids found by HPLC-DAD in the same samples, which questions the validity of such method for estimating the flavonoid content in complex matrices such as vegetable by-product extracts. In

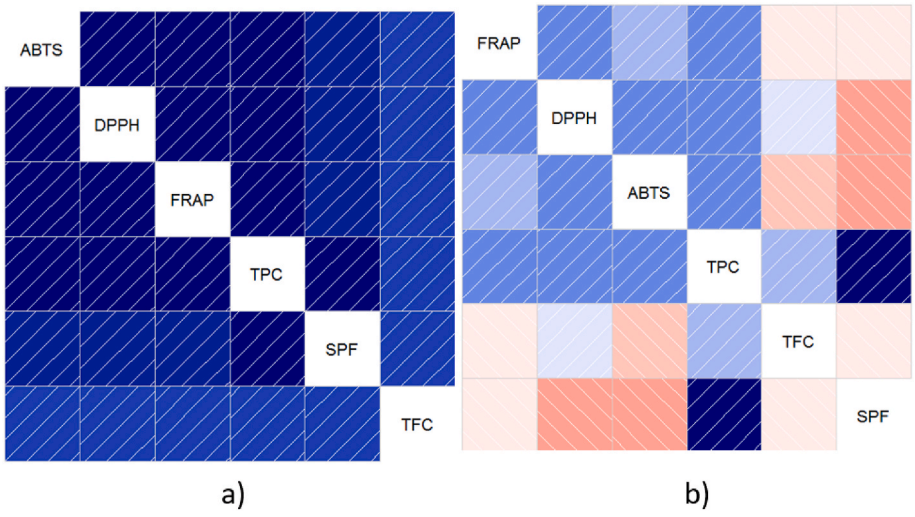

Fig. 1. Correlation heatmap of spectrophotometric data. a) Pearson's raw correlations; b) Pearson's partial correlations. (Dark blue color represents Person's correlation coefficients higher tan 0.9). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

this regard, Pękal and Pyrzynska (2014) reported that the $AlCl_3$ method, in neutral or acidic medium, is selective only for flavonols and luteolin-derived flavones, which could explain these incongruent results. Furthermore, glycosylated flavonoids cannot be determined by this method as the sugar moiety hinders the chelation with aluminum chloride (Denni and Mammen, 2012). Therefore, results obtained in this work support the theory that the TFC values obtained by this assay do not provide reliable information on the antioxidant properties of the extracts. Further studies should be carried out to determine if this is only due to the selectivity of the method or whether the presence of other components in the extracts could produce synergistic or antagonistic reactions with flavonoids and/or $AlCl_3$, leading to misleading results. Finally, it is necessary to find a simple and fast method for flavonoid estimation, given the growing importance that these compounds are acquiring due to their high bioactivity.

3.3. Photoprotective capacity of the extracts

Plant extracts contain an extensive variety of natural compounds (polyphenols, lycopene, vitamin C, carotenoids, etc.) with a wide range of UV absorption capacity, which makes them good candidates for sunscreen formulation. In fact, there are a large number of botanicals approved for its use in cosmetics for skin protection (Ngoc et al., 2019). The UV absorption spectra of the extracts obtained in this work were very varied (see some examples in Fig. S2 of the Supplementary Material). Some of them absorb in a wide range of wavelengths (i.e. peanut and garlic extracts), while others absorb in a narrow region of the spectrum (i.e. black chai tea), and others practically do not absorb in the UV region (i.e. kiwi extract). In view of this, and in order to know their photoprotective potential for future applications, the sun protection factor was determined for all the extracts analyzed in the present study. As it can be seen in Fig. 2, peanut and black chai tea extracts presented the highest SPF values, while kiwi and beet peel extracts gave the lowest values. This is in agreement with the UV absorption spectrum found for those extracts.

In vitro SPF values obtained in this study are useful to estimate the potential of the extracts as photoprotective agents. However, these values depend not only on the extract, but also on the amount of it used for the determination. Therefore, it is very difficult to compare the SPF values of the different extracts with literature data, as each research work has been conducted following different protocols (for both the extraction and the SPF determination). In the present study, all the extracts have been prepared and analyzed following the same protocol, so it is possible to establish comparisons among them. On the other hand, SPF values in cosmetic products are generally in the range of 6–50+ (Ngoc et al., 2019), what could serve as reference in order to estimate the potential of the different extracts for cosmetic applications. In view of this, it could be concluded that both peanut and black chai tea extracts could be used in sunscreens at a concentration of 0.07 mg/mL, but the rest of them should be prepared in higher concentrations to improve their SPF values. Nevertheless, it should also be noted that standard photoprotection parameters, such as SPF, do not fully reflect the effects of non-conventional sunscreens such as botanical extracts or other antioxidants (Matsui et al., 2009). This is because these substances are also able to reduce the damage caused by ROS, preventing or reducing skin damage. Reis Mansur et al. (2016) demonstrated that the addition of plant leaf extracts to an emulsion, despite not improving its *in vitro* SPF values, produced a significant increase in the *in vivo* effect of the formulation. Thus, in view of the *in vitro* SPF values obtained for the extracts at low concentrations and their antioxidant capacity results, it can be considered that these extracts from agroindustry by-products may be of great interest in the cosmetic sector as skin photoprotective agents.

Furthermore, plant extracts with high antioxidant capacity can prevent photodegradation of sunscreens by increasing their photostability (Cerqueira-Coutinho et al., 2015). In addition, their incorporation to food to improve the oxidative stability of meat products or other foods rich in lipids (Moure et al., 2001) should not be ruled out. In this regard, we found a significant correlation between the antioxidant capacity values and SPF results of the different extracts (Fig. 1a and Table S3 of the Supplementary Material), but partial correlograms (Fig. 1b) reveal that such correlation is highly dependent on other variables, so there is a weak direct correlation. The same effect was observed in the correlation between SPF and TFC. However, it is particularly noticeable the strong correlation that exists between TPC and SPF values of the extracts, which highlights the dependence of the SPF values on the TPC ones. In Fig. S3 of the Supplementary Material, the TPC and SPF of all the extracts are shown overlapping, and it clearly shows the high correlation that exists between both variables. These results agree with Ebrahimzadeh et al. (2014), who studied 20 different extracts from medicinal plants and also found good correlation between SPF and phenolic content, but no correlations between SPF and flavonoid content (measured by the $AlCl_3$ method) or antioxidant activity. In any case, it is well known that flavonoids have a considerable photoprotective effect, as well as a high antioxidant capacity. Thus, this lack of correlation also supports the idea that the $AlCl_3$ method in such complex extracts does not provide reliable information on the content of flavonoid compounds.

The correlation between SPF and TPC values found in this study was so strong that it had to be the determining factor in any regression model between SPF values and the rest of the spectrophotometric parameters.

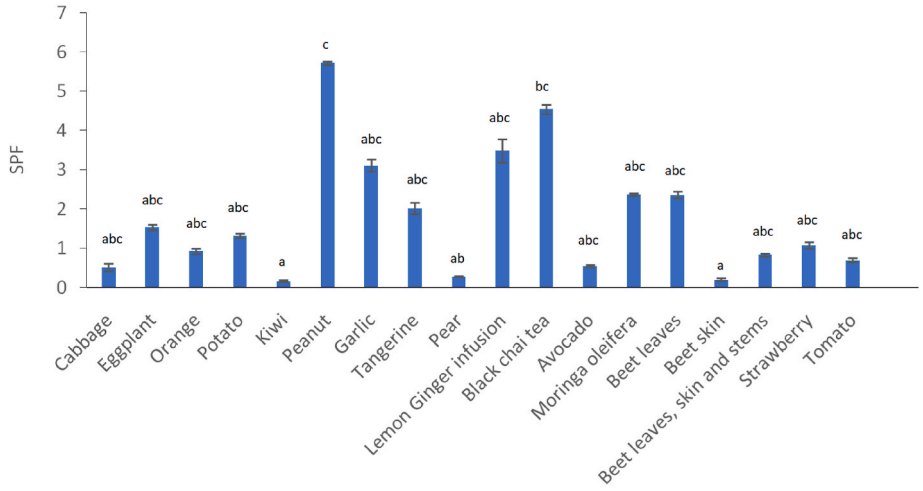

Fig. 2. Sun Protection Factor (SPF) of the different extracts obtained in this work at a concentration of 0.07 mg/mL.

However, since the increase in the antioxidant capacity of the extracts also involved an increase in the SPF values, we decided to include also this effect in the regression model. After considering different models, a logarithmic one was found that provided a valid strategy to estimate SPF values from those of TPC and DPPH:

$$Log(SPF) = \beta_0 + \beta_1 \cdot Log(TPC) + \beta_2 \cdot Log(DPPH) + \beta_3 \cdot Ei + \varepsilon$$

where *Ei* takes the value of 1 when estimating the values of extract *i*, and 0 for the rest of the extracts, and ε is the error term. The values of the different coefficients of the linear regression model are collected in Table 2.

This model allows concluding that for each 1% of increase in TPC values, a significant improvement of at least 0.5% of SPF values can be expected. Besides this, significant coefficients for pear, strawberry and tangerine indicate that, with respect to avocado extract, mean values of SPF are significantly smaller for pear and strawberry and significantly larger for tangerine. Beet peel data were excluded from the regression because they resulted in an unstable model with heteroscedasticity issues. Considering the rest of extracts, the fitted model gave an R^2 of 0.9976, which indicates that the SPF values estimated from TPC and DPPH values were very close to the experimental SPF values (see Fig. S4 of the Supplementary Material). This good fitting demonstrates, for the first time, that it is possible to estimate the sun protection factor of an extract from its total polyphenol content and antioxidant capacity values.

3.4. Phenolic compounds

Tables 3 and 4 collect the 24 different compounds identified in the 18 extracts analyzed in this study. As it can be seen, each extract presented different composition and none of them contained all the phenolic compounds together. As previously explained, it is very difficult to compare the phenolic content of different extracts from literature data, as each research work has been conducted following different protocols. Nevertheless, some similarities can be found with other works. For instance, most of the studies conducted in potato peel extracts found chlorogenic acid as the main phenolic compound, followed by caffeic acid (Sampaio et al., 2020), what agrees with the results found in this work for potato extracts. In addition, it has been shown that the most abundant phenolic compound in eggplant is chlorogenic acid (5-*O*-caffeoylquinic acid) (Alarcón-Flores et al., 2015), which is consistent with the high content of this phenolic acid in the eggplant by-product extract found in the present work.

Table 2
Statistic parameters for the linear regression model.

	Coefficient	Std. Error	t value	Pr (>|t|)
β_0 [a]	1.67501	0.63049	2.657	0.01236*
β_1	0.49445	0.15104	3.274	0.00261**
β_2	0.36681	0.22318	1.644	0.11037
β_3				
Beet leaves	0.37891	0.26115	1.451	0.15685
Beet leaves, peel and stems	−0.30678	0.18654	−1.645	0.11015
Black Chai tea	−0.47620	0.71591	−0.665	0.51086
Cabbage	0.09793	0.17797	0.550	0.58609
Eggplant	−0.18817	0.34229	−0.550	0.58643
Garlic	0.28833	0.36536	0.789	0.43600
Kiwi[b]	−0.08090	0.26241	−0.308	0.75991
Lemon ginger tea	−0.31976	0.58804	−0.544	0.59049
Moringa oleifera	0.24128	0.31153	0.775	0.44450
Orange	0.30805	0.20841	1.478	0.14947
Peanut	0.14651	0.58166	0.252	0.80279
Pear	−0.54194	0.07054	−7.683	$1.15{\cdot}10^{-8}$***
Potato	0.21847	0.16903	1.293	0.20573
Strawberry	−0.99938	0.47741	−2.093	0.04459*
Tangerine	0.47860	0.2229	2.173	0.03758*
Tomato	−0.02203	0.08026	−0.274	0.78557

Significant codes: 0 "***"; 0.001 "**"; 0.01 "*".

[a] β_0 includes the expected SPF value for "Avocado" extracts, so there is no β_3 value for this extract in the regression equation.

[b] The values corresponding to the third replicate of the kiwi extract have been removed because they affect the fit and destabilize the model (outliers).

Among the different phenolic compounds identified in the extracts, the phenolic acids, especially caffeic, chlorogenic, *p*-coumaric and protocatechuic acids, were the most frequently identified and, therefore, those that were present in a wider range of extracts. On the other hand, among all the extracts, the ones with highest number of phenolic compounds identified were lemon ginger tea and potato extracts, while kiwi and beet extracts presented only one of the phenolic compounds each, and at low concentrations, which explains that both extracts presented low antioxidant capacity and SPF values. By contrast, some extracts with a high number and/or concentration of phenolic compounds identified (such as eggplant and potato extracts) were not the extracts with highest antioxidant capacity and/or FPS values.

Besides, the composition of the three extracts with the highest antioxidant capacity and FPS values (black chai tea, lemon ginger tea and peanut extracts) was quite different. Thus, the black chai tea extract showed a high content of gallic acid, a quercetin derivative and epicatechin, while the lemon ginger tea extract contained catechin as the main component, but in a lower concentration (less than half) than that of the main components of black chai tea extract. The peanut extract was the one with the least number of identified compounds of the three, with vanillic acid as the major component. Despite these differences, the antioxidant capacity of the peanut extract was similar to that of the lemon ginger tea extract, and its SPF is even higher than that of the other two extracts. These results reveal that the complex mixture of compounds present in these extracts makes difficult to identify the molecules that most contribute to the antioxidant activity and sun protection factor. This agrees with Baldisserotto et al. (2018) who also consider that the identification of the molecules responsible of the activity of a vegetable raw material is difficult due to the complex mixture of compounds contained in it. Moreover, although individual phenolic molecules may present significant antioxidant capacity, some of them are capable of transferring electrons, in addition to hydrogen atoms, to other antioxidants, favoring their chemical regeneration (Palafox-Carlos et al., 2012). Therefore, the antioxidant capacity of an extract will depend not only on the concentration of its antioxidant compounds, but also on the structure of these compounds and the interactions with other components. These interactions may include synergistic and/or antagonistic effects, the formation of stable intermolecular complexes or irreversible reactions between the different components of the extract (Olszowy-Tomczyk, 2020).

Nevertheless, and in spite of the above, if we consider the known relationship between chemical structure and antioxidant capacity of polyphenols, we can find certain correlation between the presence of some components and the SPF and antioxidant activity values of the extracts analyzed in this work. Thus, taking into account that catechol group in the B ring is one of the most determining structural factors on the antioxidant activity of a flavonoid (Quideau et al., 2011), and that it also can be improved with a double bond in position 2–3 in conjunction with the 4-oxo group on the carbonyl of ring C and the presence of hydroxyl groups in positions 3 and 5 (Yordi et al., 2012), it can be considered that, among the analyzed flavonoids, catechins and quercetins are the most powerful antioxidants. This could partially explain the high antioxidant capacity of black chai tea and lemon ginger infusion extracts found in the present work. Moreover, the high content of gallic acid in the black chai tea could contribute to its high antioxidant values, as this molecule contains three hydroxyl groups in its structure and it is known that both phenolic hydroxyl and methoxyl groups significantly improve its free radical scavenging ability (Chen et al., 2020). However, further studies would be necessary to evaluate which phenolic compounds are the main responsible for the antioxidant activity of each extract, as well as to know their possible interactions with other phenolic and non-phenolic compounds present in the extracts.

Table 3
Phenolic acids (μg/mg dry extract) of the extracts.

Extracts	Caffeic acid	Ellagic acid	Ferulic acid	Gallic acid	Coumaric acid	Protocatechuic acid	Vanillic acid	Syringic acid	Neochlorogenic acid	Chlorogenic acid	Cinnamic acid
Cabbage	nd	nd	0.117 ± 0.003^{ab}	nd	0.005 ± 0.001^{a}	nd	nd	nd	**	nd	nd
Eggplant	nd	nd	0.036 ± 0.002^{a}	0.030 ± 0.002^{a}	0.023 ± 0.004^{ab}	nd	nd	nd	0.150 ± 0.020^{ab}	23.309 ± 2.316^{b}	nd
Orange	0.145 ± 0.023^{ab}	nd	nd	nd	0.017 ± 0.002^{ab}	nd	nd	nd	nd	nd	nd
Potato	1.297 ± 0.086^{b}	nd	0.152 ± 0.010^{ab}	nd	0.031 ± 0.002^{ab}	**	0.067 ± 0.005^{a}	0.026 ± 0.013^{ab}	0.515 ± 0.065^{ab}	6.013 ± 0.189^{ab}	nd
Kiwi	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Peanut	nd	nd	nd	nd	nd	0.010 ± 0.001^{a}	0.494 ± 0.053^{b}	nd	nd	nd	nd
Garlic	1.539 ± 0.428^{b}	nd	nd	nd	0.065 ± 0.009^{b}	0.070 ± 0.007^{ab}	0.090 ± 0.021^{ab}	0.221 ± 0.046^{b}	nd	nd	nd
Tangerine	0.474 ± 0.033^{ab}	nd	0.322 ± 0.019^{b}	nd	0.038 ± 0.006^{ab}	nd	nd	nd	nd	0.273 ± 0.011^{ab}	nd
Pear	nd	nd	nd	nd	nd	nd	nd	nd	nd	1.802 ± 0.385^{ab}	nd
Lemon ginger tea	0.060 ± 0.006^{ab}	nd	nd	0.288 ± 0.031^{ab}	nd	0.104 ± 0.010^{ab}	<0.053*	nd	0.045 ± 0.004^{a}	0.328 ± 0.019^{ab}	nd
Black chai tea	nd	0.066 ± 0.017^{ab}	nd	4.150 ± 0.540^{b}	nd	nd	nd	nd	nd	nd	0.177 ± 0.009
Avocado	nd	nd	nd	0.091 ± 0.003^{ab}	nd	0.377 ± 0.019^{b}	nd	0.015 ± 0.001^{a}	nd	nd	nd
Moringa oleifera	nd	nd	nd	nd	nd	nd	nd	nd	9.006 ± 0.824^{b}	nd	nd
Beet leaves	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Beet peel	nd	<0.013*	nd	nd	nd	nd	nd	nd	nd	nd	nd
Beet leaves, peel and stems	nd	0.017 ± 0.001^{a}	nd	nd	nd	nd	nd	nd	nd	nd	nd
Strawberry	nd	1.660 ± 0.115^{b}	nd	nd	nd	nd	nd	nd	nd	nd	nd
Tomato	0.040 ± 0.006^{a}	nd	nd	0.057 ± 0.010^{ab}	nd	0.085 ± 0.002^{ab}	nd	nd	nd	0.130 ± 0.004^{a}	nd

Results are expressed as mean ± SD except in the cases when the quantification was not possible because values were below the quantification limit (*) or due to interferences that compromise the resolution of the peak making possible only the identification of the compound (**). Different letters in the same column indicate significantly different results among the extracts (Nemenyi, significant level: 0.05). nd, not detected.

4. Conclusions

This work presents an exhaustive characterization of 18 extracts obtained from several agri-food by-products. Black chai tea, lemon ginger tea and peanut extract showed the highest TPC and antioxidant capacity values, while kiwi, pear, cabbage and avocado had the lowest. It has been demonstrated that phenolic compounds from food waste extracts could provide an important photoprotective action. In fact, a regression model that relates the total phenolic content (TPC) of the extracts with their photoprotective action (SPF) has been found. This model can be very useful to estimate the photoprotective potential of extracts from food waste in different economic sectors, either in the formulation of sunscreen lotions or as preservative additives that prevent or delay the deterioration by action of light of food, drugs or other kind of active ingredients. For these reasons and, although this work provides relevant data on the composition of plant by-products, further work is needed to evaluate the effect of the interactions between the different polyphenols present in each extract on their overall antioxidant and photoprotective capacity. In consequence, the identification of the main phenolic compounds responsible of these beneficial properties could be achieved.

Finally, this works highlights the big opportunities that vegetable by-products from the agri-food industry offer to obtain phenolic compounds, which could also be a step towards the circular economy. Food waste valorization has a double advantage. On the one hand, it contributes to alleviating the important environmental, economic and social problems related to the increase in food waste due to the constant increase in population and food demand that currently exists. On the other hand, it allows obtaining compounds of natural origin with high added value and great demand by consumers, which also have strong potential for innovative applications in the food, pharmaceutical, nutraceutical and cosmetic industries.

Credit author statement

Blanca Martínez Inda: Formal analysis, data curation, original draft preparation; **Irene Esparza**: Conceptualization, methodology, data curation, original draft preparation, writing-review and editing; **Jose Antonio Moler:** Statistical analysis; **Nerea Jiménez-Moreno:** Conceptualization, methodology, data curation, original draft preparation, writing-review and editing; **Carmen Ancín-Azpilicueta:** Conceptualization, methodology, original draft preparation, writing-review and

Table 4
Phenolic composition (μg/mg extract) of the extracts excluding phenolic acids that are shown in Table 3.

Extracts	Apigenin	Epicatechin	Epigallocatechin	Catechin	Quercetin	Quercetin-derivate	Taxifolin (dihidroquercetin)	Ishoramnetin	Tyrosol	Malvidin-3-glucoside	Procyanidin B1	Procyanidin B2	Resveratrol
Cabbage	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Eggplant	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Orange	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Potato	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Kiwi	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.031 ± 0.008[a]	nd
Peanut	0.200 ± 0.011[b]	nd	nd	nd	nd	nd	<0.041*	nd	nd	nd	nd	nd	nd
Garlic	nd	nd	nd	nd	**	nd	nd	nd	0.073 ± 0.004[a]	nd	nd	nd	nd
Tangerine	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Pear	nd	nd	nd	nd	nd	0.377 ± 0.072[ab]	nd	0.007 ± 0.002[a]	nd	nd	nd	0.073 ± 0.004[b]	nd
Lemon ginger tea	0.019 ± 0.002[a]	nd	nd	0.908 ± 0.037[b]	nd	nd	nd	nd	0.378 ± 0.021[b]	nd	nd	nd	nd
Black chai tea	nd	2.111 ± 0.400[b]	0.603 ± 0.141	nd	nd	2.539 ± 0.214[b]	nd	nd	nd	nd	nd	nd	nd
Avocado	nd	nd	nd	0.188 ± 0.013[a]	nd	0.092 ± 0.002[a]	nd	nd	nd	nd	nd	nd	nd
Moringa oleifera	nd	nd	nd	nd	0.049 ± 0.005	0.621 ± 0.092[ab]	nd	0.013 ± 0.004[a]	nd	nd	nd	nd	nd
Beet leaves	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	<0.036*	nd	nd
Beet peel	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.008 ± 0.001	nd	nd	nd
Beet leaves, peel and stems	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	<0.043*	nd	nd
Strawberry	nd	0.067 ± 0.009[a]	nd	**	nd	1.405 ± 0.112[ab]	nd	<0.005*	0.221 ± 0.087[ab]	nd	nd	**	nd
Tomato	nd	nd	nd	**	nd	nd	nd	nd	nd	nd	nd	nd	0.026 ± 0.001

Results are expressed as mean ± SD except in the cases when the quantification was not possible because values were below the quantification limit (*) or due to interferences that compromise the resolution of the peak making possible only the identification of the compound (**). Different letters in the same column indicate significantly different results among the extracts (Nemenyi, significant level: 0.05). nd, not detected.

editing, funding acquisition.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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