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Cookies enriched with coffee silverskin powder and coffee silverskin ultrasound extract to enhance fiber content and antioxidant properties

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ABSTRACT

Coffee is one of the most significant beverages consumed worldwide. However, the substantial production and consumption of coffee has led to the generation of large amounts of by-products, such as coffee silverskin (CS).

The first objective was to study the ultrasound-assisted extraction (UAE) conditions from CS according to a simple factorial design, in order to obtain natural extracts as a source of polyphenols and caffeine with high antioxidant activity. The second objective was to include CS powder (CSP) or ultrasound CS extract (UCSE) in the elaboration of cookies, in order to obtain an enriched food product with potential health benefits for consumers.

CS was characterized in terms of moisture, protein, lipids, ash, total dietary fiber, total phenol content (TPC) and antioxidant activity. The UCSE was characterized in terms of extraction yield, TPC, caffeine content and antioxidant activity (ABTS and ORAC assays).

The best UCSE used for cookie elaboration was obtained at 60 min and 180 W with the following values: 8.8 % wt; 36.8 mg GAE/g; 62.7 μmol caffeine/g; 491.1 μmol/g (ABTS assay); 1012.4 μmol/g (ORAC assay). Finally, the cookies were sensory and chemically characterized. In the cookies containing UCSE the sensory acceptability was not modified with respect to the control cookies and an increase in TPC and antioxidant activity was achieved. However, the incorporation of CSP lead to a decrease in the acceptability despite the fact that the cookies constitute a source of fiber. Results reinforce the use of green extraction technologies to obtain antioxidants compounds from natural sources.

1. Introduction

Coffee is one of the most widely consumed beverages in the world, Brazil being the largest producing country followed by Vietnam, Colombia and Indonesia (Shahbandeh, 2022). According to the International Coffee Organization (ICO), the estimated total production of coffee in the year 2021/22 was 10 million tons worldwide, while the consumption of the beverage was increased by 3.3% in the same period, showing a steady growth along the last years (ICO, 2022). Although there are more than 120 known wild species of coffee plants, only two are produced for trade and consumption: *Coffea arabica* (Arabica) and *Coffea canephora* (Robusta). Among these, Arabica accounts for a slightly larger share of global production, with 55%–60% of the world's supply (Bozzola et al., 2021).

Throughout the different stages of coffee processing and industrialization, which primarily include harvesting, pulping, fermentation, hulling, roasting, and brewing (Bevilacqua et al., 2023), tons of by-products or residues with specific characteristics are generated. Approximately 90% of the coffee cherry is discarded during processing as agricultural waste or by-product. Coffee silverskin (CS), the thin tegument that covers the outer layer of the green coffee bean, is the only by-product of the coffee roasting step, representing 4-5% (w/w) of the bean (Martuscelli et al., 2021). Coffee roasting usually takes place in non-producing countries, so the generation of this discard is widespread around the world. Recent studies have highlighted the potential use of CS as a supplement in the food sector due to its high nutritional profile. It contains a significant amount of proteins (11-19%), is rich in dietary fiber (up to 74%), and has low fat content (<4%)(Arya et al., 2022;

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Gemechu, 2020; Iriondo-deHond et al., 2020a; Narita & Inouye, 2014; Nzekoue et al., 2022). A broad range of health-promoting effects have also been attributed to this by-product, including anti-inflammatory, antidiabetic and anticholesterolemic (del Castillo et al., 2016; Iriondo-DeHond, Amaia Herrera & del Castillo, 2019). These benefits are associated to the presence of bioactive compounds, among which caffeine and chlorogenic acid outstand as the most abundant phenolics with high antioxidant activity (Fernandez-Gomez et al., 2016; Nolasco et al., 2022). The presence of bioactive peptides has also been reported, including angiotensin-converting enzyme (ACE), inhibitory peptides, antioxidants, and dipeptidyl peptidase (DPP) IV inhibitors with promising uses for the treatment of diabetes and its side effects (Nolasco et al., 2022). Other classes of bioactive compounds including vitamins, minerals, and phytosterols have been assessed in CS (Nzekoue et al., 2022), confirming its nutritional value and potential for revalorization as a functional ingredient.

One of the main concerns regarding food safety aspects that could become a limitation for CS use as a food ingredient is contamination with Ochratoxin A (OTA), a mycotoxin produced by *Aspergillus* and *Penicillium* fungi species (Khaneghah et al., 2019). Limits for OTA concentrations are established for roasted coffee beans, ground roasted coffee, and soluble coffee (EU, 2022). According to Viani (2002), most of the toxin concentrates in the skin and the risk of contamination increases significantly when the beans are exposed to high water activity (a_w) levels. Adequate storage conditions of the beans (a_w<0.7) prevents fungal growth and can be achieved by maintaining relative humidity (RH) during storage near 14 % (Barros et al., 2011). Another concern for using coffee byproducts as food ingredients is related to caffeine content in the final product. According to the European Food Safety Agency (EFSA), daily intake of caffeine should not exceed 400 mg for general population, and 200 mg for lactating women (Iriondo-deHond et al., 2020). Hence, this factor should be considered when designing a new product with CS as ingredient.

CS could be used as such within a product formulation, for example, substituting part of the flour in bakery goods to increase total dietary fiber content (Ateş & Elmaci, 2019; Pourfarzad et al., 2013), or, from a biorefinery point of view, specific components can be recovered and further used as natural extracts with high added value (Bondam et al., 2022). Effective valorization of CS depends on the extraction methods that achieve a quantitative extraction of the components of interest (Gil-Martín et al., 2022). In this sense, a wide range of extraction methods has been reported for extracting phenolic compounds from solid coffee residues. These methods include both traditional techniques like maceration, as well as novel and greener approaches such as ultrasound, microwaves, high pressure, and deep eutectic solvents (Strieder et al., 2023). Among them, ultrasound-assisted extraction is of particular interest, as extraction improvements attributed to the propagation of ultrasound pressure waves, which induce cavitation phenomena. This cavitation effect breaks down the matrix leading to a higher penetration of the solvent in the cell structure, higher mass transfer rates, shorter extraction times and higher extraction yields (Kumar et al., 2021).

Given the global concern about reducing the amount of waste generated by the coffee industry and the significant environmental pollution hazard posed by discarding CS, it seems appropriate to explore alternatives for its valorization. This would contribute to achieving a more sustainable production in line with the principles of a circular bioeconomy.. In this context, the aim of this study was to assess different options for incorporating CS in the development of sweet cookies. This ranged from the direct incorporation of coffee silverskin powder (CSP) to the addition of an ultrasound CS extract (UCSE), with the goal of creating an enriched food product with potential health benefits for consumers. Finally, sensory and chemical analyses were performed on the cookies.

2. Materials and Methods

2.1. Raw material collection, pre-treatment and characterization

Coffee silverskin from Arabic (*Coffea Arabica*) and Robusta (*Coffea Canephora*) varieties (blend) was provided by Nestlé (Uruguay). As the CS sample was in the form of pellets, a pre-treatment of grinding and drying was necessary for its processing, since the initial moisture content (AOAC 23.003) was of 19.3 %. CS was dried in a convection oven at 40°C, until reaching a_w<0.7 (Aqua LAB 4 TE), and subsequently ground in a domestic processor. The dry CS was stored at -18°C protected from light until later use. Determination of Ochratoxin A levels (<1.5 μg/kg) was performed according to (Hernández et al., 2006). The dried CS was characterized in terms of protein (AOAC 955.04), lipid (AOAC 991.36), ash (AOAC 920.153), total dietary fiber (AOAC 985.29), total phenolic content (Folin-Ciocalteau), and antioxidant activity (ABTS assay). For the total phenol content (TPC) and the antioxidant activity, an extraction with ethanol/water 1:1 was performed according to Silva et al. (2021). In the lipids, the fatty acid profile was analyzed by gas chromatography on a Shimadzu GC-2014 (Kyoto, Japan), equipped with FID and capillary column SP 2560 (100 m x 0.25 mm x 0.2 μm). The temperature program was: initial temperature 160°C, heating at 4°C/min until 230°C, and holding at 230°C for 10 min. Peak identification was accomplished through the analysis of authentic standards (Sigma-Aldrich).

2.2. Ultrasound-assisted extractions (UAE)

The effect of two independent variables was studied using a 3^2 simple factorial design in order to obtain ultrasound CS extracts with high antioxidant potential, leading to a total of nine experimental conditions. The studied factors were extraction time (15, 30 and 60 minutes) and power (54, 108 and 180 Watts), while response variables were extraction yield, total phenol content, and antioxidant activity of the extracts. The extractions were carried out in Falcon tubes placed in a laboratory-scale ultrasonic bath equipment, model Elmasonic P 60 H (Elma Schmidbauer GmbH, Singen, Germany), working at 37 kHz ultrasonic frequency. Considering the results reported in previous studies, an ethanol-water mixture (75:25) was used as solvent in order to maximize antioxidant activity and TPC extraction (Costa et al., 2014; Nzekoue et al., 2022). The ratio sample/solvent used was 50 mg CS/mL (taking into account the study reported by Iriondo-Dehond et al., 2020b). The temperature during the process was maintained at a constant 30 °C. The temperature inside the bath was regulated by a circulating water system connected to the equipment to prevent the degradation of thermolabile compounds (Kumar et al., 2021). For each experimental condition, the extraction was performed in triplicate. After extraction, the tubes were centrifuged (5000 rpm, 10 minutes) and supernatant was collected for further evaporation of the solvent under nitrogen flow. Dried extracts were preserved at -18 °C protected from light until analysis.

2.3. Extract characterization

The obtained extracts were characterized in terms of yield (% w/w), TPC, caffeine content and antioxidant activity.

To determine the TPC and the antioxidant activity of the extracts, a preparation of the sample was made, which consisted on an extraction with 80:20 methanol/water solution. Briefly, 1 mL of the ethanol/water 80:20 (v/v) extraction solution were added to 5 mg of extract sample and shaken for 1 minute (Vortex shaker). The eppendorf tube was placed in an ultrasonic bath for 15 minutes at room temperature and then centrifuged at 5000 rpm for 25 min to obtain the supernatant phase. All the samples were prepared in duplicate and the assays were performed at least in triplicate.

2.3.1. Determination of the total phenol content

Total phenol content was determined by Folin-Ciocalteau as described by Singleton et al. (1999) and modified by Fernández--Fernández et al. (2019) using a calibration curve with gallic acid (0.05-0.50 mg/mL). In a microplate, 10 μL of the polyphenolic extract or standard solution was combined with 200 μL of Na_2CO_3 (20 % w/v). After 2 minutes, 50 μL of Folin solution (1/5) was added and the reaction was left to proceed in the dark for 30 minutes. The absorbance reading was performed at 750 nm in a Multiskan^{TM} Go microplate spectrophotometer (Thermo Fisher Scientific, Waltham, Massachusetts, USA). The results were expressed as mg GAE/g of extract.

2.3.2. Determination of the caffeine content

The detection of caffeine in the extracts was performed by HPLC using a quaternary pump coupled to a diode array detector, (Dionex Ultimate 3000, ThermoScientific). Chromatographic separation of caffeine was performed using an analytical reverse- phase C18 column (Luna, 250 × 4.6 mm, 5 μm) at 1mL/min with detection at 280 nm and 320 nm. The chromatography was conducted using mobile phase consisting of H_2O/0.1% formic acid (A) and acetonitrile/0.1% formic acid (B), with the following gradient: 0 min: 8% B; 0.1-25 min: 8-15%B; 25-55 min: 15-22% B; 55-60 min: 22-40% B; 60-70 min: 40% B; 70-71 min: 40-8% B; 71-75 min: 8% B. For the quantification, a calibration curve was prepared with caffeine standard (Sigma-Aldrich) in water at concentrations ranging from 10 to 200 μM. Results were expressed as μmol caffeine/g.

2.3.3. Determination of antioxidant capacity (in vitro)

The antioxidant capacity of the extracts was determined by the ABTS radical method and the ORAC-FL method. The ABTS test was performed according to Re et al. (1999) and modified by Fernández-Fernández et al. (2019) using Trolox as a standard. An activated solution of ABTS 7mM was added to a specific volume of ethanol until an absorbance of 0.7 (750 nm) was achieved. Then, 10 μL of polyphenolic extract or standard was placed on the microplate and 190 μL of ABTS working solution was added. At 10 minutes, the absorbance reading was performed at 750 nm in a Multiskan^{TM} Go microplate spectrophotometer (Waltham, Massachusetts, USA). The ORAC-FL (Oxygen Radical Absorbance Capacity) test was performed according to Ou et al. (2001) and modified by Dávalos et al. (2005). Fluorescence measurements were made at excitation wavelengths of 485 nm and emission wavelengths of 520 nm on a Varioskan^{TM} Lux multimodal plate (ThermoScientific, Massachusetts, USA). A calibration curve with Trolox was used. The results were expressed as μmol eq. Trolox (TE)/g extract.

2.4. Cookie elaboration

For the cookie elaboration, the extract with the highest yield, TPC, and antioxidant activity was chosen. To define the different formulations, preliminary elaborations were carried out considering different objectives. For the cookies containing CSP the goal was to achieve the claim "source of fiber", meaning that fiber content should be of 3 g/100 g product or 2.5 g per portion (MERCOSUR, 2012). For the cookies with UCSE, it was determined that a 30 g portion of cookies should provide a total phenolic content equivalent to that of a cup of green tea (73.9 mg GAE/100 mL) based on Klepacka (2022). Therefore, five different formulations of cookies were elaborated: Control (C) cookies without CSP or UCSE in the formulation; cookies with the UCSE at two different concentrations defined in previous studies: 0.8 % (C-UCSE-1) and 1.2 % (C-UCSE-2); cookies with CSP at 11.9 % (C-CSP) and finally, one formulation with both CSP (11.9 %) and UCSE (0.6 %) denominated C-CSP-UCSE. The cookies were baked for 12 minutes at 180 °C. In the formulations with the incorporation of CSP or UCSE, the content of sugar and vanilla was adjusted in order to avoid the bitter taste. In Table 1 are listed and quantified the ingredients of each formulation.

Table 1
Ingredients used for each formulation of cookies.

Ingredients	C	C-CSP	C-UCSE-1	C-UCSE-2	C-CSP-UCSE
Flour (g)	45.5	30.3	34.1	34.0	30.0
Sugar (g)	23.2	27.7	31.2	31.1	27.7
Oil (g)	16.2	14.9	16.8	16.7	14.9
Egg (g)	12.1	11.2	12.6	12.6	11.2
Vanilla (g)	1.4	2.5	2.8	2.8	2.5
Baking powder (g)	1.3	1.2	1.4	1.4	1.2
Salt (g)	0.3	0.3	0.3	0.3	0.3
CSP (g)	-	**11.9**	-		**11.9**
UCSE (g)	-	-	**0.8**	**1.2**	**0.6**

2.5. Chemical and sensory characterization

The cookies were characterized in terms of moisture (AOAC 23.003), protein (AOAC 955.04), lipid (AOAC 991.36), ash (AOAC 920.153), total dietary fiber (AOAC 985.29), total phenolic content (Folin-Ciocalteau), and antioxidant activity (ABTS assay). For TPC and ABTS assays, samples were extracted with DMSO/H_2O.

Regarding the sensory evaluation, a consumer study (n = 60) was carried out to evaluate acceptability of the five samples of cookies, with regular consumers. Cookies of identical shape and size (Fig. 1) were coded and presented to each consumer in random order. They were then asked to evaluate the acceptability of each sample using a nine-point hedonic scale with Spanish legends as reported by Curia et al. (2001). Following that, they were requested to complete a CATA (check--all-that-apply) question by selecting terms that described each sample from a list of 35 terms related to flavor, texture, and appearance. The terms used were: very light color, adequate color, very dark color, with dark spots, nice color, strange color, vanilla smell, toasty smell, coffee smell, butter smell, strange smell, burnt smell, nice smell, little crisp, proper crispiness, too crispy, very soft, suitable hardness, too hard, rough, dry, low sweetness, adequate sweetness, too sweet, vanilla flavour, nice flavour, roasted flavour, butter flavour, smooth flavour, intense flavour, coffee flavour, strange taste, burnt taste, bitter and aftertaste.

2.6. Statistical analysis

All the analytical methods were carried out at least in triplicate. The data obtained for each assay were analysed by analysis of variance (ANOVA) and Tukey's test to determine the existence of significant differences between the samples. Infostat version 2020e software was used. For UAE extractions, a regression analysis was performed considering power and time as independent variables and Extraction Yield, Total Phenols and Antioxidant Activity as response variables (R Core Team, 2021).

For sensorial studies, analysis of variance (ANOVA) was conducted on the overall liking data considering sample as variation source. Tukey's test was used to determine statistically significant ($p < 0.05$) differences between samples. The frequency of use of each CATA term was determined by counting the number of consumers who used that term to describe each sample. Cochran's Q test and Bonferroni test were carried out to identify significant differences among samples for each of the sensory terms.

3. Results and Discussion

3.1. Coffee Silverskin characterization

Nutritional composition of the CS used for this study is presented in Table 2. As expected, the major component is dietary fiber (70.2 % db), which constitutes one of the main reasons for using this by-product as a food ingredient. There is abundant evidence supporting that an adequate consumption of dietary fiber leads to several health benefits,

Figure 1. Codified samples of cookies for sensory studies.

Table 2
.Chemical composition, total phenol content and antioxidant capacity of CS.

Parameter	Value
Protein* (% w/w)	12.8 ± 0.7
Lipids* (% w/w)	2.7 ± 0.1
Total Dietary Fiber* (% w/w)	70.2 ± 1.7
Ash* (% w/w)	4.9 ± 0.1
Total Phenols content (mg GAE/g CS)	11.1 ± 0.3
Antioxidant capacity ABTS (μmol TE/g CS)	49.8 ± 5.0

Results are expressed as mean ± SD (*n=3*)
* dry basis.

including maintenance of a healthy gut microbiota, reduced risk of colorectal cancer, type-2 diabetes and cardiovascular disease (Li & Komarek, 2017; Veronese et al., 2018). In fact, the World Health Organization (WHO) strongly recommends an intake of at least 25 g per day of naturally occurring dietary fiber for adults (WHO, 2023). This goal can be achieved in numerous ways, and one of them is the reformulation of manufactured products by including high-fiber ingredients, such as CS. The results obtained for total dietary fiber are similar to those reported by Borrelli et al. (2004), who found a mean value of 67.3 % (dry basis) from three different samples of 100 % *Arabica* CS, and Napolitano et al. (2007) who reported 69.2 % (wet basis) for *Robusta* CS from Ivory Coast. It is worth noting that these values are slightly higher than the average reported in the literature for CS (62.4 % on a dry basis) (Iriondo-deHond et al., 2020a). Further, the protein content was lower than the average range reported (16-18 %) (Iriondo-deHond et al., 2020a), which may compensate for the difference in macronutrient composition. Nevertheless, similar results were also reported by Zhang et al. (2021). These differences can be attributed to different origins and coffee species. The content of ash and lipids content are in accordance with previously reported values (Ballesteros et al., 2014; Beltrán-Medina et al., 2020; Costa et al., 2018). Regarding the fatty acid (FA) profile, it comprises 53.3 % saturated, 9.6 % monounsaturated and 36.1 % polyunsaturated fatty acids. The main fatty acid present in the sample was C18:2*n6c* (34.9 %), followed by C16:0 (27.8 %), C18:1*n9c* (9.1 %) and C22:0 (9.0 %). These results are quite similar to those reported by Costa et al. (2018).

The total phenol content of CS fell within the range reported by Bessada et al., (2018), who studied six samples of CS from different origins with TPC values ranging from 5.0 to 18.3 mg GAE/g and an average of 11.5 mg GAE/g. Brazilian and Indonesian samples stood out for having the highest TPC and antioxidant activity, indicating a potential significant relationship between these parameters and the geographical origin of coffee beans. They also reported 5-O-caffeoylquinic acid (5 CQA) as the main phenolic compound in all the samples studied. The antioxidant potential of the CS (49.8 μmol TE/g) was higher than the one reported by Ballesteros et al. (2014) as determined by DPPH assay and slightly lower than the values reported by Aroufai et al., (2022) according to ABTS method. The latter study found significant differences in antioxidant capacity between *Arabica* and *Robusta* samples, with higher levels observed in *Robusta*. In this case, the comparison with one or another species is not possible since the sample comprises a blend of unknown composition.

3.2. Ultrasound assisted extraction

The effect of two independent variables: time (minutes) and extraction power (W) was studied on extraction yield (% w/w), total phenol content (mg GAE/g) and antioxidant activity (μmol TE/g) of the CS extracts. Results for each experimental condition are presented in Table 3 (note that caffeine content and antioxidant activity by ORAC are also provided, although these were not considered as response variables). Each value was calculated as the average of three experiments

Table 3
Experimental design of extractions and results obtained for Yield, TPC, caffeine content and Antioxidant activity (ABTS and ORAC).

Power (w)	Time (min)	Global Yield (wt %)	TPC mg GAE/g extract	TEAC (ABTS) (μmol/g extract)	TEAC (ORAC) (μmol/g extract)	Caffeine (μmol/g extract)
54	15	3.14 ±0.02^{a}	37.1 ±2.7^{c,d,e}	429.5 ±4.1^{a}	927.9 ±4.1^{b,c}	19.7 ±4.1^{a}
54	30	3.60 ±0.23^{a,b}	34.5 ±1.99^{a,b,c}	429.9 ±11.2^{a}	939.1 ±4.1^{b,c}	21.2 ±4.1^{b}
54	60	4.17 ±0.32^{b}	38.8 ±1.89^{d,e,f}	438.7 ±15.1^{a}	954.2 ±4.1^{c}	27.2 ±4.1^{e}
108	15	3.86 ±0.89^{a,b}	33.2 ±0.46^{a,b}	427.3 ±6.1^{a}	850.0 ±4.1^{a,b}	23.4 ±4.1^{c,d}
108	30	4.17 ±0.61^{b}	34.8 ±0.73^{a,b,c}	422.8 ±5.1^{a}	892.4 ±4.1^{a,b,c}	23.7 ±4.1^{d}
108	60	6.22 ±0.62^{c}	41.2 ±1.56^{f}	475.2 ±11.9^{b}	900.8 ±4.1^{b,c}	44.1±4.1^{f}
180	15	5.34 ±0.33^{c}	32.3 ±1.20^{a}	443.4 ±6.7^{a}	796.8 ±4.1^{a}	22.6±4.1^{c}
180	30	7.73 ±0.71^{d}	35.9 ±0.30^{b,c,d}	520.9 ±5.7^{c}	840.0 ±4.1^{a,b}	28.0 ±4.1^{e}
180	60	8.76 ±0.12^{e}	39.8 ±0.96^{e,f}	501.5 ±9.7^{c}	1061.9 ±4.1^{d}	62.7 ±4.1^{g}

Different letters in the same column indicate that there is a significant difference between samples by the Tukey test ($p<0.05$).

($n=3$). Regarding the results obtained by the analysis of variance, extraction time had a significant influence in all the response variables studied ($p<0.05$), showing a positive correlation in all cases. Although this effect is more pronounced in global yield and caffeine content, it was also observed for the other variables (TPC and TEAC). For a set value of power, the maximum levels for each response variable were observed in the experiments conducted at 60 minutes. In this regard, Zhang et al. (2021) reported that the rate of extraction for total phenols increased with the extraction time and the maximum yield was obtained after 20 or 30 min of ultrasound treatment, the highest time level evaluated. Some studies have reported that long periods of sonication (> 40 minutes) could seriously affect the extracted phytochemicals, potentially leading to unintended changes and the generation of free radicals in the extracted compounds (Annegowda et al., 2010; Wang et al., 2008). Nonetheless, in our experiments, there was no observed decline in antioxidant activity or TPC as extraction time increased.. The maximum TPC recovered from CS was achieved at 180 W and 60 minutes, corresponding to 3.48 mg GAE/g CS. This value is higher than the ones reported by Zhang et al., (2021) using UAE in an aqueous medium. The extraction yield ranged from 3.14 to 8.76 % (w/w), and the maximum was also achieved for the same set of experimental conditions (180 W, 60 min).

Extraction power had a significant effect on global yield and antioxidant activity but not on TPC of the extracts (Table 4). In general, the highest efficiency of UAE in terms of yield and composition of the extracts can be achieved by increasing the ultrasound power, among other parameters (Chemat et al., 2017). In this case, an increase in the overall extraction yield was observed with the increase in power. However, TPC was not significantly affected ($p<0.05$). Further, the interaction between power and extraction time was significant for antioxidant activity. For the lower levels of power (54 and 108 W), no statistically significant differences were observed as time increased, while for the highest power (180 W), antioxidant activity of the extracts was higher for longer extraction times. Based on these results, the experimental conditions selected for further studies on a food matrix (cookies) were 180 W and 60 min, since this was also the extract that exhibited a major concentration of caffeine (62.7 μmol/g) and the highest antioxidant activity as determined by the ORAC assay. Furthermore, the regression analysis performed on experimental data showed adjusted R^2 values of 0.8953, 0.7205 and 0.5307 for Yield, TPC and TEAC respectively. In all cases, the highest responses were located in the zone of maximum power and time of extraction (180W and 60 minutes). According to the prediction equations given by regression models, the predicted values were very similar to the actual values obtained for those conditions: yield of 9.06%, TPC of 40.8 mg GAE/g and TEAC of 517.1 μmol/g. This confirms the decision of selecting the experimental conditions previously indicated, as this extract exhibits the highest potential as a functional ingredient.

3.3. Cookies

Many authors have remarked the potential use of CS primarily as a source of dietary fiber in cereal-based foods (Beltrán-Medina et al., 2020; Pourfarzad et al., 2013). Additionally, it has been explored for other applications such as a fat-replacer in cakes (Ateş & Elmacı, 2018), prebiotic (Jiménez-Zamora et al., 2015), nutraceutical (Bertolino et al., 2019; Nolasco et al., 2022), and even as weight control agent in antioxidant beverages (Martinez-Saez et al., 2014). The incorporation of novel ingredients in food products should always be supported by safety and sensory studies in order to assure successful merchandising among potential consumers. In this work, four different formulations of cookies enriched with different levels of coffee industry by-product (CS) and/or its phenolic extract were assessed: two of them with UCSE (0.8 and 1.2 %), one of them with CSP (11.9 %) and the last one with both ingredients (0.6 % UCSE and 11.9 % CSP). The addition of CSP significantly increased ($p<0.05$) the fiber content of the cookies from 1.45 % to 11.74 % (wb) compared to the control formulation. Thus, the goal of reaching the claim "source of fiber" was accomplished, as the amount of fiber provided in a portion of cookies (30 g) was 3.5 g, exceeding the minimum required (2.5 g/portion). Additionally, proteins, ash, TPC and antioxidant activity were also increased (Tables 5 and 6). In fact, the highest increase in TPC was achieved for the formulations with CSP instead of the ones with UCSE. This suggests that the antioxidant compounds present in the CSP are protected in some way by other components of the by-product matrix. The concept of “antioxidant dietary fiber” (ADF), first introduced by Saura-Calixto (1998) refers to dietary fiber associated with natural antioxidants, such as phenolic compounds, that could be jointly metabolized in the colon and utilized by the bacterial microbiota for providing health benefits for humans. Food by-products, derived from vegetables, fruits, cereals and seeds, constitute an interesting source of ADF, as extensively reviewed by Angulo-López et al. (2023).

As for the cookies enriched with UCSE, the increase in TPC was 13.8% and 31.2% for C-UCSE-1 and C-UCSE-2 respectively (Table 6). Neither of them achieved the objective of equaling the amount of phenols present in 100 mL of black tea, as it was proposed. It is well known that most phenolic compounds are heat-sensitive and easily degradable when exposed to normal cooking temperatures (Roy et al., 2007). Taking this into account, higher concentrations of UCSE should be used in the formulation to achieve the desired levels of polyphenols and antioxidant activity in the final product. Other strategies, such as encapsulation, could also enhance the preservation of phenols during cooking.

Finally, the C-CSP-UCSE significantly increased ($p<0.05$) the fiber content of the cookies (9.46 %) compared to the control formulation, and the values of proteins, ash, TPC and antioxidant activity were also higher. With these cookies, the maximum increase in the values of TPC and antioxidant activity were achieved, with a 56.6 % and 104.6 % increase, respectively, compared to the control cookies (Table 6).

Regarding the instrumental color results (Table 6), the addition of CSP contributed to a significant decrease in the values of L* (luminosity), which correlates to the description given by consumers for these samples as “very dark color” (Fig. 2). Similar results were reported by Ateş & Elmaci (2019) in cakes with untreated CS, which were determined to have a stronger coffee color (brownness) compared to control cakes. The cookies with UCSE showed a slight decrease for this parameter in comparison with the control sample (Table 6). Parameters a*

Table 4
p-values obtained for time, power and their interaction on each response variable according to the ANOVA

Factor	Response variable		
	Yield	TPC	TEAC
Time	**0.0001**	**<0.0001**	**0.0001**
Power	**<0.0001**	0.707	**<0.0001**
Time x Power	0.0239	0.255	**<0.0001**

Table 5
Chemical characterization of the different cookies.

	Moisture (wt %)	Protein (wt %)	Lipids (wt %)	Ash (wt %)	Total Dietary Fiber (wt %)
C	3.98±0.24^{b}	10.34 ±0.23^{a,b}	19.14 ±0.67^{a}	1.27 ±0.06^{a}	1.45±0.02^{b}
C-CSP	3.68±0.14^{a,b}	11.11 ±0.15^{c}	19.02 ±0.12^{a}	2.20 ±0.23^{b}	11.74±0.01^{d}
C-UCSE-1	2.96±0.19^{a}	9.79 ±0.04^{a}	16.60 ±0.63^{a}	1.64 ±0.02^{a}	0.42±0.01^{a}
C-UCSE-2	3.60±0.07^{a,b}	10.40 ±0.04^{b}	18.13 ±1.52^{a}	1.59 ±0.01^{a}	0.37±0.02^{a}
C-CSP-UCSE	3.89±0.39^{b}	11.91 ±0.14^{d}	18.79 ±0.98^{a}	2.11 ±0.02^{b}	9.46±0.54^{c}

Different letters in the same column indicate that there is a significant difference between samples by the Tukey test ($p<0.05$).

Table 6
Color, TPC, antioxidant activity and percentage of increase in TPC and antioxidant activity of the different cookies.

	L	a	b	TPC (mg GAE/30g)	% Increase	TEAC (μmol/g)	% Increase
C	72.7±1.2^{d}	5.6±0.5^{b}	26.2±0.5^{c}	33.1±0.9^{a}	—	6.8±0.4^{a}	—
C-CSP	53.0±2.1^{b}	3.1±0.4^{a}	8.9±1.4^{a}	50.9±0.6^{d}	53.9	11.8±0.4^{d}	74.7
C-UCSE-1	67.5±1.8^{c}	5.9±0.2^{b}	24.3±1.1^{b}	37.7±1.2^{b}	13.8	8.2±0.1^{b}	21.1
C-UCSE-2	64.8±1.5^{c}	5.4±0.5^{b}	22.3±1.2^{b}	43.4±1.1^{c}	31.2	9.8±0.2^{c}	45.5
C-CSP-UCSE	47.9±0.6^{a}	3.1±0.3^{a}	8.7±0.6^{a}	56.6±1.8^{e}	70.9	13.8±0.6^{e}	104.6

Different letters in the same column indicate that there is a significant difference between samples by the Tukey test ($p<0.05$).

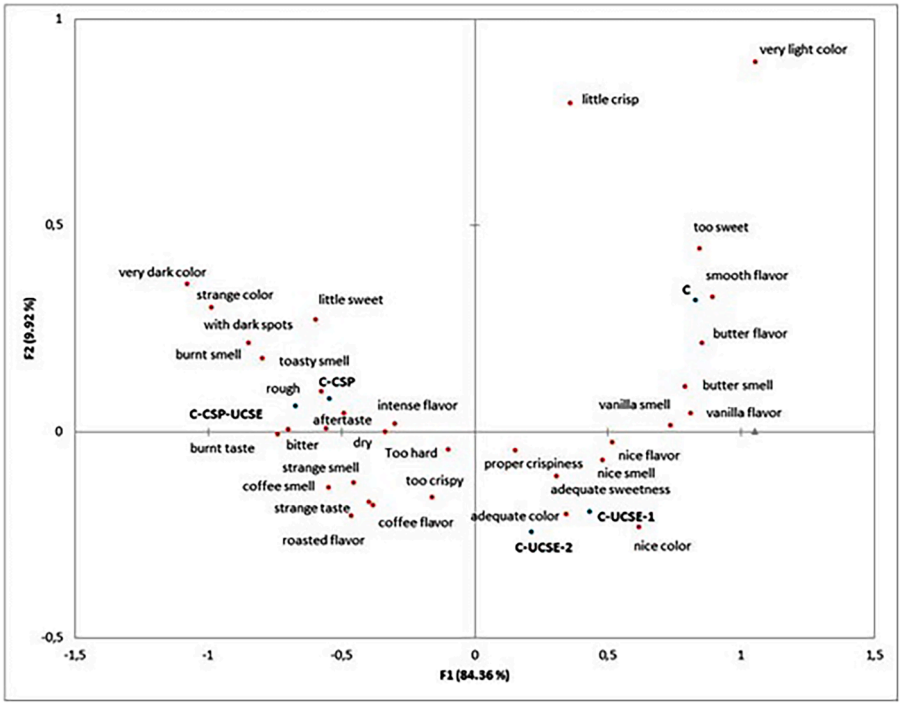

Figure 2. Representation of the samples and attributes in the first two factors of the correspondence analysis of sensory data.

(reddish) and b* (yellowish) were also strongly affected in cookies with CSP while for the cookies with UCSE only b* showed a slight decrease.

Significantly different acceptability scores ($p<0.0001$) were observed among the samples, suggesting that evaluated cookies showed differences in their sensory characteristics that affected consumers' preferences (Table 7). No significant differences were found ($p>0.05$) between the acceptability of the samples C (control) and the samples with UCSE at two different concentrations (0.8% and 1.2%). Besides, in these formulations the quality score was adequate and above the commercial quality limit (6.0 on a 9.0 point scale), established by Muñoz et al. (1992). The addition of CSP decreased the acceptability of the cookies significantly (Table 7). These cookies received lower ratings than both the control and cookies enriched with UCSE. This indicates that, despite the potential health benefits of ADF, consumers are not willing to sacrifice the taste of the product.

Table 7
Consumer's acceptability of the different cookies.

Sample	Acceptability
C	6.92^{a}
C-CSP	5.69^{b}
C-UCSE-1	6.76^{a}
C-UCSE-2	6.70^{a}
C-CSP-UCSE	5.17^{b}

Different letters in the same column indicate that there is a significant difference between samples by the Tukey test ($p<0.05$).

The five samples were clearly different regarding the sensory terms selected by consumers to describe them using the CATA questionnaire. Significant differences were found in 33 of the 35 terms of the CATA questionnaire, according to the Cochran Q test. No significant differences were found ($p > 0.05$) in the number of mentions of the terms *very soft* and *suitable hardness*.

All the samples had a high number of mentions in the terms *adequate color, suitable hardness* and *proper crispness*. Sample C (Control) received a significantly higher number of mentions than the other samples in the terms: *lighter color* and *less crispness*. When cookies with CSP were evaluated, the total number of the following attributes increased significantly ($p \leq 0.05$) according to Bonferroni test: *very dark color, with dark spots, toasty smell, coffee smell, burnt smell, low sweetness, roasted flavour, intense flavour, coffee flavour, strange taste, burnt taste, bitter* and *aftertaste*. The perception of these sensory attributes by the consumer explains the lower acceptability assigned to the two samples with CSP added.

For a better understanding of the relationship between the terms of the CATA questionnaire and the cookies samples, a correspondence analysis was performed (Fig. 2). The first two factors explained 94.3 % of the variability of the experimental data obtained. Fig. 2 shows that the samples with UCSE added at two different concentrations (C-UCSE-1 and C-UCSE-2) are located in very close zones in the space. These cookies present an adequate acceptability and they were described with the following attributes: *nice flavor, adequate sweetness, nice color, proper crispiness*.

On the other hand, samples with CSP added (C-CSP and C-CSP-UCSE) are positioned in closely zones in the space, and far from the previous ones These were the samples with lower acceptability scores (C-CSP and C-CSP-UCSE) and are situated in the left of the F1 axe associated with the terms *very dark color, strange color, with dark spots, rough, dry, too crispy, toasty smell, coffee smell, burnt smell, low sweetness, roasted flavour, intense flavour, coffee flavour, strange taste, burnt taste, bitter* and *aftertaste*.

In the work reported by Ateş & Elmaci (2019), an increase in the perception of crumb brownness, fibrousness, hardness (manual and oral texture), bitter taste, and coffee taste was also found in cakes with up to 20 % substitution of wheat flour with coffee silverskin. A decrease in crumb porosity, cohesiveness, moisture and sweet taste was also observed in these products. However, in this case the descriptive analysis was performed by a trained assessors panel (n=8) and no acceptability data are available since the cakes were not evaluated by consumers.

The sensory changes produced by the addition of CS in other baked products and the influence on the acceptability have also been reported by other authors. For instance, Cantele et al. (2022) produced vegan biscuits by replacing wheat flour with CS at 2%, 4% and 6%. These authors conducted an acceptability test of appearance, smell, taste, flavor, texture and general acceptance with 48 consumers using a nine-point hedonic scale. A decrease in the acceptability of all the attributes evaluated by the addition of CS was reported, although the sensory modifications generated were not described. These authors suggest that the low scores obtained in general acceptability could be attributed to the development of unpleasant non-volatile compounds during Maillard reactions and/or the natural presence of certain compounds in CS. These compounds include caffeine (contributing to bitterness), polysaccharides, humic acids, chlorogenic acids (contributing to astringency), and particularly carboxylic acids such as malic, citric, and acetic, which contribute to acidity.

Considering the results obtained in this study and previous findings reported by other authors regarding the low acceptability scores of food products enriched with CSP (Cantele et al., 2022; Mandura Jarić et al., 2021), it is worth exploring strategies to mitigate the undesirable sensory changes that contribute to consumers` rejection. In this sense, increasing the sugar amount or incorporating cocoa powder into the recipe could potentially improve the cookies' appeal. This could be useful to mask color and off-flavour induced by the presence of CSP and also reduce rejection related to dark color, strange color, low sweetness, roasted flavour, intense flavour, coffee flavour, strange taste, burnt taste and bitter. Another possibility is to reduce the content of CSP used in the formulations, though this would lead to a reduction in the fiber content in the cookies.

4. Conclusions

The UCSE-enriched cookies presented an adequate acceptability. They were described with the following attributes: nice flavor, adequate sweetness, nice color, proper crispiness. However, compared to the control cookie, they only increased the TPC and the antioxidant activity in 31% and 45% respectively and do not represent a source of fiber. Further, in the cookies with CSP there was an increase of 54% and 75% in TPC and antioxidant activity compared to the control cookie, and represent a source of fiber (11.8%) but they presented lower acceptability scores. They were described with the following attributes: intense flavor, dry, aftertaste, rough, bitter, toasty smell. Therefore, new strategies to mitigate undesirable sensory characteristics in this enriched food products should be done. The enriched UCSE cookies presented a pleasant appearance and flavor and no difficulties were experienced regarding the incorporation of the extract into the dough. The findings underscore the efficacy of employing environmentally friendly extraction technologies for obtaining antioxidant compounds from natural sources, particularly from by-products.

Further work related to formulation adjustments is still necessary in order to improve the sensory profile of the cookies. This could be achieved through the incorporation of new ingredients, such as cocoa powder, to potentially mask any unpleasant sensory attributes associated with CSP and enhance overall product acceptability. Moreover, conducting shelf-life studies is recommended.

Author Contribution

All authors contributed to and approved the final draft of the manuscript.

Ethical Statement

Ethical approval for the involvement of human subjects in this study was granted by la Comisión de Ética en Investigación en Seres Humanos, Facultad de Química, UdelaR, Reference number 101900-501091-21, 04/22/2022.

CRediT authorship contribution statement

Cecilia Dauber: Formal analysis, Investigation, Writing – original draft, Writing – review & editing. **Melissa Romero:** Formal analysis, Investigation. **Clarita Chaparro:** Formal analysis. **Camila Ureta:** Formal analysis. **Clara Ferrari:** Formal analysis. **Romina Lans:** Formal analysis. **Lucía Frugoni:** Formal analysis. **María V. Echeverry:** Formal analysis. **Beatriz Sánchez Calvo:** Formal analysis. **Andrés Trostchansky:** Formal analysis. **Marcelo Miraballes:** Methodology, Validation, Writing – original draft, Writing – review & editing, Supervision. **Adriana Gámbaro:** Conceptualization, Methodology. **Ignacio Vieitez:** Conceptualization, Methodology, Validation, Writing – original draft, Writing – review & editing, Supervision.

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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