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Proceeding Paper

Valorisation of Agro-Food By-Products for the Extraction of Phenolic Compounds †

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Abstract: The aim of this work was the extraction of phenolic compounds from several agro-food industry by-products and the determination of their antioxidant activity (AA). The highest extraction yields obtained were for the pineapple core, oat concentrate, and mango peel. The post-distillation residue of labdanum stems and leaves and spent coffee grounds were the samples presenting the highest total phenolic content (TPC) values, as well as those displaying the strongest 1,1-diphenyl-2-picrylhydrazyl radical (DPPH$^{\bullet}$) and 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) radical cation (ABTS$^{\bullet+}$) scavenging activities. For the ferric reducing antioxidant power (FRAP) assay, the highest values obtained were for the spent coffee ground, frozen coffee silverskins, and dried stevia.

Keywords: agro-food; by-products; phenolic compounds

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1. Introduction

The United Nations Environment Programme (UNEP) estimates that around 931 million tonnes of food waste were generated in 2019, with the majority of it coming from households (61%), followed by food service and retail (26 and 13%, respectively). This implies that 17% of total global food production may be wasted [1]. As the need to increase food production due to population rise is a concerning issue, new ways to counter agro-food waste are very important. Circular economy has taken to the centre stage as a way to sustainably use resources, with the creation of residues being kept to as little as possible [2,3]. Biomass has become a very important resource since it has lower greenhouse gas emissions than fossil fuels [4]. Residual biomass, biological material originating from biomass processing, is a common by-product of agriculture. It can be used in a variety of ways, from producing electricity, to fuels, solvents, or the extraction of phytochemicals [3,5]. Residual biomass is a very rich source of phenolic compounds which are secondary plant metabolites with strong antioxidant activity (AA) and play important roles in maintaining the nutritional and functional values of fruits [6]. These compounds have been extensively researched, with several health benefits being described, such as anti-inflammatory, antidiabetic, antioxidant, anticancer, antipyretic, hepatoprotective, antimicrobial, and antiproliferative activities [7].

In this study, the quantification of TPC and the determination of the AA of several agro-food wastes were performed. This will help identifying ways to successfully valorise the residues from some wastes commonly produced.

2. Materials and Methods

2.1. Samples

The selected samples included wastes from fruits, medicinal and aromatic plants, and coffee. Stevia (S) (*Stevia rebaudiana* Bertoni) dried plant material was collected from Bio sales prime. Mango (*Mangifera indica* L.) peels (M) and pineapple (*Ananas comosus* (L.) Merril.) peels (PP) and cores (PC) were kindly donated by Luís Vicente, SA/Nuvi Industrial. SA. Raspberry (R) (*Rubus idaeus* L.) post-liquor fermentation fruit was kindly donated by Eusébia Sousa. Coffee (*Coffea arabica* L. and *Coffea robusta* L. blend) spent coffee grounds (SCG) and silverskin (CS) were kindly donated by MoCoffee. Labdanum (*Cistus ladanifer* L.) leaves (LL) and stems (LS) were kindly donated by Naturalness Essential Oil Distillery. Oat concentrate (OC) (*Avena sativa* L.) was collected from Frulact.

2.2. Sample Preparation

All samples were dried under air at 41 °C until less than 10% moisture. Samples were ground and stored in the dark until further use.

2.3. Extraction

A preliminary study was conducted on mango peels to help select the appropriate solid:solvent ratio, temperature, extraction time, and solvent (Table 1). Of all those conditions, two were then chosen to conduct all following stirring maceration extractions: A—1:50 g sample/ mL solvent, 40 °C, 1 h and 50:50 water:methanol; B—1:100 g sample/ mL solvent, 60 °C, 1h and 50:50 water:methanol. After extraction, extracts were filtered, and the solvents were evaporated using a rotary evaporator (Büchi R-200, Flawil, Switzerland). The samples were then redissolved in methanol to a concentration of 50 mg/mL.

Table 1. Preliminary study on extraction conditions and obtained yields. M—mango peels.

Extraction	H_2O:MeOH	Temperature (°C)	Time (h)	Volume (mL)	Yield (%)	TPC (mg GAE/g dw) [1]
M1_25	50:50	25	1	50	43.56	$14.56 \pm 1.82^{a,b}$
M2_25			1	100	51.9	$17.74 \pm 0.82^{b,c}$
M3_25			2	50	43.38	13.02 ± 2.23^{a}
M4_25			2	100	57.63	$17.40 \pm 0.84^{b,c}$
M1_40	50:50	40	1	50	54.16	21.23 ± 1.46^{d}
M2_40			1	100	60.68	$18.00 \pm 1.65^{b,c,d}$
M3_40			2	50	47.91	$19.60 \pm 2.48^{c,d}$
M4_40			2	100	59.3	$18.11 \pm 2.01^{b,c}$
M1_60	50:50	60	1	50	21.51	$16.23 \pm 1.78^{a,b}$
M2_60			1	100	55.65	20.65 ± 1.09^{d}
M3_60			2	50	44.43	$17.60 \pm 1.47^{b,c}$
M4_60			2	100	54.87	22.63 ± 2.60^{d}
M80:20	20:80	60	1	50	56.3	$18.15 \pm 1.81^{b,c}$
M100	0:100	60	1	50	53.06	$18.30 \pm 1.86^{b,c}$

[1] GAE—gallic acid equivalents; dw—dry weight; Different superscript letters in the TPC column correspond to statistically significant differences ($p < 0.05$).

2.4. Total Phenolic Content

The total phenolic content (TPC) was assessed according to the Folin–Ciocalteu method using a plate reader (Synergy HT, Biotek Instruments, Winooski, VT, USA) at 765 nm, as reported by Macedo et al. [8], with minor modifications. The calibration curve was constructed using gallic acid solutions between 10 and 200 µg/mL, and the results were expressed as milligrams of gallic acid equivalents (GAE) per gram of extract on dry weight (DW) (mg GAE/g DW).

2.5. Antioxidant Activity

The antioxidant activity was evaluated by testing the ability of the extracts to scavenge 1,1-diphenyl-2-picrylhydrazyl ($DPPH^{\bullet}$) and 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) ($ABTS^{\bullet+}$) radicals according to Macedo et al. [8], with minor modifications. Assays were performed at 517 nm (for $DPPH^{\bullet}$) and 734 nm (for $ABTS^{\bullet+}$) in triplicate, and the results are expressed as IC_{50} values.

The Ferric Antioxidant Power (FRAP) was also measured following the procedure described in Macedo et al. [8], with minor modifications. The reaction mixture was incubated at 37 °C for 10 min, and the absorbance was measured at 593 nm.

2.6. Statistical analysis

The results are expressed as the mean ± standard deviation of three independent assays. Statistical analysis was performed using one-way analysis of variance (ANOVA) followed by Tuckey's test, and $p < 0.05$ was considered to be statistically significant. Both statistical analysis and IC_{50} values determination were performed using GraphPad Prism 8.0.1. software (GraphPad Software Inc., San Diego, CA, USA).

3. Results

3.1. Preliminary Study

The objective of the preliminary study was to determine the best conditions for the extraction of the antioxidants. For this, mango peels were used, and several different conditions were tested. The extraction yield and TPC values for each extraction were assessed and can be seen in Table 1. The three highest TPC values obtained were for the M4_60, M1_40, and M2_60. The highest yield obtained was 60.68% for the M2_40 extraction, followed by the M4_40 and the M4_25, with 59.30 and 57.63%, respectively. The chosen conditions for further extractions were then the M1_40 and the M2_60 since they displayed high TPC values, good extraction yields, and corresponded to just 1 h extractions.

3.2. Extraction Yield and Total Phenolic Content

For every sample, extractions were performed in two different conditions. Each extraction resulted in different yields and TPC values (Table 2).

The highest yields were obtained with the PC and the OC in the 2_60 extraction (64.70 and 59.92%), while for S, only 5.80% was obtained. As for the TPC, the labdanum and the SCG displayed the highest results, with 201.16 ± 4.02 mg/g for LS in the 1_40 extraction being the highest TPC obtained. Benali et al. [9] assessed the labdanum yield and TPC, achieving different results from the ones described here, having obtained, for an aqueous extract, 6.64 ± 0.06% yield and 76.98 ± 4.66 mg GAE/g of extract. Tavares et al. [10] achieved higher TPC values, with 275.6 ± 0.0 mg GAE/g extract in the extracted solid residue with 70% acetone, and 177.5 ± 0.2 mg GAE/g extract in an ethanolic extraction. Andrade et al. [11] also studied labdanum and described a TPC of 334.46 ± 31.83 mg GAE/g plant extract in acetone extract, with a 14.19% yield and 255.19 ± 7.12 mg GAE/g plant extract in ethanolic extract, with an 8.49% yield. Ballesteros et al. [12] extracted phenolic compounds from SCG through autohydrolysis, achieving a maximum TPC of 40.36 mg GAE/g SCG. Mussatto et al. [13] extracted phenolic compounds using 60% methanol in a 40 mL/g SCG and achieved a TPC of 16 mg GAE/g SCG. Solomakou et al. [14] applied a microwave-assisted extraction with 68% ethanol, achieving a maximum of 34.43 mg GAE/g SCG.

Table 2. Yield, TPC, and AA of the tested extracts.

Sample	Extraction Conditions	Yield (%)	TPC (mg GAE/g dw) [1]	DPPH• IC_{50} (µg/mL)	$ABTS^{•+}$ IC_{50} (µg/mL)	FRAP (mg AAE/g dw) [1]
Mango (M)	M1_40	54.16	21.23 ± 1.46 ^{a}	253.88±38.96 ^{a}	89.51 ± 9.74 ^{a}	6.58 ± 0.40 ^{a,b}
	M2_60	55.65	20.65 ± 1.09 ^{a}	212.23 ± 7.99 ^{a}	84.39 ± 2.71 ^{a}	8.10 ± 1.17 ^{a,b}
Raspberry (R)	R1_40	37.18	8.80 ± 1.25 ^{a,b}	360.59 ± 16.67 ^{a}	171.39 ± 11.14 ^{a,b}	3.69 ± 1.73 ^{b}
	R2_60	35.52	8.31 ± 1.37 ^{a,b}	- *	207.46 ± 6.41 ^{a,c}	4.43 ± 0.27 ^{b}
Stevia (S)	S1_40	6.43	19.76 ± 7.47 ^{a,b}	264.84 ± 23.51 ^{a}	101.84 ± 4.36 ^{a}	15.47 ± 2.27 ^{c,d}
	S2_60	5.80	57.29 ± 19.13^{c}	119.73 ± 7.02 ^{a}	68.58 ± 3.57 ^{a}	22.32 ± 3.25 ^{d}
Labdanum leaves (LL)	LL1_40	27.22	175.24 ± 21.82 ^{d}	20.54 ± 1.42 ^{a}	7.33 ± 0.59 ^{a}	10.91 ± 2.03 ^{a,c}
	LL2_60	36.49	146.53 ± 11.68 ^{e}	18.68 ± 0.25 ^{a}	9.21 ± 0.05 ^{a}	11.64 ± 2.96 ^{a,c}
Labdanum stems (LS)	LS1_40	11.31	201.16 ± 4.02 ^{f}	24.66 ± 2.40 ^{a}	9.52 ± 0.56 ^{a}	2.05 ± 0.57 ^{b}
	LS2_60	20.69	158.31 ± 24.62 ^{d,e}	64.72 ± 37.57 ^{a}	7.03 ± 0.38 ^{a}	9.42 ± 0.91 ^{a,b,c}
Oat concentrate (OC)	OC1_40	44.95	4.08 ± 1.56 ^{b}	- **	- *	0.70 ± 0.06 ^{b}
	OC2_60	59.92	4.52 ± 1.38 ^{b}	- **	1587.72 ± 294.12 ^{d}	0.89 ± 0.18 ^{b}
Spent coffee grounds (SCG)	SCG1_40	22.61	134.64 ± 14.73 ^{e}	41.18 ± 0.74 ^{a}	17.15 ± 0.60 ^{a}	86.06 ± 5.74 ^{e}
	SCG2_60	25.08	104.30 ± 14.56 ^{g}	29.31 ± 0.29 ^{a}	16.99 ± 0.61 ^{a}	79.66 ± 11.34 ^{f}
Coffee silverskins (CS)	CS1_40	10.48	23.56 ± 5.54 ^{a}	389.11 ± 6.68 ^{a}	155.12 ± 10.19 ^{a,b}	10.63 ± 2.73 ^{a,c}
	CS2_60	13.81	32.93 ± 8.90 ^{a}	120.58 ± 16.77 ^{a}	45.40 ± 5.17 ^{a}	20.39 ± 3.14 ^{d}
Frozen coffee silverskins (FCS)	FCS1_40	12.07	53.58 ± 6.11 ^{c}	118.88 ± 11.99 ^{a}	42.10 ± 1.25 ^{a}	30.60 ± 6.81 ^{g}
	FCS2_60	17.44	67.33 ± 9.49 ^{c}	79.25 ± 3.95 ^{a}	43.13 ± 3.73 ^{a}	27.84 ± 7.68 ^{d,g}
Pineapple peels (PP)	PP1_40	42.91	7.37 ± 1.90 ^{a,b}	- *	400.11 ± 105.54 ^{c}	1.35 ± 0.33 ^{b}
	PP2_60	48.39	7.92 ± 1.08 ^{a,b}	- *	334.45 ± 10.96 ^{b,c}	1.64 ± 0.25 ^{b}
Pineapple cores (PC)	PC1_40	57.58	4.58 ± 1.34 ^{b}	- *	- *	1.27 ± 0.49 ^{b}
	PC2_60	64.70	4.60 ± 1.07^{b}	4714,81±198.74^{b}	- *	1.69 ± 0.14 ^{b}

[1] GAE—gallic acid equivalents; AAE—ascorbic acid equivalents; dw—dry weight; Highest tested concentration: *—555.56 µg/mL; **—5555.56 µg/mL.; In each column, different superscript letters correspond to statistically significant differences ($p < 0.05$).

3.3. Antioxidant Activity

The AA of the extracts was measured by DPPH•, $ABTS^{•+}$, and FRAP. The IC_{50} values for DPPH• and $ABTS^{•+}$ can be seen in Table 2, as well as the ascorbic acid equivalents (mg/g) in the case of FRAP. For the former two, labdanum displayed the highest scavenging activity, followed by the SCG. In the DPPH• assay, the LL extractions displayed the highest AA and therefore the lowest IC_{50}, followed by the 1_40 extraction of LS and the 2_60 extraction of SCG, although IC_{50} values are only statistically different from those of PC extracts. Andrade et al. [11] described an IC_{50} = 7.85 µg/mL for the ethanolic extract and an IC_{50} = 39.51 µg/mL for an acetone extraction of labdanum. Coelho et al. [15] reported an IC_{50} = 12.39 ± 0.56 mg/mL for $scCO_2$-extracted SCG. For the $ABTS^{•+}$, the labdanum extracts displayed the highest scavenging activity in both 1_40 extractions, with SCG also displaying some activity. Nonetheless, there were no statistically significant differences between the LL, LS, SCG, CS, FCS, S, M, and R samples. Balzano et al. [16] reported an IC_{50} of 1.5 ± 0.9 µg/mL for an ethanolic extraction of SCG. Considering both DPPH• and $ABTS^{•+}$ assays, the less active samples were found to be those of OC, PP, and PC.

The coffee samples displayed higher antioxidant power in the FRAP assay, with SCG clearly showing the highest values, followed by the frozen coffee silverskins (FCS) and stevia. Ballesteros et al. [12] and Mussatto et al. [13] reported activity of 69.50 mg Fe(II)/g SCG when autohydrolysis was used and activity of 0.10 mM Fe(II)/g SCG for an extract obtained with a solid-liquid extraction using 60% methanol, respectively. López-Linares et al. [17] reported a 1.52 mg TE/ g SCG for an extraction using natural deep eutectic solvents (NADES). Despite having the highest TPC content and the highest AA in

DPPH$^{\bullet}$ and ABTS$^{\bullet+}$ assays, labdanum displayed far lower ferric-reducing power, with only 10.91 ± 1.34 mg AAE/g extract in LL.

4. Conclusions

The aim of this work was to assess the TPCs and AA of several by-products of agro-food industries. The extractions were performed with 50:50 methanol:water, at different volumes and temperatures, with the highest yields obtained for the PC, OC, and M samples. Labdanum post-distillation by-products displayed the highest TPC followed by SCG. The strongest DPPH$^{\bullet}$ and ABTS$^{\bullet+}$ scavenging activities were verified for the labdanum samples, followed by SCG. On the other hand, a higher reducing power was observed for SCG in FRAP assay while the labdanum samples displayed far lower reducing power. The FCS and the dried S also displayed reducing power. The results obtained offer valuable information that demonstrates the potential for the future valorisation of these by-products. The labdanum and coffee samples, particularly the SCG in the case of coffee, appear particularly interesting for further research. These extracts may potentially be used in the cosmetic, pharmaceutical and food industries. Depending on the application, different extraction types and conditions may be required, which warrants further investigation.

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