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Green Extracts from Coffee Pulp and Their Application in the Development of Innovative Brews

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Abstract: Coffee pulp, a by-product of coffee production, contains valuable compounds such as caffeine and chlorogenic acid with high antiradical activity. In this study, aqueous solutions of β-cyclodextrin (β-CD) were used as a non-conventional solvent for the extraction of targeted compounds from coffee pulp. The parameters of β-CD concentration ($C_{\beta cd}$), liquid-to-solid ratio (L/S), and temperature (T) were evaluated based on the antiradical activity (A_{AR}) and the caffeine content (C_{Caf}). The optimum operational conditions were found to be $C_{\beta cd}$: 9.25 mg/mL, L/S: 30 mL/g and T: 80 °C. The sensory profiles of brews prepared with coffee and coffee pulp with or without cyclodextrin were studied with quantitative descriptive analysis. The brew from the by-product had fruity, botanic, sweet and sourness sensory properties, and cyclodextrin was found to be able to affect the overall taste of the brew.

Keywords: chlorogenic acid; caffeine; β-cyclodextrin; coffee pulp; sensory analysis; cold brew

1. Introduction

Agricultural production leads to the accumulation of agro-food wastes and by-products. These wastes can be reused as they are a source of bioactive compounds such as phenolics that can be extracted by several conventional and non-conventional techniques.

Coffee is one of the most consumed products in the word; world coffee production for 2018/19 was estimated to be about 170,937,000 bags (60 kg/bag) [1]. For the preparation of coffee, coffee grounds must be released from the coffee cherry. During this step, many parts of the cherry are thrown away. Coffee pulp is the main by-product; other by-products of coffee production include husks, silver skin, and spent waste coffee. It is estimated that around one ton of coffee pulp is produced during the manufacture of two tons of coffee [2]. Coffee pulp is rich in cellulose, hemicellulose, phenolic compounds, and caffeine [3]. Chlorogenic acid is the main phenolic compound in coffee pulp contributing to the sensory attributes of coffee [4,5]. Specifically, it is responsible for the bitter, sour, and astringency tastes. During roasting, chlorogenic acid turns into lactones and contributes to the bitter taste of the brew [6,7]. Caffeine is another significant compound in coffee, contributing 10–30% of the final bitter taste of the brew [8].

Solid–liquid extraction is the most common technique for the extraction of polyphenols. The solvent penetrates into the plant tissue and contributes to the dilution of the bioactive compounds [9,10].

The nature of solvent, the temperature, the volume of the solvent and the time of extraction can control the final yield of the process. A novel approach in extraction processes is the development of environmentally-friendly systems, such as deep eutectic solvents and solutions of cyclodextrins [9]. Cyclodextrins are cyclic oligosaccharides which are composed of six to eight glucopyranose units. Cyclodextrin has a hydrophobic cavity and a hydrophilic surface. This structure may be used as a vessel for the extraction of hydrophobic compounds that create an inclusion complex with cyclodextrin [11]. Recent studies have investigated the extraction ability of cyclodextrin solutions and reported efficiencies that were adequate, and in some cases even higher than those obtained with conventional solvents [12–14]. Moreover, cyclodextrins can be used as masking agents. Based on the characteristic molecular structure of cyclodextrins, they can capture specific compounds and prevent the interaction with taste receptors [15]. Several scientific reports have investigated the creation of complexes between cyclodextrins and the bitter compounds in coffee, such as chlorogenic acid and caffeine [16–18].

Coffee brewing is a solid–liquid extraction which affects the quality and flavor of the drink. Commercial coffee vendors have invested in cold extraction, suggesting that a cold brew coffee possesses a different sensory profile than a conventionally brewed coffee [19]. Due to the huge coffee consumption, large amounts of by-products are generated in the coffee industry. The current study suggests that by-products from the coffee industry can be utilized as potential functional ingredients. Coffee pulp valorization could lead to the development of innovative brews containing substantial amounts of functional components, such as caffeine and chlorogenic acid, which may lead to health benefits in drinkers [20], while demonstrating unique and appealing sensory properties. However, coffee pulp has not been investigated for its sensory properties.

The purpose of this study was the optimization of the extraction process of bioactives from coffee pulp using eco-friendly β-cyclodextrin solutions. The optimization process was based on a central composite design and the responses considered were the antiradical activity and the caffeine content. Moreover, the sensory profile of the brews obtained from coffee pulp was examined.

2. Materials and Methods

2.1. Chemicals and Reagents

Folin–Ciocalteu phenol reagent was obtained from Merck (Darmstradt, German). β-cyclodextrin (CD, molecular weight of ~1135), gallic acid, caffeine (99%), Trolox™, 2,2-diphenyl-picrylhydrazyl (DPPH$^{\bullet}$) stable radical were obtained from Sigma-Aldrich, Chemie GmbH (Taufkirchen, Germany). Dichloromethane (99.8%) and chlorogenic acid (Chemical Reference Standard) were supplied from Che-Lab NM (Zedelgem, Belgium) and the European Directorate for the Quality of Medicines (Starsbourg, France), respectively. All of the organic solvents used for extraction studies were of analytical grade and purchased from Sigma-Aldrich, Chemie GmbH (Taufkirchen, Germany). All UHPLC grade solvents (methanol and formic acid) were purchased from Sigma-Aldrich, Chemie GmbH (Taufkirchen, Germany) and water for HPLC was produced form a Milli-Q apparatus (Merck KGaA, Darmstadt, German).

2.2. Plant Material

Coffee pulp from the *Cutuai* (*Coffea arabica*) variety with a mean particle size of 0.24 mm was supplied by Peralta Coffees, Nicaragua. The constituents of plant material were as follows: moisture 10.36% *w*/*w*, protein 7.71% *w*/*w*, lipid 0.75% *w*/*w*, ash 5.23% *w*/*w*, carbohydrates 75.95% *w*/*w*, raw fiber 32.73% *w*/*w*. For sensory analysis, Arabica coffee seeds were bought from a local supermarket.

2.3. Experimental Design and Response Surface Methodology

A central composite design was applied to determine the effects and the optimum levels of the examined parameters. The variation of extraction yield-dependent variable was studied at different temperatures (T, 30–80 °C), cyclodextrin (CD) concentrations ($C_{\beta cd}$, 0–18.5 mg/mL), and liquid-to-solid ratio values (L/S, 13–47 mL/g). The experimental conditions were selected based on data from the literature. A three-factor, five-level central composite rotatable design (2^3 + star) was used. This design consisted of three groups of design points, including two-level factorial design points, axial or star points, and center points. Therefore, the three selected independent variables were studied at five different levels, coded as −α, −1, 0, 1, and +α (Table 1). The value for alpha (1.68) was chosen to fulfill the rotatability in the design. According to the central composite design matrix, a total of 20 experiments was required (Table 2). The caffeine content (C_{Caf}) and the antiradical activity (A_{AR}) were chosen as the dependent variables.

Table 1. Experimental values and coded levels of the independent variables used for the central composite design. $C_{\beta cd}$, β-cyclodextrin concentration; L/S, liquid-to-solid ratio; T, temperature.

Independent Variable	Coded Variable Levels				
	−1.68	**−1.00**	**0**	**+1.00**	**+1.68**
$C_{\beta cd}$ (% *w*/*v*)	0	3.75	9.25	14.75	18.5
L/S (mL/g)	13	20	30	40	47
T (°C)	30	40	55	70	80

2.4. Extraction Process

Ground coffee pulp was mixed with an aqueous solution of β-cyclodextrin of different concentrations ($C_{\beta cd}$, 0–18.5 mg/mL), in different liquid-to-solid ratios (L/S, 13–47 mL/g), at different temperatures (T, 30–80 °C) in a stoppered glass bottle based on the experimental conditions presented in Table 2. The material was subjected to extraction under stirring at 600 rpm. In all experiments, the extracts were collected after 120 min. The time of extraction was selected based on previous experiments. Following extraction, samples were centrifuged in a bench centrifuge (Hermle Z300K, Germany) at 9000 rpm for 5 min and were separated under vacuum filtration. The clear supernatant was stored in the refrigerator until used for further analysis.

Table 2. Measured and predicted values of extracts antiradical activity (A_{AR}, μmol TRE*/g) and caffeine extraction yield (C_{Caf}, mg/g) for the individual design points.

Independent Variables				Responses			
Design Point	$C_{\beta cd}$ (mg/mL)	L/S (mL/g)	T (°C)	A_{AR} (μmol TRE/g)		C_{Caf} (mg/g)	
				Measured	Predicted	Measured	Predicted
1	3.75	20	40	20.013	19.254	4.186	4.181
2	14.75	20	40	19.862	18.615	4.016	4.047
3	3.75	40	40	22.164	21.276	4.447	4.494
4	14.75	40	40	24.202	28.323	4.438	4.364
5	3.75	20	70	32.347	28.486	4.357	4.445
6	14.75	20	70	21.019	22.167	4.496	4.463
7	3.75	40	70	25.261	26.769	4.878	4.861
8	14.75	40	70	27.117	28.136	4.863	4.883
9	0	30	55	21.949	24.453	4.698	4.638
10	18.5	30	55	27.938	25.065	4.505	4.544
11	9.25	13	55	14.201	17.102	4.151	4.110
12	9.25	47	55	27.157	23.894	4.712	4.733
13	9.25	30	30	23.634	23.023	4.073	4.080
14	9.25	30	80	30.324	30.56	4.761	4.733
15	9.25	30	55	22.425	22.882	4.579	4.567
16	9.25	30	55	21.472	22.882	4.490	4.567
17	9.25	30	55	25.615	22.882	4.611	4.567
18	9.25	30	55	22.637	22.882	4.588	4.567
19	9.25	30	55	22.679	22.882	4.568	4.567
20	9.25	30	55	22.401	22.882	4.566	4.567

TRE*: Trolox Equivalent.

2.5. Determination of Antiradical Activity (A_{AR})

The antiradical activity was determined according to a previously described protocol [21]. Briefly, an aliquot of 0.025 mL of extract was added to 0.975 mL DPPH$^{\bullet}$ solution (100 µM in MeOH) and the absorbance at 515 nm was read at 0 and 30 min. Trolox™ equivalents (mM TRE) were determined from a linear regression, after plotting $\%\Delta A_{515}$ of known solutions of Trolox™ against concentration, where:

$$\%\Delta A_{515} = \frac{A_{515}^{t=0} - A_{515}^{t=30}}{A_{515}^{t=0}} \times 100 \quad (1)$$

Results were expressed as µmol TRE per g of coffee pulp weight.

2.6. Determination of Caffeine Extraction Yield

Caffeine quantitation was based on a previously described protocol [22]. Briefly, 12 mL of extract was mixed with an equal amount of dichloromethane. Then, using a separatory funnel, the caffeine was extracted using dichloromethane. The caffeine extraction yield (C_{Caf}) was expressed as mg caffeine per g of coffee pulp.

2.7. Determination of Total Polyphenol Yield (Y_{TP})

The total phenolic content of the brews was determined according to a protocol [21] using the Folin–Ciocalteu methodology. Yield in total polyphenols (Y_{TP}) was expressed as mg gallic acid equivalents (GAE) per g of coffee pulp weight.

2.8. Determination of Caffeine and Chlorogenic Acid Content

The quantification of caffeine and chlorogenic acid in brews was performed by ultra-high-performance liquid chromatography (UHPLC) using an ECOM spol. s r.o., Czech Republic system (model ECS05). The system is comprised of a quaternary gradient pump (ECP2010H), a gradient box with degasser (ECB2004), a column heating/cooling oven (ECO 2080), an autosampler (ECOM Alias) and diode array detector (ECDA2800 UV-VIS PDA Detector). A Phenomenex® reversed-phase column (Synergi™ Max-RP 80 Å; 4 µm particle size, 150 × 4.6 mm) was used at 25 °C. The sample injection volume was 10 µL. Chromatographic analysis was performed using a gradient of Milli-Q water with 0.1% formic acid (solvent A) and methanol with 0.1% formic acid (solvent B), at constant flow rate of 0.5 mL/min. The gradient program was as follows: solvent A was decreased from 70% to 55% after 5 min; followed by another decrease to 35% until 15 min; while it was finally reduced to 10% at 18 min. Then, solvent A was maintained at 10% for 2 min and returned to initial conditions (70% solvent A). Detection was accomplished with the diode array detector and chromatograms were recorded at 276 nm for caffeine and 330 nm for chlorogenic acid. Identification of caffeine and chlorogenic acid was performed by comparing the retention time and the UV-Vis spectra with those of reference standards, while quantification was established with the aid of calibration curves (Equations (2) and (3), respectively). All chromatographic data were analyzed using Clarity Chromatography Software v8.2 (DataApex Ltd.).

$$\text{Caffeine Concentration } \left(\frac{mg}{mL}\right) = 0.000051 * x * \text{area} - 0.000893; \ (R^2 = 0.99952) \quad (2)$$

$$\text{Chlorogenic acid concentration } \left(\frac{mg}{mL}\right) = 0.000041 * x * \text{area} + 0.000691; \ (R^2 = 0.99999) \quad (3)$$

2.9. Sensory Analysis

2.9.1. Preparation of Brews

A set of four samples was prepared for the sensory evaluation. The applied experimental conditions are displayed in Table 3 for design point 13, as these conditions were closest to the conditions of the cold brewing technique. All brews were prepared by using a liquid-to-solid ratio of 30 mL/g, an extraction time of 2 h, and a temperature of 30 °C. Two of the four samples were from Arabica coffee seeds (AQC) while the other two samples (AQCW) were made by using coffee pulp as the main extractable raw material. In each subset of samples, β-cyclodextrin was dissolved in water at a concentration of 9.25 mg/mL in one of the two samples prior to the start of the coffee extraction (AQC/CD and AQCW/CD). Detailed sample formulation is presented in Table 3. Subsequently, the extracts were collected by filtration and stored in a refrigerator until the sensory session. For each panelist, a quantity of 20 mL was served at room temperature in a plastic cup with a lid. The samples were randomly presented in four hourly sessions on three consecutive days.

Table 3. Experimental conditions for the samples used in the sensory evaluation. t, time.

Sample Name	Raw Materials	$C_{\beta cd}$ (mg/mL)	L/S (mL/g)	T (°C)	t (h)
AQC	Arabica coffee beans	0	30	30	2
AQC/CD	Arabica coffee beans	9.25			
AQCW	Arabica coffee pulp	0			
AQCW/CD	Arabica coffee pulp	9.25			

AQC: aqueous extract from coffee seeds, AQC/CD: β-cyclodextrin extract from coffee seeds, AQCW: aqueous extract from coffee pulp, AQCW/CD: β-cyclodextrin extract from coffee pulp.

2.9.2. Quantitative Descriptive Analysis

Nine trained panelists from the Department of Food Science and Technology (Aristotle University of Thessaloniki, Greece), who had already participated in several trained panel studies for other food products and with at least 1 year of experience in sensory evaluation, developed a consensus vocabulary of 11 descriptors using a Quantitative Descriptive Analysis approach. The attributes were categorized under the modalities of aroma and taste and reference standards were used when required. For a better explanation of the developed attributes, the reference standards are presented in Table 4. The terms botanic, fruity, earthy, and roasted were used to describe the aroma characteristics, whereas the words bitter, sweet, sourness, sour-roasted, botanic, earthy, and astringency were used to characterize the taste of the samples.

Table 4. Developed attributes and references standards in the sensory evaluation.

Modality	Descriptor	Definition
Odor	Botanic	Characteristic odor associated with typical dried black tea notes
	Fruity	Overall odor associated with floral, sweet, ripe fruits and characteristic odor of coffee pulp
	Earthy	Odor associated with bread and wet soil
	Roasted	Characteristic odor of over-roasted hazelnuts (220 °C/10 minutes)
Taste	Bitter	The fundamental sensation associated with caffeic acid
	Sweet	The fundamental sensation associated with sucrose
	Sourness	Taste associated with sour/fermented-like aromatics
	Sour-roasted like	Characteristic acidic, sharp and pungent taste associated with excessively roasted coffee beans
	Botanic	Characteristic taste of black tea infusion
	Earthy	Characteristic taste associated with bread crust
Tactile sensation	Astringent	A dry penetrating sensation in the nasal cavity

Attributes were scored using unstructured line scales (0–100) and panelists were seated in booths under appropriate environmental conditions (in accordance with International Organization for

Standardization recommendation (ISO 8589:2010). Panelists individually rated samples in duplicate on two separate examinations. Samples were presented monadically according to a balanced design, labeled with arbitrary three-digit codes in opaque white plastic cups and maintained at ambient temperature before serving for evaluation. Bottled water and unsalted crackers were provided as palate cleanser between samples.

2.10. Statistical Analysis

The response surface methodology (RSM) data were analyzed using the statistical software MINITAB (release 13.32). To identify the significance of the effects and interactions between them, analysis of variance (ANOVA) was performed for each p-value less than 0.05, which was considered to be statistically significant. Regression analysis was used to fit a full second order polynomial to the data of the response variables. To evaluate the goodness of fit of each model, two criteria were used: the coefficient of determination, R^2, which is the relative variance explained by the model with respect to the total variance.

Sensory data were analyzed using the SENPAQ software (Qi Statistics, Ruscombe, UK). A two-way model analysis of variance (ANOVA) was performed; panelists were treated as random effects and samples and replicates as fixed effects. Multiple pairwise comparisons were conducted by Tukey's method and a significant difference was determined at an alpha risk of 5% ($p \leq 0.05$). All determinations were carried out in triplicate. The values obtained were averaged.

3. Results and Discussion

3.1. Extraction Yield

Figure 1 presents the effect of the process variables on the responses A_{AR} and C_{Caf}. In the case of A_{AR}, it can be observed that an increase of $C_{\beta cd}$ generally leads to increased values of A_{AR}. Similar studies on plant extracts rich in phenolics showed that the extract antiradical activity increased with the concentration of β-cyclodextrin in the solution [23,24], since the existence of β-CD can lead to inclusion complex formation with phenolic compounds. According to [9], the protection of phenolic compounds with β-CD can contribute to increased values of A_{AR}. However, the β-CD concentration does not seem to affect the concentration of caffeine in the extract and it can be observed that the highest C_{Caf} was obtained in the absence of cyclodextrin. The inclusion complex formation of caffeine has already been studied [25] and the usage of cyclodextrin has been proposed for the decaffeination process [26]. In complicated extracts, it is difficult to figure out which specific compounds form complexes [13]. It seems that the presence of cyclodextrin preferably boosted the extraction of more hydrophobic polyphenol molecules than caffeine [27].

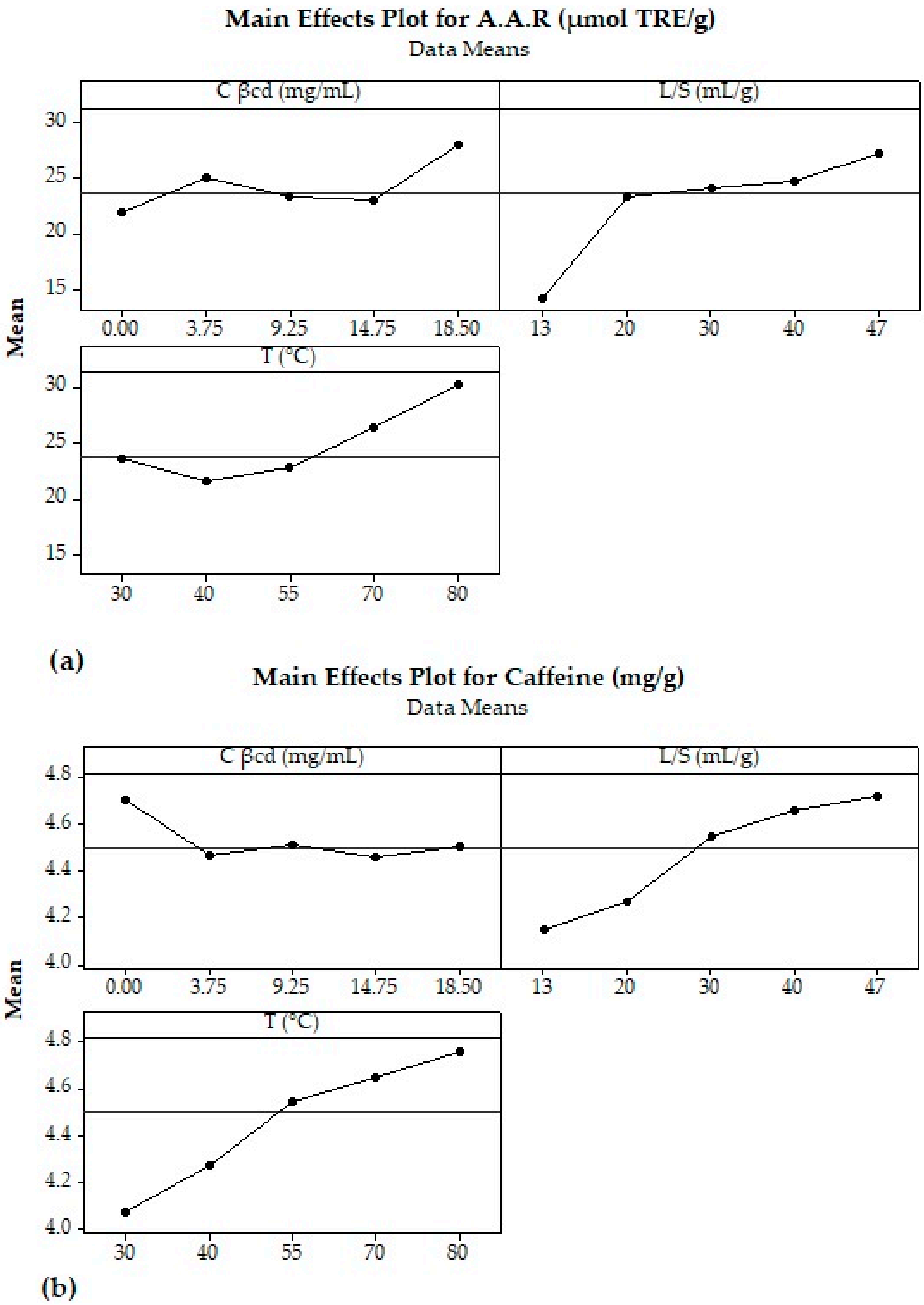

Figure 1. Main effects plots presenting the effect of β-cyclodextrin concentration ($C_{\beta cd}$, mg/mL), liquid-to-solid ratio (L/S, mL/g), and temperature (T, °C) (**a**) on extract antiradical activity (A_{AR}, µmol TRE/g) and (**b**) on caffeine extraction yield (C_{Caf}).

The liquid-to-solid ratio (L/S) has a great impact on the examined responses. The extraction yield increased with increasing ratio. A similar trend was reported by many researchers [12,28,29]. A higher ratio results in a larger concentration gradient during diffusion from the solid into the solution and in excessive swelling of the plant material, increasing the contact surface area between the material and the solvent [30].

The positive effect of extraction temperature on A_{AR} and C_{Caf} is presented in Figure 1. It is widely recognized that temperature enhances mass transfer by improving the extraction rate; a fact that can be attributed to the effect of temperature on the vapor pressure, surface tension, and viscosity of the solvent. Moreover, the increase in the yield may be associated with the increased ease with which solvent diffuses into cells and the enhancement of desorption and solubility at high temperatures [31]. Previous studies have reported a decrease of extract antiradical activity with temperature due to the possible degradation of polyphenolic compounds [32].

The regression coefficients were calculated and the data was fitted to second-order polynomial equations:

$$A_{AR}(\mu mol\ TRE/g) = 12.32 - 0.474 * C_{\beta\ cd} + 0.714 * L/S - 0.191 * T + 0.022 * C_{\beta\ cd}^2 - 0.008 * (L/S)^2 + 0.006 * T^2 + 0.035 * C_{\beta\ cd} * L/S - 0.017 * C_{\beta\ cd} * T - 0.006 * L/S * T \quad (4)$$

$$C_{Caf}\ (mg/g) = 2.660 - 0.036 * C_{\beta\ cd} + 0.039 * L/S + 0.032 * T + 0.280 * 10^{-3} * C_{\beta\ cd}^2 - 0.500 * 10^{-3} * (L/S)^2 - 0.260 * T^2 + 0.020 * 10^{-3} * C_{\beta\ cd} * L/S + 0.460 * 10^{-3} * C_{\beta\ cd} * T + 0.170 * 10^{-3} * L/S * T \quad (5)$$

The coefficient of determination, R^2, for A_{AR} and C_{Caf} was 0.729 and 0.969, respectively, indicating that 72.9% and 96.9% of the total variability in the responses could be explained by the specific models. According to the regression analysis, the temperature, the liquid-to-solid ratio, and their quadrates significantly influence the extraction yield ($p < 0.05$).

The measured and the predicted values of the responses are analytically depicted in Table 2. The extraction conditions of 9.25 mg/mL ($C_{\beta cd}$), 30 mL/g (L/S), and 80 °C (T) are the optimum conditions. Under these conditions, the predicted values of antiradical activity and caffeine content were 30.56 μmol TRE/g dry matter and 4.733 mg/g, respectively, whereas the observed experimental values were 30.324 μmol TRE/g dry matter and 4.761 mg/g, respectively.

3.2. Sensory Analysis

A consensus vocabulary of eleven attributes was developed to describe the samples, under the modalities of odor, taste, and mouthfeel, while using reference standards where required. Similar sensory descriptors were generated by other trained panels to characterize brewed coffee beverages [33]. All sensory descriptors differ significantly between samples ($p < 0.05$, Table 5). To the best of the authors' knowledge, this is the first time that coffee pulp has been used scientifically for cold brewed coffee drink preparation and the developed lexicon is unprecedented. Cold brewing is an innovative technique and several researchers have worked on its sensory evaluation. Generally, cold brews are characterized by a sweeter and sour taste as compared to the product of the classical technique. Hence, the purpose of the sensory profiling was to investigate the sensory quality of an alternative coffee drink/beverage formulation and provide a measure of the alterations in coffee drink descriptors occurring when the raw material used is changed.

The sensory profiles of the different brews are presented in Figure 2. Brews prepared from coffee grounds (AQC and AQC/CD) were compared to the brews obtained from coffee pulp (AQCW and AQCW/CD). The panelists scored the latter samples as significantly more botanic and fruitier in odor and more sweet, sour, and botanic in taste. The earthy odor and taste intensities were significantly higher for the coffee bean brew samples as compared to the other two samples. In addition, the AQC and AQC/CD samples had significantly higher scores in bitter and sour-roasted taste than their counterparts. The results of this study show that the sensory characteristics of the cold brew coffee bean samples resembled the usual organoleptic properties of coffee samples prepared with the hot brewing technique. Attributes such as earthy, roasted, and fruity aromas, bitter and sour tastes, and astringency had been previously used to evaluate hot brewed coffee [33]; however, the perceived intensities are not similar. On the contrary, the sensory attributes of the AQC and AQC/CD samples possessed similar characteristics to tea beverages, since they were characterized by high intensities of botanic and fruity aromas, and sweet, sour, and botanic taste sensations.

Table 5. Mean intensity scores of aroma and taste attributes for the different brews.

Characteristics **	Attributes	Samples			
		AQC ***	AQC/CD	AQCW	AQCW/CD
O	Botanic	7.8 [b]	6.4 [b]	54.6 [a]	52.1 [a]
O	Fruity	5.8 [b]	1.8 [b]	49.5 [a]	33.2 [a]
O	Earthy	30.7 [a]	31.9 [a]	11.9 [b]	13.8 [b]
O	Roasted	61.6 [a]	60.5 [a]	1.1 [b]	1.6 [b]
T	Bitter	56.4 [a]	35.9 [b]	3.1 [c]	2.3 [c]
T	Sweet	6.2 [c]	5.9 [c]	23.5 [b]	38.5 [a]
T	Sourness	1.1 [b]	2.1 [b]	49.4 [a]	43.6 [a]
T	Sour-roasted	26.3 [a]	37.5 [a]	2.0 [b]	1.4 [b]
T	Botanic	4.9 [b]	4.5 [b]	42.5 [a]	38.6 [a]
T	Earthy	33.1 [a]	31.1 [a]	8.7 [b]	9.5 [b]
MF	Astrigency	21.9 [ab]	24.6 [a]	17.7 [ab]	10.1 [b]

abc: different letters represent statistically significant differences, $p < 0.05$ (Tukey's method). **: O for odor, T for taste, MF for mouthfeel. ***: AQC: aqueous extract from coffee seeds, AQC/CD: β-cyclodextrin extract from coffee seeds, AQCW: aqueous extract from coffee pulp, AQCW/CD: β-cyclodextrin extract from coffee pulp.

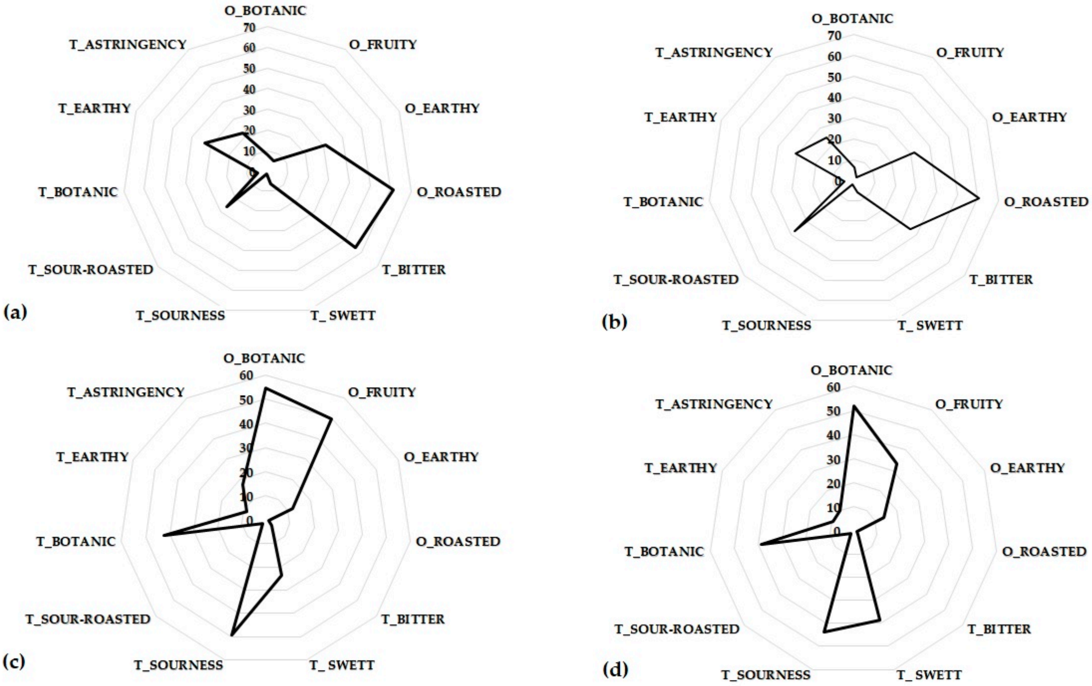

Figure 2. Spider graph for the sensory attributes of the different brews. (**a**) AQC: aqueous extract from coffee seeds, (**b**) AQC/CD: β-cyclodextrin extract from coffee seeds, (**c**) AQCW: aqueous extract from coffee pulp, (**d**) AQCW/CD: β-cyclodextrin extract from coffee pulp.

As far as the effect of *β*-CD is concerned, it was found that the AQC sample had a less bitter taste intensity as compared to the AQC/CD sample. Based on the fact that the content of chlorogenic acid and caffeine was the same in both brews (Table 6), it seems that the selective inclusion formation of the extracted ingredients affects the sensory characteristics of the brews. The same behavior has been observed in tea infusion, in which cyclodextrin appeared selective in the extraction of different compounds from tea [34]. These results give a new insight in the contribution of cyclodextrin in taste differentiation. Cyclodextrin has been used for taste masking in different products. In bitter gourd juice, cyclodextrin contributes to the reduction of the bitter taste [35] and this effect was associated with the fact that the molecules responsible for bitter sensations, caffeine and chlorogenic acid, partially form stable inclusion complexes with β-CD [36], resulting in masking of the bitter sensation by more than 30%. The mechanism of complex formation between β-CD and chlorogenic and caffeic acids in aqueous solutions was previously investigated, suggesting that catechol hydroxyl groups are trapped inside the β-CD cavity [17]. Another effect of β-CD addition is related to the intensity of astringency. More specifically, the coffee pulp brewed sample with β-CD was characterized by a less astringent mouthfeel as compared to the other coffee bean brewed sample.

Table 6. Total phenolics, caffeine, and chlorogenic acid content in the different brews.

	AQC *	AQC/CD	AQCW	AQCW/CD
Total phenolics (mg GAE/mL)	558.96 ± 4.18	684.45 ± 9.02	278.99 ± 4.51	198.30 ± 2.95
Caffeine (mg/mL)	0.4211	0.4257	0.1357	0.1388
Chlorogenic acid (mg/mL)	0.1065	0.1086	0.152	0.151

* AQC: aqueous extract from coffee seeds, AQC/CD: β-cyclodextrin extract from coffee seeds, AQCW: aqueous extract from coffee pulp, AQCW/CD: β-cyclodextrin extract from coffee pulp.

β-CD can be considered as a slightly sweet substance, showing a recognition threshold for sweetness in water around 0.5% [36]; hence, its sweetness cannot be ignored. This study showed two results concerning sweetness: there was no perceived variation in sweet taste between the coffee bean brewed samples, whereas sweetness was more pronounced in AQCW/CD than in the sample prepared by brewing coffee pulp with water. This observation could be attributed to the phenomenon of mixture intensity suppression; in the case of coffee brew samples, intensity was at such high levels that it suppressed the perception of sweet taste, whereas the coffee pulp brewed samples scored very low in bitterness, "allowing" the perception of different intensities in sweetness.

Earlier studies utilized coffee by-products as a source of antioxidants and dietary fibers in bakery products, such as biscuits [37] and muffins [38], investigating consumer acceptance and the sensory quality of the novel products. There is currently very little research published on the development of functional beverages from coffee waste. Coffee silver skin was brewed and used as an ingredient in a beverage aiming to reduce body fat accumulation [39]. The study concluded that the acceptance level of the beverages made with coffee silver skin was about 95%. However, the effect of coffee by-product use on the sensory profile of the functional beverages was not investigated. To the best of our knowledge, the current study is the first systematic approach in determining variations in sensory attributes of cold brew coffees made from coffee seeds or coffee waste with or without β-CD addition. The brewed coffee waste samples demonstrated sensory properties similar to beverages based on tea brew, with highly perceived intensities of fruity and botanic aroma characteristics and sweet, sour, and botanic taste characteristics. This novel beverage prepared by utilizing a coffee by-product could be widely accepted for its sensory quality and consumed by non-coffee drinkers as well.

4. Conclusions

The present study showed, for the first time, that aqueous solutions of β-cyclodextrin could be very effective solvent systems regarding the extraction of phytochemicals from coffee pulp. The experimental design based on a response surface methodology optimized the conditions, enabling the production of extracts with enhanced antioxidant and caffeine content. The applied extraction method with cyclodextrin resulted in innovative brews from coffee and coffee by-products with modified sensory attributes.

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