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Ultrasound-assisted extraction of a condensed tannin and its application for removal dyes from water

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Abstract

The woody oil industry generates a large amount of seed shell waste during its production. Therefore, this paper employs a central composite design (CCD) methodology to optimize the ultrasonic-assisted extraction process, thus utilizing the byproduct. The infrared spectrum and thermogravimetric analysis is adopted to explore the chemical structure and thermal stability of tannin. Besides, the tannin resin (TF) is prepared to elaborate its adsorbing performance for dyes in water. The results verify the role of ultrasonic-assisted extraction method in increasing yield rate. The optimal conditions are as follows: 60% ethanol as extraction solvent, the solid-liquid ratio of 1:25 (g/mL), ultrasonic power of 160 W, and extracted at 80°C for 60min. The yield rate of tannin under such conditions stands at 32.04% (w/w). Temperature is vital for the extraction rate. *Acer truncatum* tannin as a condensed tannin, performs favorable thermal stability, and TF effectively absorbs cationic dye in water. The maximum adsorption capacity of methylene blue peaks at 176.13 mg/g. *Acer truncatum* shell serves as a resource of condensed tannin, and *Acer truncatum* tannin resin can be potentially developed into a new biomass adsorbent.

Keywords: tannin; adsorbent; dye; response surface method.

Practical Application: Food factories may use a large amount of wastes from their plant oil processing to prepare biomaterials. So that they can reduce wastes and improve their economic benefits.

1 Introduction

Acer truncatum Bunge refers to a unique ornamental plant and economic-tree species in China, whose leaves contain plenty of bioactive compounds (Fan et al., 2022) covering flavonoids, organic acids, cardiac glycosides, tannin, terpenes, and phenols. In addition, the leaves with strong physiological effects are produced as a functional food. The seed of *Acer truncatum* includes 48% (w/w) edible oil that abounds unsaturated fatty acids covering a vital fatty acid named nervonic acid, which prevents brain nerve aging (Li et al., 2021; Fan et al., 2022). The output of the seed oil has jumped in recent years, bringing about substantial seed shell wastes.

Tannin, as a natural polyphenol compound mainly found in plant wood, bark, seeds, fruits, leaves, especially in some foods (Belkacemi, 2022; Santos et al., 2022), falls into two camps based on chemical structure: condensed tannins, and hydrolyzable tannins. The former refers to the condensation products of hydroxyflavanes, which are interconnected by C-C bonds and are not easy to decompose in water, while the latter is an ester whose ester bond is easy to hydrolyze under the action of alkali, enzyme or acid. Tannin can react with enzymes and metal ions, and is able to combine with proteins, generating pharmacological effects on convergence, anti-inflammation, sedation, anticoagulation, anti-oxidation, and anti-tumor (Manzoor et al., 2022; Ha et al., 2022; Das et al., 2020; Fraga-Corral et al., 2020). What's more, its extensive application can be observed in other various aspects including tanning agents' adhesives, additives, preservatives, and water treatment (Das et al., 2020; Guo et al., 2020).

Tannin is generally extracted by water or organic solvent method (de Hoyos-Martinez et al., 2019; Das et al., 2020). Compared with other new technologies including supercritical fluid extraction, subcritical fluid extraction, ionic liquid extraction, and microwave assistant extraction, ultrasonic-assisted extraction has been widely recognized for its lower cost and simple operation (de Hoyos-Martinez et al., 2019; Fraga-Corral et al., 2020). The ultrasound process shows a good synergistic effect in food processing (Guimarães et al., 2021). Compared with high-temperature short-time sterilization, the combination of ultrasound and heat treatment can reserve more active substances and produce better quality cheese (Scudino et al., 2022b). And it is proved to be efficient in treating Jamun fruit dairy dessert (Lino et al., 2022). The high-intensity ultrasound also can improve the physical stability and change the bacterial diversity of the milk during refrigerated storage (Scudino et al., 2022a).

Response surface methodology (RSM), a comprehensive optimization method of experimental design and mathematical modeling, effectively reduces the number of experiments and investigates the interaction between processing factors (Luo et al., 2019; Rhazi et al., 2019). The central composite design (CCD)

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serves as one of the most used response surface methods in process design, especially for optimization of extraction (Bae et al., 2015; Belwal et al., 2016). The prediction results are reliable.

Given its planting area of above 40,000 hectares in China, *Acer truncatum* witnesses an annual output of about 4500 kg seeds per hectare in the best scenario, and 2250 kg seed shell after oil-processing. Its seed shell tannin stands out due to its rich resources, renewability, pollution-free, biodegradability, and low cost, which supports the urgency to optimize the extraction process and develop biomass products, thus making full use of the resource and comprehensively benefiting the *Acer truncatum* industry.

Acer truncatum seed shell tannin can be extracted with water as extraction solvent and ultrasonic-assisted extraction, however, the process has not been optimized, nor has the tannin been adopted as an adsorbent. As a result, this paper takes lower cost ethanol and water as solvents to extract tannin through ultrasound-assisted extraction from *Acer truncatum* shell, and CCD response surface methodology is employed to optimize the extraction process. The application of tannin in the adsorption of dyes is also explored.

2 Materials and methods

2.1 Materials

The seed shell residue employed in the paper was provided by an *Acer truncatum* Bunge oil factory. The shell was dried at 60 °C for four hours, sieved through a 30-mesh screen after crushing, and stored in a refrigerator for further use. Tannin acid (≥95%) and Folin-Denis reagent were purchased from Solarbio company (Beijing, China). Potassium bromide was the chromatographic grade, while Methylene blue (MB), Congo red (CR) and other chemicals analytical grade. Water adopted was deionized water.

2.2 Extraction method

The extraction was performed in a SB-5200 DT ultrasonic device (Ningbo Xinzhi Ultrasonic Co. Ltd., China). The dried shell residue was mixed with ethanol solution in a flask equipped with condensing device at designed condition. Following extraction, the mixture was centrifuged at 8000 rpm for 10 min, and the tannin content in the supernatant was measured.

2.3 Determination of tannin content

The Folin-Denis method (Belwal et al., 2016) was employed to measure tannin content in the extraction solution. Briefly, the mixture of 0.5 mL of Folin-Denis reagent and 5 mL of diluted extract solution was prepared, which was then added with 3.5 mL of distilled water and 1.0 mL of 7% sodium carbonate at room temperature. After standing in the dark for 20 min, the absorbance was measured at 700 nm against blank (distilled water) using UV-VIS spectrophotometer (UV-1800, Mapada, China), obtaining a standard curve of tannic acid. Tannin extracted yield was expressed as a percent of tannic acid equivalent to dry weight of seed shell wastes.

2.4 Optimization of tannin extraction process

A CCD method was used in experiments to investigate the extraction factors and optimize the process. The extraction variables consist of ultrasonic time (A, min), ultrasonic power (B, W), and extraction temperature (C, °C), each of which was coded into 5 levels (0, ±1, ±α), where "0" serving as the center point, "±1" the low and high level, and "±α (α = 1.68179)" the extreme value corresponding to the axial point. What's more, the response value standed for the extraction yield of tannin (Table 1).

2.5 Preparation of tannin resin

The preparation of *Acer truncatum* shell tannin was as follows. One hundred grams of defatted shell powder was extracted based on the optimized extraction method, which was concentrated at 40 °C, and pre frozen to solid at -20 °C. Then, the extract was freeze-dried by a vacuum freeze dryer (FD5-3T, Jinximeng Co., Ltd., Beijing) at -58 °C for 12 h.

The preparation of tannin resin (TF) was based on the published methods (Torrinha et al., 2020) with a minor modification. Tannin extract (1.0 g) was dissolved in 20 mL of sodium hydroxide solution (0.25 mol/L), and reacted with formaldehyde solution (37 wt%, 4 mL) at 85 °C for 8 hours. After cooling, the obtained solid was washed and dried at 60 °C in a vacuum oven, and grounded into powders for further use.

2.6 Adsorption of dyes

To determine TF adsorption for dyes, 20 mg of adsorbent was mixed with 25 mL of MB solution or CR solution, and vibrated (180 rpm) for 4 hours at 30 °C. After filtration, the dyes content was measured with an ultraviolet spectrophotometer. The adsorption capacity and rate of TF was calculated according to the Equation 1 and Equation 2,

$$E = \frac{(C_0 - C_e)}{C_0} \times 100\% \quad (1)$$

$$Q_e = \frac{(C_0 - C_e)V}{m} \quad (2)$$

where, E refers to the adsorption rate (%), Q_e the adsorption capacity (mg/g). C_0 and C_e the initial concentration and equilibrium concentration of dyes (mg/L), V the volume of the solution (mL), while m the weight of the absorbent (mg).

Table 1. Codes and levels of factors in central composite design.

x_i Level	Code		
	A/min (Time)	B/W (Power)	C/°C (Temperature)
-α	6.36	52.73	26.36
-1	20	80	40
0	40	120	60
1	60	160	80
+α	73.64	187.27	93.64

2.7 Data analysis

Design Expert 10.0.3 was adopted for experimental design, Office Excel 2013 and SPSS Statistics 17.0 for data processing and statistical analysis, one-way ANOVA and Tukey for the significance of difference, while Origin 2020 for mapping. All experiments were conducted at least thrice.

3 Results and discussions

3.1 Optimization of ultrasonic-assisted extraction process

Model fitting and optimization

Table 2 lists the 20 experimental conducted according to the CCD. The input factors and the levels are determined by single factor experiments, and each experimental run performs a combination of factor and level. The extraction yield ranging from 11.94% to 31.12% is calculated with Equation 1 and listed in Table 2.

Table 2. Experimental design and results of CCD.

run	A /min	B/W	C /°C	Extraction rate/%
1	40	52.7283	60	26.6
2	40	120	60	25.6
3	60	160	40	15.79
4	40	120	26.3641	11.94
5	73.6359	120	60	27.38
6	60	80	40	15.71
7	20	160	80	31.12
8	20	80	40	14.73
9	40	120	60	25.64
10	60	80	80	29.28
11	40	120	60	25.8
12	40	120	60	27.23
13	40	120	93.6359	28.83
14	60	160	80	31.08
15	20	160	40	13.73
16	40	120	60	23.58
17	6.36414	120	60	24.31
18	40	187.272	60	26.12
19	40	120	60	26
20	20	80	80	22.93

The regression analysis of the results is implemented, obtaining the following regression equation:

Analysis of variance (ANOVA) confirms the adequacy of the prediction model by evaluating the lack of fit, the regression coefficient (R^2), and the Fisher test value (F-value) (Bagheri, 2019). Despite the significant fitting model, the value gap between Adj R-Squared and Pred R-Squared is obvious, with their square deviation more than 0.2, indicating the insufficient explanation, the poor adaptability of data, and the necessity to optimize for a more reasonable model (Luo 2012).

Following a model analysis that appoints the standard of automatic screening as Ad-squared and other variables unchanged, an optimized model Equation 3 is obtained,

$$Y = -5.22580 + 0.053135 \times A - 0.087156 \times B + 0.078513 \times C + 1.70469 \times 10^{-3} \times B \times C - 5.71957 \times 10^{-3} \times C^2 \quad (3)$$

Table 3 proves the highly significant optimized model equation, and the negligible difference of lack of fit. Besides, Pr>F=0.0755>0.05 confirms the appropriate fitted model.

The optimized equation shows a R-Squared value of 0.9178, indicating a favorable fit, and the modified model sufficiently reflects the observed values. The values of Adj R-squared and Pred R-squared are high and close, with a square deviation less than 0.2, indicating the model's full explanation of the process. The C.V. % (8.38) less than 10 verifies the reliability and accuracy of the experiment, among which accuracy refers to the ratio of effective signal to noise. Adeq Precision, usually employed to represent the noise ratio, stands at 21.622 and outnumbers 4, suggesting a desirable adequate result. The modified fitting equation performs favorable adaptability after testing (Luo, 2012; Belwal et al., 2016).

Table 3. Results of ANOVA.

	Sum of squares	Degree of Freedom	Mean square	F value	P value	Significance
Model	614.79	5	122.96	31.27	<0.0001	yes
A	15.42	1	15.42	3.92	0.0667	
B	5.00	1	5.00	1.27	0.2785	
C	502.68	1	502.68	127.82	<0.0001	yes
BC	14.88	1	14.88	3.78	0.0721	
C^2	76.81	1	76.81	19.53	0.0006	yes
Residual	55.06	14	3.93			
Lack of fit	48.13	9	5.35	3.86	0.0755	no
Pure error	6.93	5	1.39			
Total	669.85	19				
Standard Deviation	1.98		R-Squared	0.9178		
Mean	23.67		Adj R-Squared	0.8885		
Mean Coefficient of Variation %	8.38		Pred R-Squared	0.7524		
PRESS	165.82		Adeq Precision	21.622		

Response surface analysis

The term "BC", shows their influence and interaction on tannin yield clearly illustrated in Figure 1. The increase of B (power) and C (temperature) brings about the growth, even to the peak, of the tannin content, validating the negative impact of any further increase of these two factors on the extraction yield (Zheng et al., 2022). Higher temperature increases the movement of molecules, thus facilitating the extraction of tannin. What's more, higher ultrasonic power, combined with higher temprature, brings about more favorable extraction rate, which can be explained by the role of ultrasonic in strengthening the mechanical and cavitation effect and the thermal effect of the whole system (Zhang et al., 2022; Guo et al., 2022). The extraction rate shows a greater slope along the C-axis, indicating that temperature had a greater impact on the extraction rate than ultrasonic power. It was consistent with ANOVA results.

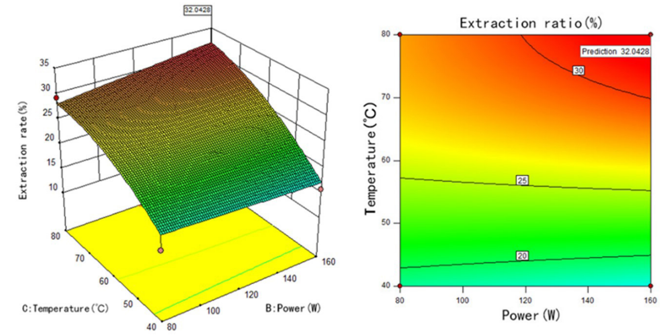

Figure 1. Response Surface and Contour of ultrasonic power (B) and temperature (C).

3.2 IR of Acer tannin

Figure 2 reveals the infrared spectrum of the *Acer* seed shell tannin. The strong absorption peak near 3400 cm^{-1} is related to the stretching vibration peak of —OH, while the absorption peak at 2937 cm^{-1} to the stretching vibration peak of C—H bond. The peaks at 1610 cm^{-1} and 1524 cm^{-1} are the characteristic bands of aromatic ring stretching vibration (Li et al., 2019). The IR spectrum of the tannin is very close to catechin and extracted tannin from *P. radiata* bark, and the aromatic nature of the extract is evidenced by signals at 1400 to 2000 cm^{-1} range and under 900 cm^{-1} (Soto et al., 2005). Bands at 1110 and 1280 cm^{-1} that show the presence of condensed tannins are absent in the spectra of gallo- and ellagi tannins (Falcão & Araujo, 2013).

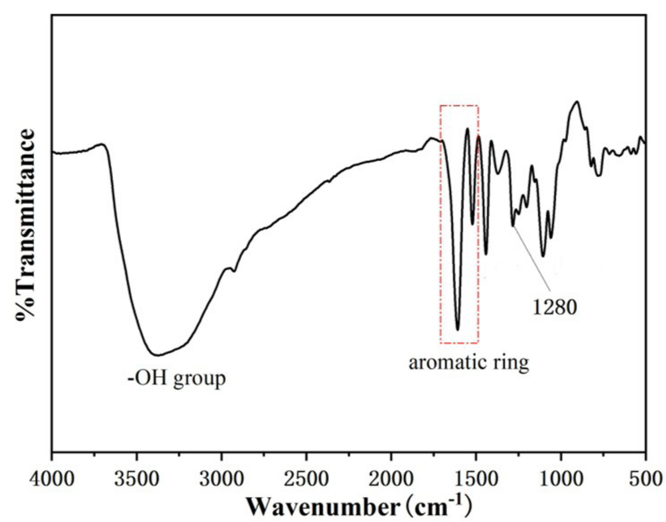

Figure 2. FT-IR spectrum of *Acer* tannin.

3.3 TGA of Acer tannin

Figure 3 depicts the thermogravimetric analysis of the tannin. The weight loss of tannin is divided into two stages. The former starts from room temperature to 156.3 °C with a wight loss of 7.65%, which comes down to the sample's evaporation of water. Besides, certain low molecular weight in the extract, such as organic acids and sugars, are decomposed. The latter is from 156.3 °C to 900 °C with a weight loss of 48.08%, during which *Acer* tannin starts to decompose. What's more, the tannin composite peaks at 271.7 °C, and the residue rate is 44.27% at the end of the decomposition (900 °C). *Acer* tannin extract shows better thermal stability as radiata pine condensed tannins that start to degrade at 150 °C (Luo et al., 2010), and hydrolyzable tannins of pomegranate peels (Saad et al., 2012) start to degrade at 149 °C with a residue rate of 36.4%. The diverse thermal decomposition of tannins is associated with their composition and structure, polymerization degree, and the nature of interflavonoid bonds (Ben Mahmoud et al., 2015).

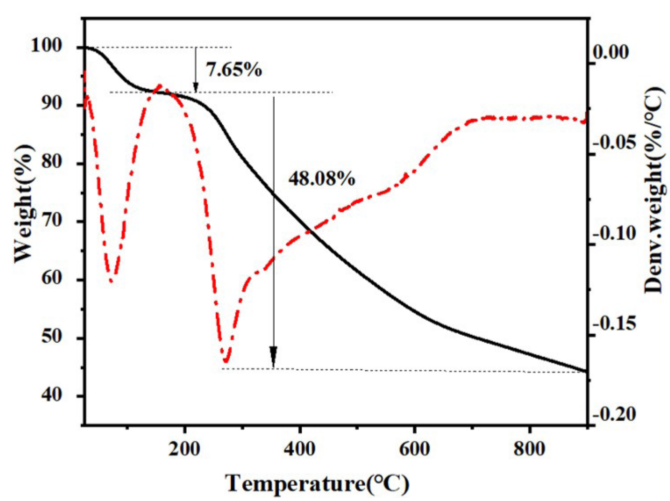

Figure 3. Thermal weight analysis of *Acer* tannin.

3.4 Adsorption for dyes

Basic dyes, which are substances with cationic behavior in aqueous solution, are mostly studied as a removal target by the tannin-based adsorbent, among which MB usually serves as a model in studies (Bacelo et al., 2016; Dassanayake et al., 2021). Figure 4 supports the direct proportion between the adsorption of the tannin adsorbent and the initial concentration. The maximum adsorption to MB records 176.13 mg/g, far higher than to CR (11.08 mg/g). However, the adsorption rate is in inverse proportion to dye concentration. With CR and MB as

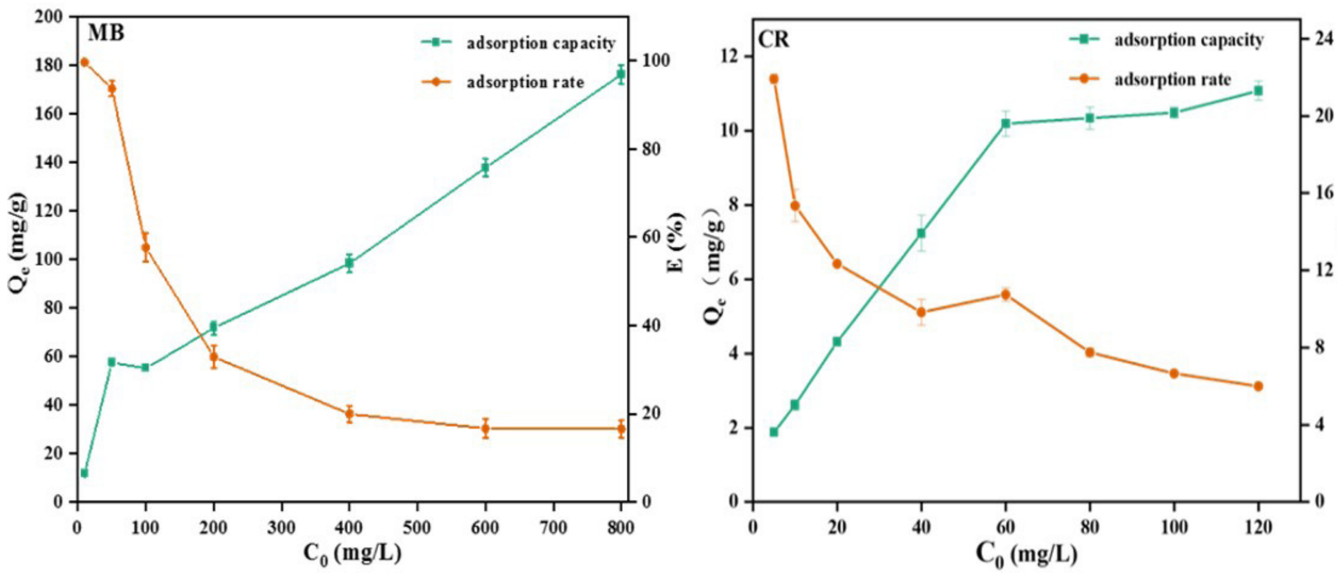

Figure 4. Adsorption for MB and CR.

textile dyes, the highest adsorption rate is 22% and more than 90%, respectively. It's obvious that TF's adsorption performance depends on the properties of the dye itself, and the TF displays better adsorption performance for MB (a cationic dye) than CR (an anionic molecular). The CR adsorbing ability of TF proves that tannin resins are not perfect adsorbents for anionic pollutants. However, tannin based gels induce adsorption of both cationic and anionic dyes, and tannin resins are excellent proven adsorbents for cationic species, such as heavy-metals and dyes, thanks to their anionic character (Bacelo et al., 2016).

4 Conclusion

The optimized ultrasonic-assisted extraction process of *Acer* seed shell tannin is as follows: 60% ethanol as extraction solvent, the solid-liquid ratio of 1:25 (g:mL), ultrasonic power of 160 W, and extract at 80 °C for 60 min. The tannin extraction yield could reach 32% (wt) under such conditions. The shell tannin is proved to be condensed tannin through infrared analysis. The adsorption capacity of TF to MB peaks with 176.13 mg/g. Tannin from *Acer truncatum* Bunge seed shells serves as a potential biomass material for cation dyes removal from water. Moreover, the application of condensed tannin relieves the formaldehyde emission in the resin system (Kizilcan & Sert, 2019; Li et al., 2019). *Acer truncatum* shell, as an industrial waste, can be widely sourced, which further supports the prospect of developing environmental-friendly *Acer* tannin-based adsorbents.

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