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Original Research Article

Thorough Utilization of Chestnut Shells as a Source for Phenolic Compounds Extraction and Biochar Production

Ángeles Cancela*[1], Antia Iglesias[2], Martina Martinez[3], Ángel Sánchez [4]

[1] Department of Chemical Engineering, Forestry Engineering School, University of Vigo, Campus Universitario Lagoas-Marcosende, CP: 36310 Vigo, Pontevedra, Spain
e-mail: chiqui@uvigo.gal
[2]Department of Draw, Faculty of Fine Arts, University of Vigo, Campus Universitario Lagoas-Marcosende, CP: 36310 Vigo, Pontevedra, Spain
e-mail: antia.iglesias@uvigo.gal
[3] Forestry Engineering School, University of Vigo, Campus Universitario Lagoas-Marcosende, CP: 36310 Vigo, Pontevedra, Spain
e-mail: martinamartinezsoto@gmail.com
[4] Department of Chemical Engineering, Industrial Engineering School, University of Vigo, Campus Universitario Lagoas-Marcosende, CP: 36310 Vigo, Pontevedra, Spain
e-mail: asanchez@uvigo.es

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ABSTRACT

Research on chestnut residues has shown that phenolic and antioxidant compounds can be extracted. In addition, the waste from the process can serve as a source for biochar production. In this study, three extraction methods were employed. To optimize the extraction process and determine the best experimental conditions, response surface methodologies were applied, including three-level factorial designs (3^2) and the Box-Behnken design for each extraction method. Results indicated that the Soxhlet method, distilled water for 4 h, and the 50:50 distilled water / ethanol mixture for 6 h yielded the most promising results. The highest phenolic content (679.69 mg GAE/g) was achieved by the Soxhlet method. The residues resulting from the extractions, were mixed and used were used to obtain biochar through the application of fast pyrolysis. Biochar yields were 47%, and the percentage of ashes and volatiles generated during the process was high.

KEYWORDS

Phenolic compounds, biochar, response surface methodology (RSM), solid-liquid extraction, Soxhlet extraction, Ultrasound-assisted extraction, Microwave-assisted extraction.

INTRODUCTION

The European chestnut is a hardwood species with substantial agro-economic importance in Galicia and the NorthWest Spain, providing various ecosystem services. In Galicia, despite being assumed than during Roman conquest chestnut cultivation was introduced, the earliest documented evidence of chestnut cultivation dates back to the Middle Ages [1]. From the middle of the 9th century AD, a change in the management of these resources took place, as other species began to be used for timber production and the fruits of the chestnut tree began

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[*] Corresponding author

to be harvested [2]. European chestnut (*Castanea sativa*) plays an important role in Galician society [3] because Galicia is one of the main producers of chestnuts in Spain, marketing half of the production and transforming the other half into valuable products derived from chestnuts, discarding those of lesser value for fodder.

In the present day, according to official forest maps, there are around 49.000 ha of chestnut stands in Galicia. This region is the main region in the production and export of chestnuts in Spain. The production of chestnuts in Galicia was 20,000 t, representing almost 95 % of the total production in Spain (21,000 t). In order to process this fruit, a large amount of waste material is generated, including inner shell, outer shell, and leaves [4]. Bearing in mind that the edible portion of the chestnut is 82 g out of 100 g, the waste generated in Galicia amounts to approximately 30.400 t/y. These wastes are generally discarded during post-harvest and food processing, with a high impact on environmental quality [5].

Previous studies have demonstrated that by-products derived from the waste generated during chestnut processing possess potential for the extraction of bioactive compounds, in addition to their application in biochar production [6]. These compounds are phenolic compounds [7,8] and can be found in pericarp, burs, and leaves. The bioactive compounds have a high biological value [9], especially for the treatment of diseases, due to their high content of phenolic compounds with potential health applications, such as food additives [10,11] to enhance or preserve products, as well as antioxidant, antimelanogenic [12], anticancer, antibacterial [13], anti-inflammatory, hypoglycaemic and obesity treatment applications [14]. Additionally, they have applications in the cosmetics industry, that have previously been explored using green technologies. Taking into account the chemical structure, phenolic compounds extracted from chestnut shells are divided into phenolic acids, flavonoids, and tannins. Previous studies [15] have found large amounts of pyrogallol and protocatechuic acid in the chestnut shells, so that the presence of significant contents of total phenols, low molecular weight phenols (gallic and ellagic acid), condensed tannins, and ellagitannins [16].

In addition, the residue resulting from the extraction of phenolic compounds can be used in biorefinery [17] or to produce biochar [18]. Biochar can be prepared from organic waste, by the pyrolysis of biomass [19]. Its applications are diverse, ranging from energy production to use in agriculture and livestock farming, in addition to its use in environmental remediation. In all these processes, solid samples are generated, which are initially waste, but which can be used to make art. [20]

The aim of this work is to lay the foundations for developing a process for extracting value-added compounds on a larger scale with optimum efficiency. This is achieved through valorization of the waste generated during the processing of the fruit. by reusing waste material and reducing the environmental impact. Different solid-liquid extraction methods will be used to evaluate the most efficient one. To optimise the extraction methods, response surface methodology will be used to determine the best extraction conditions. The residue resulting from the extractions will be used to obtain biochar through the application of fast pyrolysis.

MATERIALS AND METHODS

Sample preparation

The chestnuts used in this study were collected in the surroundings of the Castiñeiras Lake, located in Moaña, Galicia, Spain in October 2021. The chestnuts harvested are of the "*De Parede*" variety, which is located in climate zone II in Galicia and its traditional area of use is the eastern mountains of Lugo (Os Ancares, Folgoso do Courel) and the Mariña de Lugo. The harvested chestnuts were dried and frozen for preservation until the start of the experimental part. To prepare the sample, the chestnuts were peeled and the shells obtained were taken to the Wood Chemical Transformation Laboratory of the School of Forestry Engineering in Pontevedra. The shells were crushed in a Retsch ® SM 100 cutting mill with a 6 mm grit size, which facilitates the extraction of compounds. The moisture content of the sample was determined by weight difference according to the UNE-EN ISO 18134-3:2016 standard. This material was then stored in plastic bags to avoid mass variations.

Solvents

Different polar solvents suitable for the extraction of phenolic compounds were used. The solvents selected were distilled water and ethanol.

Distilled water (H_2O) has a dielectric constant (F/m) of 80, high polarity, a boiling temperature of 100 °C, and a density (g/mL) at 20 °C of 1000 [21]. The advantages of this solvent are its polarity, non-toxic and non-flammable properties, affordable price and its ability to dissolve various substances. On the other hand, its limitations include the easy and rapid promotion of fungal and bacterial growth, as well as the fact that it requires more energy to concentrate the extract obtained than other solvents such as ethanol.

Ethanol (C_2H_6O) has a dielectric constant (F/m) of 24.3, it has polarity, its boiling temperature is 78 °C, and a density (g/mL) at 20 °C of 0.80. This solvent evaporates before water, has a low toxicity, has hydroxyl groups that allow it to dissolve ionic compounds, and can be recovered at the rotary evaporator. Moreover, azeotropic mixtures can be obtained with water as ethanol is miscible with water. Previous studies [22] indicate that the best results for obtaining phenolic compounds with organic solvents are obtained with 50 % solutions. Therefore, the third solvent chosen was ethanol and water, 50:50.

Extraction methods

Three solid – liquid (SL) extraction methods were used for this experiment: Soxhlet, ultrasound-assisted extraction, and Microwave-assisted extraction. All experiments were carried out with 7.5 g of organic matter (chestnut shells) and 150 mL of solvent. The solvents used in the three methods were distilled water (W), ethanol (E) and a mixture of distilled water and ethanol (E:W). The solvent is brought into contact with the chestnut shell and left to act under certain conditions. The variables in these extraction methods were the type of solvent, the temperature, and the contact time between the solvent and the organic matter. The variables chosen for each of the methods can be seen in **Table 1**.

Table 1. Variables for the three methods studied

Variables	Soxhlet			Ultrasound			Microwave		
t/ min	120	240	360	20	40	60	0.5	1	1.5
Solvent	E	W	E:W	E	W	E:W	E	W	E:W
T^a/ ºC	-	-	-	40	60	80	-	-	-

Soxhlet method. According to previous studies [23] the cartridge of chestnut shells was placed in the extraction system coupled to a round-bottom flask where the solvent was placed. This flask was heated with a heating mantle Nahita blue series 655. The extractions were carried out three times each experiment and, taking into account that each cycle lasts 30 min the total extraction times were: 120 (4 cycles), 240 (8 cycles) and 360 (12 cycles) min.

Ultrasound-assisted extraction method. Based on previous studies [21] the ultrasonic extraction was carried out with a Bandelin Sonorex Super RK 102 H with an ultrasonic frequency of 35 kHz, a timer from 1 to 15 min, and with a continuous operation option and a maximum temperature of 80 °C. In the case of water, shaking is necessary to promote mixing.

The round-bottom flask with the solid mixture and the solvent is placed in the ultrasonic bath with pre-heated water. The variables were: solvent, temperature, and time.

Microwave-assisted extraction method. Microwave – assisted extraction **[24]** was carried out with Nevir NVR-6224 M microwave oven at 540 W power, varying the exposure time and solvent.

Solvent recovering. After the extraction procedure, the solutions obtained by ultrasound and microwave were filtered, while those obtained by Soxhlet did not need to be filtered. The solvents in the resulting solutions were evaporated using rotary evaporator IKA RV 3V (IKA, Staufen, Germany) at 100 °C. This concentrated solution was analysed by Folin-Ciocalteu colorimetric method to determine the total phenolic content and sent to the analysis performing laboratory CACTI to identify the phenolic compounds by HPLC measurement. The extracted chestnut shell samples were dried in the oven at a temperature just under 100 °C to a constant mass. This residue was weighed on a pre–weighed holder, and the total extraction yield was calculated.

Statistical analysis

A two-way analysis of variance (two-way ANOVA) was performed when there are two variables (ultrasound-assisted extraction method) and one dependent variable (Soxhlet and microwave-assisted extraction method). The test was carried out with Statgraphics Centurion 19 software, determining coefficient of determination (R^2 and adjusted R^2), probability value (p-value), and Fisher's variance index (F-value), using a 95 % confidence level. In the case of the experiment using ultrasound as the extraction method, a multidimensional ANOVA was used with the same confidence conditions.

Experimental design

An experimental design was carried out to determine the optimal conditions for extracting phenolic compounds for each extraction method. For this purpose, the Response Surface Methodology (RSM) was employed, RSM comprises a set of statistical and mathematical techniques designed to optimize and improve processes by evaluating the effects of multiple independent variables on a given response. **[25]**. A functional relationship is developed between the response and the number of variables, which can be approximated with polynomial and linear models by modifying them until the optimal conditions are reached. In case the variables do not fit a linear model, the second-degree model is used (**Equation (1)**)

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{1 \le i \le j}^{k} \beta_{ij} x_i x_j + \varepsilon \tag{1}$$

Where: k: number of variables; β_0: constant term; β_i: coefficients of the linear parameters; $x_{i,j}$: variables; ε: residual associated to the experiments; β_{ij}, coefficients of the interaction parameters.

The calculations to obtain the response surfaces were carried out with the Design-Expert 13 software (StatEase). Depending on the variables in each method, a three-level factorial design (3^2) or a Box-Behnken design was used.

Extraction yield calculation

The extraction yield of phenolic compounds from chestnut shells by all extraction methods was calculated with **Equation (2) [24]**.

$$Yield\ (\%) = \frac{\left(1 - \frac{Hm}{100}\right) \times (Mraw2) \times Ns)}{\left(1 - \frac{Hm}{100}\right) \times (Mraw1 \times Ns)} \tag{2}$$

Where: Y is the total extraction yield; Hm is the humidity of the sample; $Mraw^1$ is the mass of the sample before extraction (g); $Mraw^2$ is the mass difference between initial sample and sample after extraction (g); Ns is the number of samples.

Chemical characterisation

The extracts obtained were analysed in the laboratory Wood Chemical Transformation Laboratory of the Faculty of Forestry Engineering of Pontevedra, using the Folin-Ciocalteu colorimetric method [8], to determine the total phenolic content of the samples. The samples were diluted with distilled water, sodium carbonate, and Folin-Ciocalteu's reagent. Gallic acid was used as the standard for the estimation of phenolic compounds. The LLG-uniSPEC 2 spectrophotometer was used for this purpose, working at a wavelength of 765 nm. A gallic calibration curve was obtained with seven concentrations (0.1, 0.2, 0.3, 0.4, 0.7, 1 and 1.2 mg/L). Total phenolic content (TPC) was calculated using the **Equation (3)** and expressed as mg gallic acid equivalent (GAE) per g of plant material (mg GAE/mg).

$$TPC = \frac{CGA \times V}{M} \tag{3}$$

Where: CGA: concentration of gallic acid equivalent (mg/mL); V: volume of extracts used for experiments (mL); M: mass of the chestnut shell used (g).

The samples that showed a higher concentration of phenolic compounds were analysed at the CACTI laboratory in Ourense by High Liquid Performance Chromatography (HPLC) to identify the phenolic compounds obtained, as the Folin-Ciocalteu method does not allow the identification of these compounds. A Shimadzu chromatograph equipped with a UV-DIODE ARRAY detector was used to identify flavonoids and phenols.

Biochar production

The resulting shell residue after carrying out the extraction was placed in a ceramic crucible and then in the furnace with an initial temperature of 160 °C. The temperature progressively increased until it reached 400 °C during 60 min. The sample was weighed before and after pyrolysis to calculate the yield using **Equation (4)**, also calculating the percentage of ash and volatiles obtained.

$$Biochar\ production\ yield = \frac{(BM - CM)}{BM} \times 100 \tag{4}$$

Where: BM: mass of the biomass; CM: mass of carbonised biomass.

RESULTS AND DISCUSSION

Humidity of the samples

The moisture content of the samples was determined following the guidelines of the Spanish standard UNE-EN ISO 18141-3 and is shown in **Table 2**.

Table 2. Moisture content different sample

Sample	Moisture (%)
1	20.63
2	32.99
3	84.21
4	26.74
5	27.23

Extraction performance of the different methods

Figure 1 shows the yields obtained for each of the methods used.

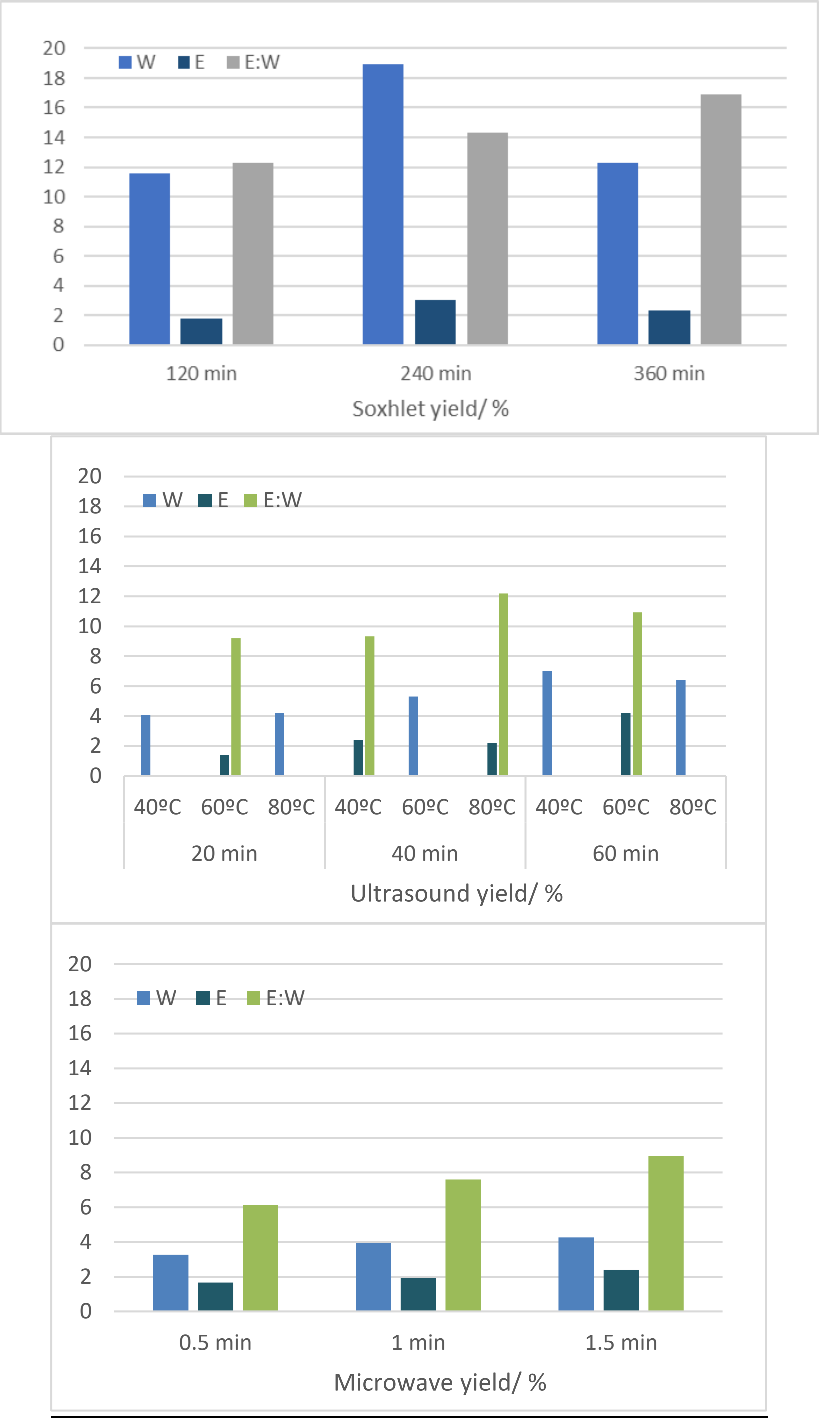

Figure 1. Influence of solvent types and times on extraction performance depending on the method used

According to previous studies, microwave-assisted extraction slightly improved extraction compared to solvent maceration extraction [26]. The best results are obtained with the Soxhlet method with yields close to 20 %. The highest yield for this method was achieved with distilled water as a solvent at a time of 4 h, reaching a value of 18.94 ± 0.67, followed by the mixture of ethanol and water at a time of 6 h, with a yield of 16.93 ± 0.40. The highest yields with ultrasound-assisted method are obtained with the mixture of ethanol and water. The highest value (12.2 ± 0.2 %) is obtained during a time of 40 min operating at a temperature of 80 °C, followed by yield of 10.93 ± 0.0 % which is obtained by operating for 60 min at a temperature of 60 °C. The microwave-assisted extraction method generally achieves low yields. The extractions performed with the solvent mixture do not reach 10 %. The highest yield with this method is achieved with the solvent mixture for a time of 1.5 min reaching a value of 8.94 ± 0.94 %. Ethanol is the worst-performing solvent for all three methods used, as the values for the extraction yield do not exceed 4 %. While ethanol-water solvent reaches similar values to others solvents in Soxhlet, in the other two methods, ultrasound-assisted and microwave-assisted extraction, it can be seen that this mixture obtains the best results. The ethanol: water mixture gives the best extraction results because the water swells the material, allowing the solvents to penetrate the plant fibres, which increases extractability due to increased specific surface area of the material (percentage). On the other hand, organic solvents inactivate an enzyme that oxidises phenolic compounds, polyphenol oxidase, which could decrease the total phenolic content in the extracts if they are obtained with 100 % water.

Figure 2 shows the 2D contour and 3D response surface plots for the Soxhlet extraction method, showing the influence of the studied variables, time (A) and solvent (B), on the optimisation of the phenolic content obtained from the samples by means of a three-level factorial response surface design (3^2). The graph shows that the best results are obtained with an exposure time between 2 and 4 h using distilled water as solvent (in red). The ANOVA test carried out for the three methods, for ultrasound-assisted extraction method based on the Box-Behnken statistical design for the three independent variables. These variables are statistically significant ($p < 0.05$) and the relationships between them are only significant for the quadratic variable of the solvent CC ($p = 0.02$). This model is significant since the coefficient of determination is 95.95 and the adjusted one is 88.66. The ANOVA test for the microwave-assisted extraction method is performed with two independent factors. The solvent and extraction time are significant ($p < 0.05$), but the interactions between them do not influence the experiment. The model is significant (adjusted $R^2 = 93.65$). The statistical analysis carried out for the Soxhlet extraction method, for which a Box-Behnken experimental design has been used, shows that the only variable that could be adapted to the 95 % probability test is the solvent ($p = 0.0647$).

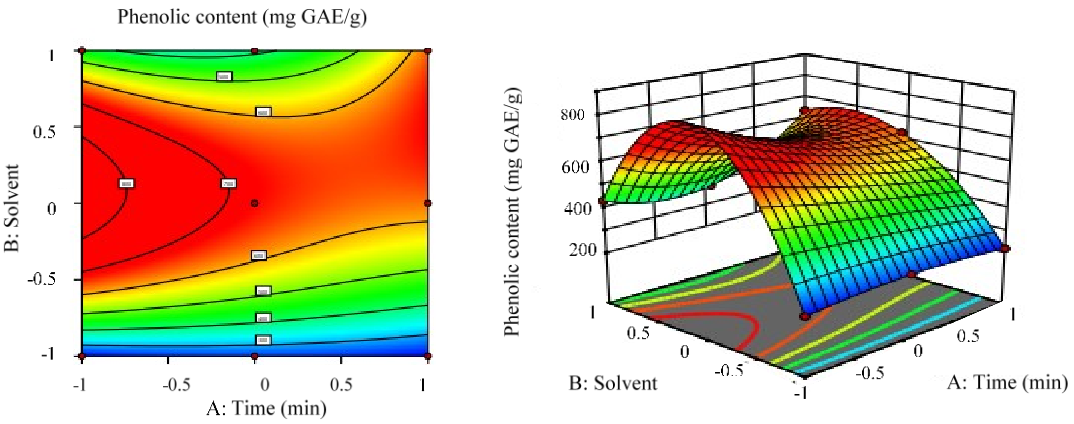

Figure 2. 2D contour plots and 3D response surface for Soxhlet method

Content of total phenolic compounds obtained by the extractions

Table 3 shows the total phenolic content. The highlighted cells represent the optimised conditions for carrying out the extractions. Solvents: distilled water (W), ethanol (E) and a mixture of water and ethanol 50:50 (E:W).

Table 3. Total phenolic content (mg GAE/g) obtained by Soxhlet method

Solvent	Time/ min (level)	Time/ min	FT (mg GAE/g)
(W)	-1	120	411.49 ± 34.36
	0	240	679.69 ± 47.18
	1	360	628.92 ± 29.23
(E)	-1	120	114.56 ± 9.23
	0	240	244.31 ± 41.54
	1	360	219.18 ± 27.69
(E:W)	-1	120	433.54 ± 92.31
	0	240	385.33 ± 3.08
	1	360	635.59 ± 12.31

In the case of Ultrasound-assisted method, the trend is similar (**Table 4**). The highlighted cells represent the optimised conditions for carrying out the extractions. Solvents: distilled water (W), ethanol (E) and a mixture of water and ethanol 50:50 (E:W). The best solvents are water and ethanol:water mixture. In the case of water, the highest phenolic content is obtained with a duration of 60 min at two temperatures, 40 °C (306.87 ± 17.95 mg GAE/g) and 80 ºC (439.69 ± 24.62 mg GAE/g).

Table 4. Total phenolic content (mg GAE/g) obtained by Ultrasound-assisted extraction method

Solvent	Level	Time/ min	Level	Temperature/ ºC	FT (mg GAE/g)
(W)	-1	20	-1	40	196.62 ± 40.00
	-1	20	1	80	262.26 ± 19.49
	0	40	0	60	286.36 ± 3.59
	1	60	-1	40	306.87 ± 17.95
	1	60	1	80	439.69 ± 24.62
(E)	-1	20	0	60	116.62 ± 3.08
	0	40	-1	40	105.33 ± 17.44
	0	40	1	80	200.21 ± 12.82
	1	60	0	60	187.9 ± 13.85
(E:W)	-1	20	0	60	299.18 ± 20.51
	0	40	-1	40	343.28 ± 0.00
	0	40	1	80	319.69 ± 35.90
	1	60	0	60	368.41 ± 12.82

Table 5 shows total phenolic content obtained by Microwave-assisted extraction method. The highlighted cells represent the optimised conditions for carrying out the extractions. Solvents: distilled water (W), ethanol (E) and a mixture of water and ethanol 50:50 (E:W).

Table 5. Total phenolic content (mg EAG/g) obtained by Microwave-assisted extraction method

Solvent	Level	Time/ min	FT (mg GAE/g)
Water (W)	-1	0.5	100.72 ± 24.10
	0	1	218.67 ± 73.33
	1	1.5	225.85 ± 18.97

Ethanol (E)	-1	0.5	56.62 ± 11.79
	0	1	59.18 ± 15.38
	1	1.5	79.69 ± 13.33
Ethanol:Water (E:W)	-1	0.5	162.77 ± 25.64
	0	1	264.82 ± 20.00
	1	1.5	320.21 ± 30.26

Figure 3 and **Table 6** show the total phenolic content determined by the three methods to have a joint vision. The Soxhlet extraction method gives the highest overall total phenolic content. For this method, the water and the mixture used as extractants obtain very similar results, whose maximum values are in the range between 600 and 700 mg GAE/g. On the other hand, ethanol recorded the worst results, not reaching 300 mg GAE/g. The results obtained by the ultrasound extraction method do not exceed 500 mg GAE/g, similar to the previous case, where the results obtained with water and the mixture are very similar, while the results for ethanol do not exceed 200 mg GAE/g. The results obtained with the microwave extraction method are the lowest, with a value of 300 mg GAE/g for the highest value obtained with the solvent mixture. In this case the results with water do not exceed 200 mg GAE/g and in the case of ethanol, the results do not exceed 100 mg GAE/g. Ethanol has the worst extraction results in all three methods.

Table 6. Values obtained by HPLC for the solutions of each of the extractions and the retention time for each compound (tR). Solvents: water (W), ethanol (E) and a mixture of water and ethanol 50 % (E:W). Methods: Soxhlet (S), Ultrasound (U) and Microwave (M)

Variables				Compound/ mL							
Solvent	Method	T/ ºC	t/ min	PA	PHA	PHEB	FA	R	Q	R1	R2
W	S	-	240	0	0	0	0	111	0	0	0
	U	40	60	118.4	0.7	0.1	1.7	2.2	0	0	0
		80	60	0	0	0.3	0	4.5	0.1	0	0
	M	-	1	66.4	0	0	0	18	0	0	0
		-	1.5	0	0.7	0.1	0	3.4	0	0	0
E	S	-	240	0	0	0	0	21	3.1	0	0
		-	1.5	0	0	0	0	1.1	0	0	0
E:W	S	-	360	0	0	0	0	66.8	1.5	4	0.3
	U	40	40	0	0	0	0	10.4	0.9	0	0
		60	60	36.1	0	0	0	18	0	0	0
	M	-	1.5	0	0	0	0	8	0.7	0	0
		-	1	0	0	0	3.5	6.2	0.5	0	0
t_R / min				6.5	7.9	9.2	15.3	25.3	29.1	26.9	28.1

PA: Protocatechuic acid, PHA: p-Hydroxybenzoic acid, PHB: p-Hydroxybenzaldehyde, FA: Ferulic acid; R: Rutin, Q: Quercetin, R1: Rutin derivative nº1, R2: Rutin derivative nº2

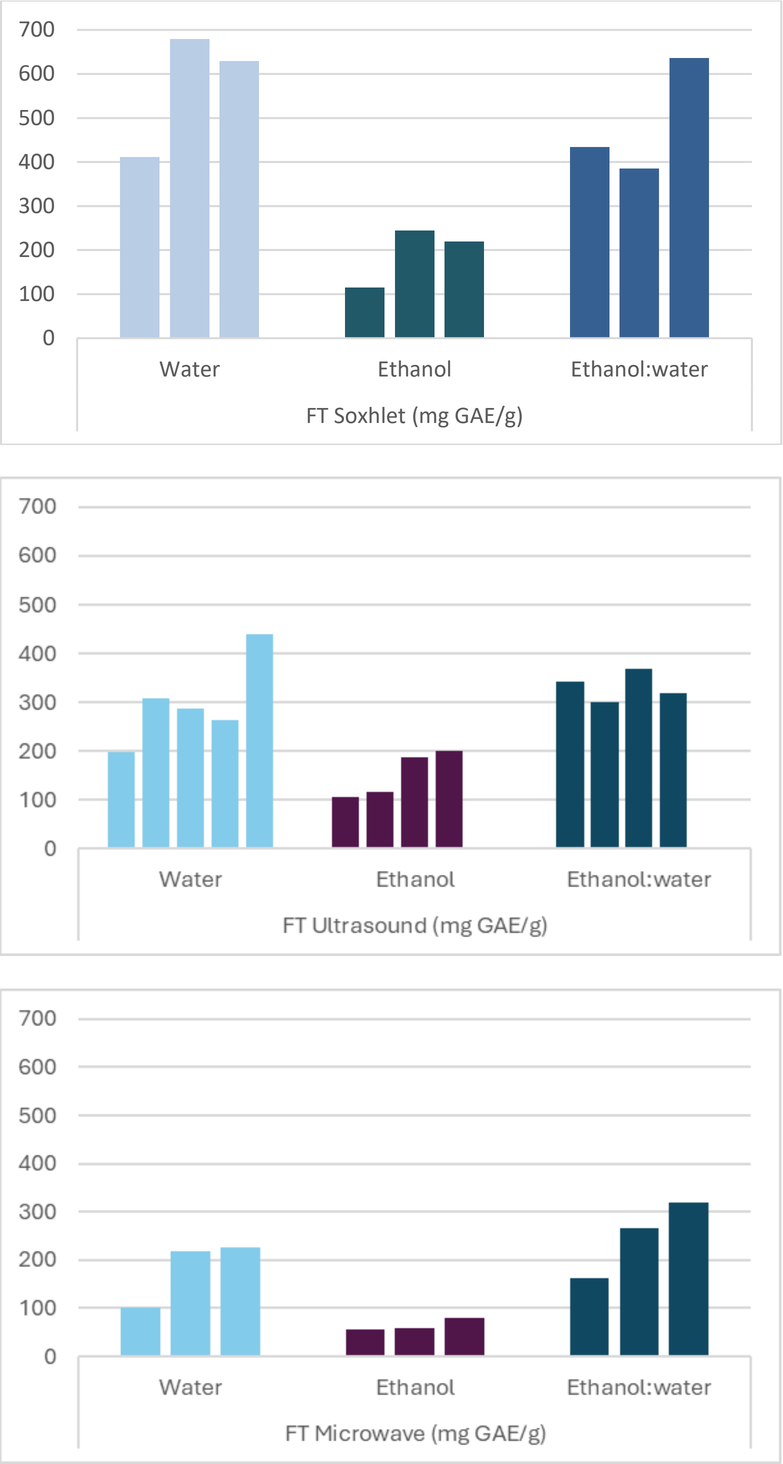

Figure 3. Total phenolic content (mg AGE/g) obtained by the different methods used

The compounds identified in this study were protocatechuic acid, p-hydroxybenzoic acid, p-hydroxybenzaldehyde, ferulic acid, rutin, quercetin and two unidentifiable flavonoids, as they were peaks with spectra very similar to that of rutin, but differed from it by retention time and slight deviations in the spectrum.

The compounds obtained by Soxhlet were rutin in the three conditions studied, quercetin in one case with ethanol and for 240 min and in a second case with ethanol:water for 360 min. The two unidentifiable flavonoids were obtained under the same conditions, with the etanol:water mixture and for 360 min. The Soxhlet method favours the extraction of the most complex compounds, obtaining rutin under all the conditions studied.

For the ultrasonic method, all compounds except the two unidentifiable flavonoids were obtained. In the case of protocatechuic acid, the conditions under which the highest amount of compound was obtained were with water at 40 °C and for 60 min, while the lowest amount was obtained with the mixture of ethanol and water at 60 °C and for 60 min. It is observed that considering the same time interval, the extraction was better at a lower temperature (40 °C) than at the higher temperature (80 °C). In the case of the ethanol:water mixture, the extraction was better at higher temperature and for a longer time (tª 60 °C and for 60 min), while a lower values (tª 40 °C and for 40 min) this compound is not obtained. Using water as solvent, at a temperature of 40 °C and for 60 min, all compounds except quercetin and the two unidentifiable flavonoids are obtained. In the case of quercetin, quercetin is only obtained under two conditions: with water at 80 °C for a 60-min exposure; and with the ethanol:water mixture at 40 °C for 40 min. Rutin is obtained in all the cases studied, obtaining a better value using the ethanol:water mixture as solvent, with a temperature of 60 °C and during 60 min of exposure. With the ultrasonic method, very little quantity of the following compounds is obtained: p-hydroxybenzoic acid, p-hydroxybenzaldehyde and ferulic acid.

Using the microwave method, the compound obtained is mainly rutin. Also 66.4 mL of protocatechuic acid are obtained with water for 1 min. The conditions under which the other minority compounds were obtained (p-hydroxybenzoic acid, p-hydroxybenzaldehyde, ferulic acid and quercetin) are with water for 1 min and with the ethanol:water mixture at two times, 1 min and 1.5 min.

Rutin was the compound obtained by all three methods and in the majority of the cases, while the two unidentifiable flavonoids were obtained in smaller quantities. Protocatechuic acid and rutin are the most mainly obtained. It is observed that the first compounds to come out are those of lower molecular mass. In the case of rutin and quercetin, although rutin is a bulkier structure than quercetin, it elutes before quercetin.

Analysis of biochar

The biochar yield (**Table 7**) is obtained by the difference between the weight of the sample before and after the pyrolysis process, as well as the percentage of ash and volatiles. Yields for the production of biochar from chestnut shells were 47 % and the percentage of ashes and volatiles generated during the process were high (**Table 8**).

Table 7. Yield of biochar obtained from chestnut shells

Sample	Biochar	Yield (%)
21.26	11.26	47.07
22.00	11.58	47.38

Table 8. Ash percentage and Percentage of volatiles for biochar and for the sample

Biochar Ash				Sample Ash			
Crucible	Sample	Ashes	% ashes	Crucible	Sample	Ashes	% ashes
31.33	33.33	31.37	94.12	32.26	34.04	32.28	94.81
Biochar Volatiles				**Sample Volatiles**			
Crucible	Sample	Volatiles	% volatiles	Crucible	Sample	Volatiles	% volatiles
38.24	39.45	38.97	98.8	33.21	34.28	33.47	97.63

CONCLUSIONS

This study tests the feasibility of a method for using waste from a product destined for consumption such as chestnuts. To this end, three methods for extracting phenolic compounds from chestnut shells are tested. The phenolic compounds are isolated and the resulting residue after the extractions is transformed into biochar through a fast pyrolysis process. Rutin is the compound obtained by all extraction methods, which together with protocatechuic acid gives the best extraction results. In the case of acid, it is not obtained in all the experimental conditions studied, unlike rutin. It is observed that the flavonoids obtained from chestnut shells (rutin and quercetin) are obtained without problem by all methods. With rutin, good results are obtained in all cases, while for quercetin, better results are obtained using the Soxhlet method with ethanol present in the solvent, both alone and in the mixture. For the other flavonoids identified (the rutin derivatives), these are only obtained by the Soxhlet method and with the ethanol:water mixture. Few quantities are obtained, especially of the latter (there is no difference because the structures are not known). The best methods for obtaining phenolic acids were ultrasound and microwaves, although in general, with the exception of protocatechuic acid, very small quantities are obtained from all of them. The best conditions for obtaining the protocatechuic acid were ultrasound (solvent: water, temperature: 40 °C, time: 1 h), where the greatest amount of acid was obtained, and (solvent: mixture, temperature: 60 °C, time: 1 h), where the least amount was obtained. In the case of the microwave, this compound was only obtained under certain conditions, using water as solvent for 1 min in the microwave.

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