Application of Ultrasonic Intensification Technology in the Extraction of Bio-Actives from Spent Coffee Grounds and Spent Tea Leaves

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Spent coffee grounds (SCG) and spent tea leaves (STL) are leftovers of the two most diffused beverages all over the world, coffee and tea. Functional components commonly found within raw materials, i.e. caffeine and phenolic compounds such as chlorogenic acid, gallic acid, and catechin derivatives, are not fully extracted from coffee grounds and tea leaves during beverage preparation. Therefore, SCG and STL can be regarded as a source of bioactives to be valorised in the formulation of various functional foods and beverages.
In the present study, ultrasound-assisted extraction (UAE) and conventional solvent extraction aimed at the recovery of natural antioxidants and caffeine from SCG and STL were compared. In particular, ethanol 60% (v/v) and boiling water were used as solvent media and the effects of two different ultrasound (US) waves amplitudes (80 and 152 µm) and treatment time (5 and 10 min) were investigated. This study closes a literature gap on UAE from SCG and STL using GRAS (Generally Recognized as Safe) solvents, in particular with water. US had an evident positive effect on the recovery of natural antioxidants when the solvent media was aqueous ethanol. In particular, applying the US treatment at 152 µm for 10 min, the total phenolic compounds content of SCG (24.01 ±0.01 mgGAE/g_{TS}) and STL (243.67 ±12.36 mgGAE/g_{TS}) extracts doubled and quadrupled, respectively. Likewise, caffeine recovery significantly increased after sonication up to 2.19 ±0.02 mg/g_{TS} from SCG and up to 12.74 ±0.36 mg/g_{TS} from STL.

1. Introduction

Embracing the personalized nutrition approach, food industries have been formulating functional foods with the promise to promote a reduction in the level of risk factors for chronic disease, to support dietary needs for people under special diets for medical conditions and to deliver key nutrients to athletes and sportspeople. Indeed, functional foods market value is expected to expand at a compound annual growth rate of 8.5% from 2022 to 2030 (GVR, 2023), therefore, the first and foremost activity is mining bioactive ingredients to be added in the formulation of many different foods and drinks.

Food by-products are attracting enormous attention amongst researchers as potential raw materials for the manufacturing of value-added compounds with high functionality and/or bioactivity. Valorisation and upcycling of food by-products or waste discarded along the food chain is a practice perfectly aligned with the Circular Economy principles and with the SDGs. However, depending on the investigated substrate, the extraction of high added-value compounds may face some technical issues such as the incomplete release of intracellular content from solid matrixes. Environmentally friendly techniques coupling the use of green solvents with technologies such as ultrasounds (US) are being investigated to reduce the mass transfer resistances of target solutes and solvents through the cell envelop (i.e. membrane, wall) (Carpentieri et al., 2022).

Tea and coffee are the most-consumed beverages worldwide with associated huge amounts of residual waste substrates. Since functional ingredients commonly found within raw materials (tea leaves and coffee grounds), namely, caffeine and phenolic compounds such as chlorogenic acid (CGA), gallic acid (GA), and catechin derivatives, are not fully extracted during beverage preparation, their solid vegetal residues can be regarded as

a precious source of bioactives. Conventional extraction processes using solvents have been already largely applied on spent coffee grounds (SCG) and spent tea leaves (STL) (Vandeponseele et al., 2021; Nadiah and Uthumporn, 2015). Since SCG are of lignocellulosic nature while hemicellulose and cellulose are the main components of STL, US appear to be efficient in assisting the extraction process. Indeed, the collapse of cavitation bubbles during sonication implies the release of large amounts of energy that destroys the cell walls of the vegetal matrix aiding the cell content discharge into the medium (Guglielmetti et al., 2017). US were successfully tested for the extraction intensification of catechins, caffeine and other antioxidant compounds from tea leaves (Gu et al., 2007) and coffee beans (Menzio et al., 2020). A recent study proved also their good potential in improving the recovery of phenolic compounds from SCG using methanol as a solvent (Okur et al., 2021). However, to the best of the Authors' knowledge, there are no literature data concerning US application on STL and SCG using GRAS (Generally Recognized as Safe) solvents.

Therefore, the purpose of this work is to evaluate the effects of US at two different amplitudes (80 and 152 μm) and treatment duration (5 and 10 min) on conventional solid-liquid extraction using two GRAS solvents, namely hot water (100 ºC) and ethanol:water (60:40 v/v). Total phenolic content and antioxidant activity of SCG and STL extracts were measured and the concentrations of caffeine, chlorogenic acid, gallic acid and epigallocatechin gallate (EGCG), the most abundant catechin in tea, were analysed by HPLC.

2. Materials and methods

SCG and STL were collected from a local cafeteria and a local tea shop nearby the University of Milano, they were vacuum-dried (-100 mbar) for 24 h at 60 °C, vacuum-packed in plastic bags and stored at 4 °C until further analysis. Analytical standards and reagents were purchased from Merck (Italy).

Determination of total solids (TS) and ashes on dried SCG and STL was performed according to the methods described by AOAC (2000).

2.1 Extraction procedure

Extracts were obtained by comparing two different solvents, namely ethanol:water (60:40 v/v) and boiling water (Ballesteros et al., 2014). A substrate to solvent ratio of 1:15 (w/v) was fixed and the extraction time was set at 30 min under magnetic stirring (Nadiah and Uthumporn, 2015; Guglielmetti et al., 2017). UAE was implemented using a sonicator (Fisherbrand, FB505EUK-220, USA) working at a 20 kHz frequency. Treatments were carried out at 80 μm and 152 μm amplitudes based on the Authors' previous studies. SCG and STL were sonicated for 5 and 10 min and subsequently stirred for additional 25 or 20 min, respectively, to complete the solvent extraction. The applied specific US energy (E_i) was calculated based on Eq(1).

$$E_i \left(\frac{MJ}{kgTS} \right) = \left[\frac{Power(W) * time\ (\text{sec})}{sample\ weight\ (\text{gTS})} \right] / 10^3 \tag{1}$$

Samples were then centrifuged (LISA - AFI, GTB Castle Limited, France) at 11,000 rpm for 10 min, supernatants were filtered (0.45 μm, Millipore - Merck) and immediately stored at -18 °C under dark till analysis. Sample preparation and relevant codes are listed in Table 1.

2.2 Characterization of SCG and STL extracts

Total phenolic content (TPC) and antioxidant activity
The Folin-Ciocalteu assay was performed to determine the TPC of the extracts following the method reported by Buratti et al. (2017). TPC was expressed as gallic acid equivalents (GAE) by a calibration curve built with the pure standard of gallic acid.

The antioxidant activity was evaluated through the DPPH assay as previously reported (Prieto et al., 2011). Data were converted into Trolox equivalents (TE) using a calibration curve built with Trolox reagent.

Folin-Ciocalteu and DPPH assays were performed in triplicate.

High-Performance Liquid Chromatography (HPLC)
Chromatographic analysis was carried out with a Hitachi Elite LaChrom HPLC. Data acquisition was performed with EZChrom Elite v. 3.2.1 software. Each extract was properly diluted with ultrapure water. The method used a Spherisorb ODS2-C18 (5 μm, 4.6 mm × 250 mm) column eluted with a binary gradient composed of 5% acetic acid in water (eluent A) and acetonitrile (eluent B). SCG extracts were analysed according to Vignoli et al. (2011). In SCG extracts, chlorogenic acid was detected at 320 nm and caffeine was detected at 272 nm. For STL extracts, the elution gradient was modified as follows: from 0 to 15% eluent B in 35 min, then up to 30% in additional 5 min, followed by re-equilibration to 100% eluent A; flow rate was 0.7 mL/min and injection volume was 20 μL. In STL extracts, gallic acid, EGCG and caffeine were detected at 280 nm. Identification and quantification of selected compounds was obtained by calibration curves built with pure standards.

HPLC analyses were performed in duplicate.

2.3 Statistical analysis

JMP 5.0 software (SAS Institute Cary NC, USA) was used for statistical analysis by one-way analysis of variance (ANOVA) with Tukey-Kramer HSD with the level of significance set up at $p \leq 0.05$ value.

Table 1: Extracts preparation from SCG and STL using ethanol or water with and without US treatment.

By-product type	Sample name	Solvent	US amplitude (µm)	US duration (min)
SCG	C-e	ethanol (60%)	/	/
	C-e-80-5	ethanol (60%)	80	5
	C-e-80-10	ethanol (60%)	80	10
	C-e-152-5	ethanol (60%)	152	5
	C-e-152-10	ethanol (60%)	152	10
	C-w	water	/	/
	C-w-80-5	water	80	5
	C-w-80-10	water	80	10
	C-w-152-5	water	152	5
	C-w-152-10	water	152	10
STL	T-e	ethanol (60%)	/	/
	T-e-80-5	ethanol (60%)	80	5
	T-e-80-10	ethanol (60%)	80	10
	T-e-152-5	ethanol (60%)	152	5
	T-e-152-10	ethanol (60%)	152	10
	T-w	water	/	/
	T-w-80-5	water	80	5
	T-w-80-10	water	80	10
	T-w-152-5	water	152	5
	T-w-152-10	water	152	10

3. Results and discussion

Coffee beans and tea leaves contain several classes of health-related chemicals that do not completely leach out during brewing. Hence, SCG and STL represent an opportunity for functional food and beverage industries as a relevant feedstock of bioactives. Intensification effects of US technology on the leach out of caffeine and antioxidant compounds from SCG and STL using both aqueous ethanol and water as GRAS solvents were investigated.

Field-collected samples of SCG and STL contained 98.49 ±0.06 % total solids and 1.55 ±0.08 % ashes and 96.29 ±0.07 % total solids and 3.90 ±0.01 % ashes, respectively. Amounts of TPC, antioxidant activity, caffeine and individual phenolics as determined by HPLC in the extracts of SCG and STL obtained through UAE at 80 and 152 µm for 5 and 10 min are reported in Tables 2 and 3.

The analysis and interpretation of the analytical data collected during the present study enables to assert that, even when using GRAS solvents (including water), US acoustic waves can significantly intensify the extraction yield of caffeine and antioxidant compounds from the treated vegetal matrixes.

Solvent type, in accordance with its physical and chemical properties, plays an important role in the extraction process affecting the amount of substances eluted from the solid matrix. Cavitational energy is also influenced by the characteristics (viscosity, surface tension and vapour pressure) of the fluid which transmits the energy to the treated substrate (Miano et al., 2021). Water can be a more effective solvent than ethanol due to its higher polarity coefficient which favours phenolic solubility (Miano et al., 2021; Nadiah and Uthumporn, 2015). However, ethanol exhibits a lower density and higher diffusivity that allows it to easily diffuse into the pores of the plant materials to leach out the bioactive constituents (Nadiah and Uthumporn, 2015). As a result, the coupled effect of US (which increase the solid matrix porosity) and ethanol can lead to higher antioxidants extraction yield.

Table 2: SCG extracts characterization in terms of total phenolic content, antioxidant activity, caffeine and chlorogenic acid (mean value ± standard deviation).

Sample	TPC (mgGAE/g$_{TS}$)	antiox act. (µmolTE/g$_{TS}$)	caffeine (mg/g$_{TS}$)	chlorogenic acid (mg/g$_{TS}$)
C-e	11.42 ±0.14 [g]	55.02 ±2.34 [c]	1.50 ±0.01 [f]	0.46 ±0.02 [f]
C-e-80-5	16.14 ±0.07 [c]	54.01 ±0.57 [c]	1.70 ±0.01 [d]	0.60 ±0.00 [de]
C-e-80-10	17.58 ±0.03 [b]	71.58 ±2.95 [b]	1.86 ±0.02 [b]	0.62 ±0.01 [cd]
C-e-152-5	16.31 ±0.04 [c]	78.47 ±3.52 [b]	1.41 ±0.00 [g]	0.55 ±0.02 [e]
C-e-152-10	24.01 ±0.01 [a]	102.94 ±0.75 [a]	2.19 ±0.02 [a]	0.80 ±0.00 [a]
C-w	12.59 ±0.17 [f]	51.30 ±4.59 [cd]	1.57 ±0.02 [e]	0.62 ±0.01 [cd]
C-w-80-5	13.04 ±0.19 [ef]	51.77 ±2.91 [cd]	1.69 ±0.01 [d]	0.66 ±0.01 [bc]
C-w-80-10	13.16 ±0.23 [e]	54.02 ±0.76 [c]	1.61 ±0.02 [e]	0.61 ±0.01 [cd]
C-w-152-5	13.45 ±0.04 [e]	49.28 ±2.04 [cd]	1.59 ±0.01 [e]	0.62 ±0.02 [cd]
C-w-152-10	14.59 ±0.00 [d]	42.97 ±0.50 [d]	1.74 ±0.01 [c]	0.67 ±0.01 [b]

Different letters in the same column indicate significant differences between samples (p < 0.05). 'a' is the highest value.

Table 3: STL extracts characterization in terms of total phenolic content, antioxidant activity, caffeine, gallic acid and epigallocatechin gallate (mean value ± standard deviation)

Sample	TPC (mgGAE/g$_{TS}$)	antiox act. (µmolTE/g$_{TS}$)	gallic acid (mg/g$_{TS}$)	EGCG (mg/g$_{TS}$)	caffeine (mg/g$_{TS}$)
T-e	65.19 ±1.82 [f]	478.73 ±20.42 [f]	1.04 ±0.01 [h]	39.90 ±0.91 [h]	2.96 ±0.11 [e]
T-e-80-5	139.03 ±0.47 [c]	1049.76 ±11.99 [c]	1.23 ±0.02 [g]	106.79 ±1.42 [d]	7.72 ±0.31 [c]
T-e-80-10	180.70 ±3.65 [b]	1111.25 ±5.17 [c]	1.34 ±0.01 [ef]	135.04 ±0.07 [b]	9.48 ±0.04 [b]
T-e-152-5	171.04 ±3.66 [b]	1346.02 ±35.13 [b]	1.32 ±0.02 [f]	129.66 ±0.64 [c]	9.36 ±0.06 [b]
T-e-152-10	243.67 ±12.36 [a]	1730.36 ±53.56 [a]	1.44 ±0.01 [cd]	193.66 ±0.48 [a]	12.74 ±0.36 [a]
T-w	93.22 ±0.88 [e]	675.90 ±11.23 [e]	1.39 ±0.01 [de]	66.71 ±0.57 [g]	6.56 ±0.06 [d]
T-w-80-5	121.27 ±4.44 [cd]	818.41 ±18.64 [d]	1.46 ±0.01 [bc]	69.74 ±0.27 [f]	7.15 ±0.06 [cd]
T-w-80-10	126.73 ±1.68 [cd]	869.05 ±43.94 [d]	1.50 ±0.02 [bc]	77.00 ±0.64 [e]	7.72 ±0.29 [c]
T-w-152-5	115.21 ±5.72 [d]	792.68 ±29.32 [d]	1.52 ±0.02 [ab]	71.84 ±0.22 [f]	6.98 ±0.03 [cd]
T-w-152-10	111.38 ±3.34 [de]	776.78 ±5.84 [de]	1.56 ±0.02 [a]	65.83 ±0.24 [g]	6.42 ±0.11 [d]

Different letters in the same column indicate significant differences between samples (p < 0.05). 'a' is the highest value.

In agreement with the above mentioned scientific assumptions, the outputs of the present research confirm the highest efficiency of US when coupled with ethanol. In addition, the effect of UAE is influenced by sonication intensity and duration meaning that the wave amplitude has a crucial role in causing a greater fragmentation and pore-formation, thus increasing the extraction efficiency. Such fact was again confirmed by the results displayed in Tables 2 and 3. A sonic wave amplitude of 80 µm for 5 min was not sufficient for a proper permeabilization of the investigated vegetal materials. Longer sonication time (80 µm for 10 min) could reach similar effect of 5 min cavitation consequences at 152 µm. Evidently, in this research study, the highest amplitude acoustic cavitation (152 µm) operated for the longest time (10 min) significantly ($p \leq 0.05$) enhanced the release of extracted components into ethanol by increasing the mass transport both for SCG and STL. Concentrations were increased up to 24.01 ±0.01 mgGAE/g$_{TS-SCG}$ and 243.67 ±12.36 mgGAE/g$_{TS-STL}$ and up to 102.94 ±0.75 µmolTE/g$_{TS-SCG}$ and 1730.36 ±53.56 µmolTE/g$_{TS-STL}$. Under the same sonication conditions but using water as a solvent, the increase of output data was not significant. Furthermore, the lower antioxidant activity detected in water-extracts C-w-152 and T-w-152 than in those treated at 80 µm (C-w-80 and T-w-80) can be ascribed to the possible degradation of some sensitive compounds as a consequence of high-intensity US treatment (Kobus et al., 2022) combined with high temperature (water was at 100 °C). The same trend was found when measuring the TPC which was lower in T-w-152 samples with respect to T-w-80 ones. Results clearly show that temperature also played a crucial role in performing the extractions since, when US were not applied, boiling water had a significantly better impact in enriching the extracts (TPC and antioxidant activity of C-w and T-w with respect to C-e and T-e), in agreement with Menzio et al. (2020). Differently, Nadiah and Uthumporn (2015) concluded that ethanol (50 % v/v) had a higher efficiency compared to boiling water in extracting phenolics from several tea extracts. However, they reported maximum increase limited to +4 % only (Nadiah and Uthumporn, 2015). Last thing to point out is that TPC and antioxidant activity of the samples were in some cases not directly related. This may be due to the presence in the extracts of different compounds with antioxidant capacity apart from polyphenols, as well as polyphenols or other compounds with a scant or null antioxidant activity (Jiménez-Moreno et al., 2019).

The *ad hoc* HPLC gradients allowed the separation of chlorogenic acid and caffeine in SCG samples and of gallic acid, EGCG and caffeine in SLT ones. The highest caffeine (2.19 ±0.02 mg/g$_{TS}$) and chlorogenic acid

(0.80 ±0.00 mg/g$_{TS}$) contents were found in the extract C-e-152-10, confirming the US best efficiency at 152 µm amplitude for 10 min. Such US condition enabled to double the extraction of caffeine (12.74 ±0.36 mg/g$_{TS}$) from STL extracts with respect to those produced using water and to triplicate their EGCG content (193.66 ±0.48 mg/g$_{TS}$). Gallic acid, instead, appeared to be more concentrated in water-extracts. However, its concentration within T-w-152-10 (1.56 ±0.02 mg/g$_{TS}$) was only 8% higher than that in T-e-152-10 (1.44 ±0.01 mg/g$_{TS}$).

Output data displayed in this paper highlight how sonication (152 µm, 10 min) led to the recovery of extracts highly richer in bioactives with respects to literature data where the reported TPC values are 9.5 mgGAE/g$_{TS}$ for SCG (Okur et al., 2021) and 180.4 mgGAE/g$_{TS}$ for STL (Nadiah and Uthumporn, 2015). Chiefly, the high EGCG extraction yield emphasizes the importance of upcycling STL also in view of the expanding tea-extracts-market that is forecasted to reach 9 billion Euros in 2029 (CBI, 2023).

The applied specific US input energy, Eq(1), for both SCG (98.49 ±0.06 %$_{TS}$) and STL (96.29 ±0.07 %$_{TS}$) was equal to ~4 MJ/kg$_{TS}$ and 8 MJ/kg$_{TS}$ when sonication was operated for 5 and 10 min, respectively. Considering a medium energy price in the EU of 0.25 Euros/kWh (Eurostat 2023 data), the expenditure related to 10 min US application would be of ~560 Euros/ton$_{TS}$. Even considering the lowest selling price of 15 Euros/kg of mass-produced extracts (CBI, 2023), the recoverable amount of value-added compounds (up to 24.01 ±0.01 mgGAE/g$_{TS}$ for SCG and to 243.67 ±12.36 mgGAE/g$_{TS}$ for STL) gives confidence in the possibility of a profitably scaled-up UAE dealing with STL alone or mixed with SCG. Notably, right the ready-to-drink tea and coffee industry could find advantageous the set-up of an in-situ sonication unit to be operated on their residues in order to recover caffeine and antioxidants compounds to be included in their products formulations.

Acknowledging the guiding principles for a sustainable healthy diet and the trend of upcycling waste and by-products, this study confirms that various bioactives can be mined from SCG and STL and utilized for development of food products providing functionality, benefits to body and mind, naturalness, and sustainability. Therewithal, they can easily be source segregated, which is an advantage that has not to be underrated when considering safety issues dealing with common food waste and by-products valorisation. Caffeine, notably accounted among the ingredients accepted as having positive cognitive health benefits, can be added to sport beverages and energy drinks as well as to snacks and energy gels. Chlorogenic acid, indexed as the main phenolic compound within SCG (Okur et al., 2021), has been already tested as an additive to several foods (i.a. bread, cookies, meat stuffing) and it favourably affected their aroma properties (Budryn et al., 2016). Gallic acid, one of the major phenolic compounds in tea leaves together with catechin derivatives (Luaibi et al., 2019), can be used as additives in the production of foods for people under special diets thanks to their capacity to raise anti-inflammatory responses and to combat oxidative stress (Dludla et al., 2018). Finally, catechins, thanks to their radical scavenging properties, can be used in foodstuffs to retard lipid oxidation in oils and fats (Yilmaz, 2006) besides improving the overall nutritional value of the food products supplemented with such functional ingredients.

4. Conclusions

This study aimed to evaluate the effect of cavitation to improve the extraction of caffeine and antioxidant compounds from SCG and STL both using ethanol and water as GRAS solvents.

The significant enrichment of the extracts treated with US confirms that such green technology helps avoiding the use of solvents that are not generally recognised as safe, besides favouring the leach out of bioactive components from vegetal matrixes. The remarkable highest effect of US was reached when ethanol (60 % v/v) was used as extraction solvent. In particular, UAE enriched the TPC of the extracts up to +110 % and +274 % when US were applied at 152 µm for 10 min on SCG and STL, respectively. Besides, US at 152 µm for 10 min could significantly intensify caffeine (2.19 ±0.02 mg/g$_{TS}$) and chlorogenic acid (0.80 ±0.00 mg/g$_{TS}$) contents within SCG extracts and of EGCG (193.66 ±0.48 mg/g$_{TS}$) and caffeine (12.74 ±0.36 mg/g$_{TS}$) in STL ones.

Waste materials used in this study contain remarkable amounts of precious bioactive compounds within the functional food and beverage market arena. As a result, SCG and STL can be considered as valuable bio-resources and UAE at large scale might be favoured for industrial applications.

Acknowledgments

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