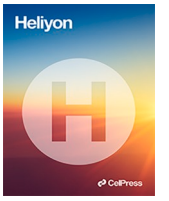
Heliyon 9 (2023) e13377

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Research article

Optimization of tannin extraction from coconut coir through response surface methodology

Rinlada Sirisangsawang, Natacha Phetyim *

Department of Chemical and Materials Engineering, Faculty of Engineering, Rajamangala University of Technology Thanyaburi, Pathum Thani, Thailand

ARTICLE INFO

Keywords:
Coconut coir
Solvent extraction
Tannin
Response surface methodology
Central composite design
LC–MS/MS

ABSTRACT

Coconut coir is produced by extracting tiny grains of coir from coconut shell, which is an agricultural product that is abundant in Thailand. Coir is typically discarded, but it is a useful material because it contains tannins. Tannin is a polyphenolic compound that is prevalent in the plant kingdom, including in wood, bark, rhizomes, roots, and fruits. In this study, we extracted tannins from coconut coir using two types of solvents: water and ethanol. Optimization of the tannin content was achieved via RSM (response surface methodology) with the assistance of Design-Expert software. Meanwhile, the temperature (30–70 °C), ratio of solid to solvent (1:20–1:40), and solvent type (water or ethanol) were optimized via central composite design (CCD). The optimum model using analysis of variance revealed R^2 = 0.9825, and adj.R^2 = 0.9687. Temperature is affected by tannin content, and high temperatures result in a higher tannin content when using water than when using ethanol. Optimum conditions for coconut coir extraction using water with ethanol include a ratio for solid to solvent of 1:30 and the temperature set to 70 °C. The validated model exhibits errors of 8.24 and 11.08% for water and ethanol, respectively. Confirmation of the presence of tannins in the crude extract was carried out via Fourier-transform infrared spectroscopy and through the use of liquid chromatography with tandem mass spectrometry.

1. Background

Coconut (*Cocos nucifera* L.) plants are monocotyledonous members of the palm family (Palmaceae) and can be found in both tropical and subtropical regions [1]. The external coconut structure is composed of the testa, pericarp, coconut kernel (solid endosperm), coconut water (liquid endosperm), and embryo. The fiber of the coconut shell is known as the mesocarp, and it sits inside the much smoother exocarp. Meanwhile, the harder inner shell itself, or endocarp, serves to contain the seed. The coconut fiber industry generates coconut noir as a byproduct. A layer of fibrous threads exists between the outer husk and coconut shell. The outer husk and coconut find applications in textiles and other coconut-based products, whereas coir is typically underutilized. The coconut is the most widely cultivated plant in Thailand. Large quantities of coconut coir are used for various purposes. It is frequently mixed with soil to produce plant pots and beds, and several studies have been conducted on the use of coir in concrete, reinforced polymers, or adsorption [2–4]. However, coconut coir contains phenolic compounds, flavonoids, and tannins, such as gallic acid, vanillic acid, catechin, and epicatechin [1]. Tannin are widely used as an antioxidants [1,5–7], antibacterial agents [8], adhesives [9], and also for inhibiting

* Corresponding author.
E-mail address: nattacha.p@en.rmutt.ac.th (N. Phetyim).

https://doi.org/10.1016/j.heliyon.2023.e13377
Received 24 July 2022; Received in revised form 19 January 2023; Accepted 29 January 2023
Available online 3 February 2023

corrosion in steel [10].

Tannins are compounds in the phenolics group which have a relatively high molecular weight, such that they rank third in importance among all phenolic compounds. Tannins can be further classified as either condensed or hydrolysable types. One group of condensed tannins is known as the proanthocyanidins, which are polymeric flavonoids. While there is already a good understanding of flavonoid synthesis in terms of the biosynthetic pathways involved, the stages which lead to the processes of condensation and polymerization lack detailed explanation to date. The condensed tannins which have been most widely studied are those which have their basis in flavan-3-ols (−)-epicatechin and (+)-catechin. Hydrolyzable tannins are derived from gallic acid (3,4,5 trihydroxyl benzoic acid), which undergoes esterification to produce a core polyol, while the galloyl groups can undergo additional esterification to obtain hydrolysable tannins of greater complexity; similar outcomes can be achieved by oxidative crosslinking [11]. Many different components of plants have been shown to contain tannins including, but not limited to, the leaves, bark, and seeds.

Tannins are typically known to dissolve in water, but in the case of tannins which have a high molecular weight, the solubility is not high in any type of solvent [12]. It is relatively simple to extract tannins through the use of one or more polar solvents: these include ethanol, methanol, acetone, or simply water [13–16]. A number of approaches have been examined in attempts to maximize the yield of tannins. For instance, the extraction of tannins from *Acacia mearnsii* has been performed using supercritical fluids at a temperature of 60 °C and pressure of 200 bar, yielding 25% tannins from acacia bark [17]. A microwave treatment was used to extract tannins from the bark. After 1 min of extraction, the maximum polyphenol concentration was 481.4 mgGAE/g bark [15]. The ultrasonic-solvent extraction of tea leaves resulted in a 23.1% tannin yield after 8 h, which was 1% greater than that obtained using the conventional method [18]. Furthermore, tannin was extracted from mangrove bark using a solvent with the addition of chemicals such as Na_2SO_3 and Na_2CO_3, which increased the tannin yield [19]. The yields were 22.45 and 26.14% without and with the addition of Na_2SO_3 and Na_2CO_3, respectively [19]. Tannin was extracted from green coconut mesocarp with water and Na_2SO_3 at 80 °C for 2 h. Treatment with 5% Na_2SO_3 resulted in a 3.37% tannin yield, whereas the yield was 1.53% when only water was used for extraction [9]. Some researcher compared different methods, such as Soxhlet (boiling point), supercritical fluid (60 °C, 200 bar), and pressurized water extraction (100 °C, 100 bar). The extraction yields using water as the solvent were 26.2, 17.8, and 27.0% g/g sample, respectively. The gallic acid contents obtained from the three methods were 1.15, 0.48, and 0.65% g/g extract, respectively [13]. When using pistachio hull, solvent extraction resulted in a higher phenolic content than ultrasonic extraction [20]. Although several techniques increase the yield, some techniques decrease it. Therefore, in this study, we aimed to extract tannins through solvent extraction, which is simple and inexpensive.

Many variables affect tannin yield. The different key parameters which govern solvent extraction, including the type of solvent involved, the ratio of solid to solvent, and the temperature for the procedure have all been examined [5,10,13,14,19,21]. According to a previous study, the temperature affects the yield. Tannins were extracted from *Quercus infectoria* galls using distilled water. The yield of tannin increased with temperature; however, the yield decreased at the boiling point. The antioxidant activity increased with increasing concentrations of tannins [5]. Tannin was extracted from acorn using enzymes and ultrasonication at 25, 35, 45, 55, and 65 °C, with a temperature of 45 °C providing the greatest yield [10]. Coconut mesocarp and endocarp extraction was investigated through ultrasonication. The solvent used was 60% acetone. The key study variables included time, ranging from 20 to 100 min, temperature, in the range of 20–80 °C, the ratio of solid to liquid, ranging from 1:5 to 1:25 g/mL, and ultrasonic power, in the range of 180–360 W. The total flavonoid content initially increased and subsequently decreased [22]. A sulfur dioxide solution was used to extract anthocyanidins and phenolics from blackcurrants at temperatures ranging from 6 to 74 °C. The extraction rate was shown to be increased as the temperature increased, while equilibrium could be achieved more rapidly. It can therefore be concluded that tannin extraction is directly influenced by temperature [23].

The extraction process is directly affected by the ratio of solid to solvent since this in turn causes an increase in the concentration gradient linking solid and solvent, so that the rate of diffusion also rises [23–25]. In addition, the antioxidant capabilities are also influenced [25,26], although there is a decline in the overall flavonoid content level which occurs as the amount of solvent rises [22]. A number of different polar solvents including, but not limited to, methanol, ethanol, acetone, and water, individually or in combination, are sometimes employed to extract tannins. The extraction yields were 18.5, 20.8, 22.5, 26.4, 27.1, and 26.2% g/g sample when using 70% acetone, 70% ethanol, 50% ethanol, 30% ethanol, 20% ethanol, and water, respectively [13]. Three different solvents (methanol, ethyl acetate, and water) were tested for extraction with water demonstrating the greatest phenolic content at 34.7 mg of tannic acid equivalent/g dry weight of sample [20]. For propolis yields, the values reported were 1.81% (water), 3.71% (25% ethanol), 42.14% (50% ethanol), 47.60% (75% ethanol), 49.36% (95% ethanol), and 51.3% (100% ethanol) [27]. The type of solvent affects the yield; however, water and ethanol are preferred because they are environmentally friendly solvents, and the products extracted from these two solvents are suitable for human use. Therefore, three factors, namely, temperature, solid-to-solvent ratio, and type of solvent (water or ethanol), were studied.

In conventional experimentation, all variables were held constant and modified one by one, following a technique known as OVAT (one variable at a time). This approach does, however, have the drawback that it cannot be applied to assess the effects of the various factors acting in combination, since it considers only the response to changes in individual variables [28]. Owing to the expensive and time-consuming nature of OVAT techniques for optimizing diesel engine operation [29], RSM (response surface methodology) was used for the optimization process, this method allows complex procedures to be modeled [30], and is especially effective when the results of an experiment are likely to be affected by a number of different variables. Many previous studies have employed RSM as a means of optimizing the production of biodiesel through determining exactly how each parameter affects the process as a whole [31–33]. One widely used statistical software product capable of determining optimal points is Design-Expert (Stat-Ease, Inc.). This software allows screening and characterization to be performed along with comparative analyses and optimization processes. It is very useful in parameter design and combined designs, allowing the screening of matrices encompassing up to 50 different variables [34].

Table 1
The factors and coded levels used for RSM (response surface methodology).

Variables	Symbols	Coded levels				
		−α	−1	0	1	+α
Temperature (°C)	A	22	30	50	70	78
Ratio of solid to solvent (g:mL)	B	16	20	30	40	44
Type of solvent	ethanol water					

ANOVA (analysis of variance) was carried out to assess which of the variables could be considered statistically significant, while further tools in the software package permitting the assessment of data anomalies which might offer insights into the particular effects of each of the variables in terms of the eventual results [35].

This study sought to examine the influence of the different extraction parameters in terms of the tannin content extracted from coconut coir; RSM was used to determine the optimum conditions. Three response parameters, namely, temperature, solid-to-solvent ratio, and type of solvent (water and ethanol) were chosen for the study. Furthermore, the composition of the ethanol and water extracts was analyzed using FT-IR spectroscopy along with liquid chromatography with tandem mass spectrometry (LC-MS/MS). Where different solvents are used, the process of tannin extraction should also be different. Hence, in the present study, the RSM method was used for predicting and optimizing solvent extraction parameters influencing the tannin content of coconut coir. Thus far, coconut coir has not been extensively investigated, while earlier works have focused predominantly upon flavonoids and phenolic acids.

2. Materials and methods

2.1. Preparation of the materials

In the initial stage, the coconut coir was first washed and then oven-dried for 24 h at a temperature of 60 °C. Before use it was then stored in a desiccator. The tannic acid was supplied by Himedia, while the sodium carbonate and Folin-Ciocalteu reagent were obtained from Sigma-Aldrich. Qrec. Chemical Co. Ltd. supplied the ethanol (95%). Analytical grade reagents were used in all cases.

2.2. Tannin extraction

Firstly, 1 g of dry coconut coir was added to a conical flask before mixing with ethanol (95%). This mixture was then placed in a water bath and shaken for a period of 2 h. The temperature of the water bath was controlled at the extraction temperature, and the temperature inside the conical flask was lower than that of the water bath by approximately 2 °C. Subsequently, the mixture was subjected to vacuum filtration through filter paper (No. 1). A rotary evaporator was then employed to carry out the evaporation of the extract at pressures of 175 mbar using ethanol, and 72 mbar using water, while the temperature was 40 °C in all cases. Amber glass bottles were used to store the samples at a temperature of at 4 °C prior to performing the UV–visible spectrophotometric analysis.

2.3. UV–visible spectrophotometric analysis

The extracted samples were analyzed for tannins via the Folin-Ciocalteu method, which was modified from that used in a previous study [27]. Folin-Ciocalteu assays are among the general assays employed for condensed tannins and hydrolyzable tannins. These assays can be considered non-specific, and are capable of detecting the various sample compounds which can be oxidized, as well as all the phenolics [36]. To prepare the samples, crude extract (2.5 mL) was added to 7% Folin reagent (2.5 mL) and 7% sodium carbonate (2 mL), whereupon the mixture was produced via vortex mixer. The samples were then held in darkness for 1 h, before the absorbance at 760 nm was measured with a UV–visible spectrometer (UV-5100, VIS-5100). Eq. (1) can be used to calculate the tannin content in the form of mg tannic acid equivalents per g of dried sample.

$$Tannin\ content\left(\frac{mg}{g}\right)=\frac{concentration\left(\frac{mg}{L}\right)\times volumeof\ solvent(L)}{gram\ of\ dried\ coconut\ coir} \tag{1}$$

2.4. Design of experiment

A central composite design (CCD) comprising five levels and three factors was employed for the purposes of this research, necessitating 26 experiments. Three variables of the experiment, temperature (A), solid-to-solvent ratio (B), and type of solvent (C), were studied for their effects on tannin content (Y). Two types of solvents, water and ethanol, were selected for extraction. RSM was used to maximize the tannin content of the crude extract. Experimental design and results analysis was performed using Design-Expert 13 software (Stat-Ease Inc., Minneapolis, USA). Table 1 presents the three factors and their coded levels.

Table 2
Content of crude tannin extracted from coconut coir as determined using RSM.

Std	Run	A: temperature (°C)	B: ratio (g/mL)	C: Solvent type	Tannin content (mg/g)
9	1	50	30	water	4.55 ± 0.06
22	2	50	30	ethanol	2.88 ± 0.08
14	3	30	20	ethanol	2.88 ± 0.045
16	4	30	40	ethanol	2.37 ± 0.08
23	5	50	30	ethanol	2.815 ± 0.025
26	6	50	30	ethanol	2.93 ± 0.04
1	7	30	20	water	4.31 ± 0.37
5	8	22	30	water	3.95 ± 0.58
24	9	50	30	ethanol	2.825 ± 0.045
6	10	78	30	water	9.495 ± 0.525
13	11	50	30	water	4.45 ± 0.07
11	12	50	30	water	4.545 ± 0.045
10	13	50	30	water	4.475 ± 0.025
17	14	70	40	ethanol	4.975 ± 0.155
2	15	70	20	water	8.365 ± 0.475
20	16	50	16	ethanol	3.315 ± 0.535
12	17	50	30	water	4.465 ± 0.055
19	18	78	30	ethanol	6.825 ± 0.425
15	19	70	20	ethanol	4.88 ± 0.66
4	20	70	40	water	8.69 ± 0.32
21	21	50	44	ethanol	2.86 ± 0.07
7	22	50	16	water	4.495 ± 0.285
8	23	50	44	water	5.35 ± 0.54
3	24	30	40	water	3.21 ± 0.27
18	25	22	30	ethanol	2.495 ± 0.205
25	26	50	30	ethanol	2.885 ± 0.095

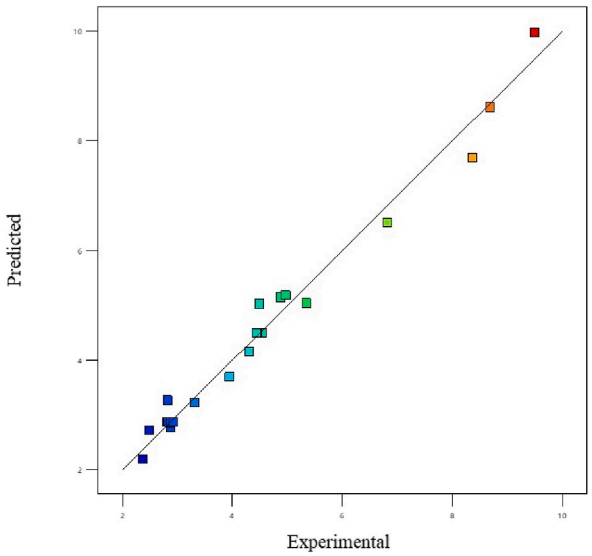


Fig. 1. Predicted and experimental tannin contents.

2.5. FT-IR analysis

An FT-IR spectrometer (Nicolet iS50, USA) was used to analyze the samples to determine the characteristic functional groups. These samples then underwent scanning at a resolution of 4 cm^{-1} at wavelengths from 4000 to 400 cm^{-1}. A deuterated triglycine sulfate–attenuated total reflectance (DTGS ATR) detector was used.

2.6. LC-MS/MS analysis

LC-MS/MS was used to carry out the crude extract sample analysis, employing a high-performance liquid chromatograph (LC-20ADXR, Shimadzu Corp., Japan) in combination with a mass spectrometer (LCMS-IT-TOF, Shimadzu Corp., Japan). An ODS-3 column (4.6 mm × 150 mm, 5 μm) was used at room temperature for sample separation. There were two mobile phases; the first comprised 1%

Table 3
Variance analysis of regression equations.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	93.87	11	8.53	71.36	<0.0001	significant
A-temperature	50.73	1	50.73	424.21	<0.0001	
B-ratio	0.0637	1	0.0637	0.5324	0.4776	
C-solvent type	6.64	1	6.64	55.54	<0.0001	
AB	0.7290	1	0.7290	6.10	0.0270	
AC	3.09	1	3.09	25.85	0.0002	
BC	0.0767	1	0.0767	0.6412	0.4367	
A^2	14.49	1	14.49	121.17	<0.0001	
B^2	0.4380	1	0.4380	3.66	0.0763	
Residual	0.1815	1	0.1815	1.52	0.2383	
Lack of Fit	0.3066	1	0.3066	2.56	0.1317	significant
Pure Error	0.1166	1	0.1166	0.9754	0.3401	
Cor Total	1.67	14	0.1196			

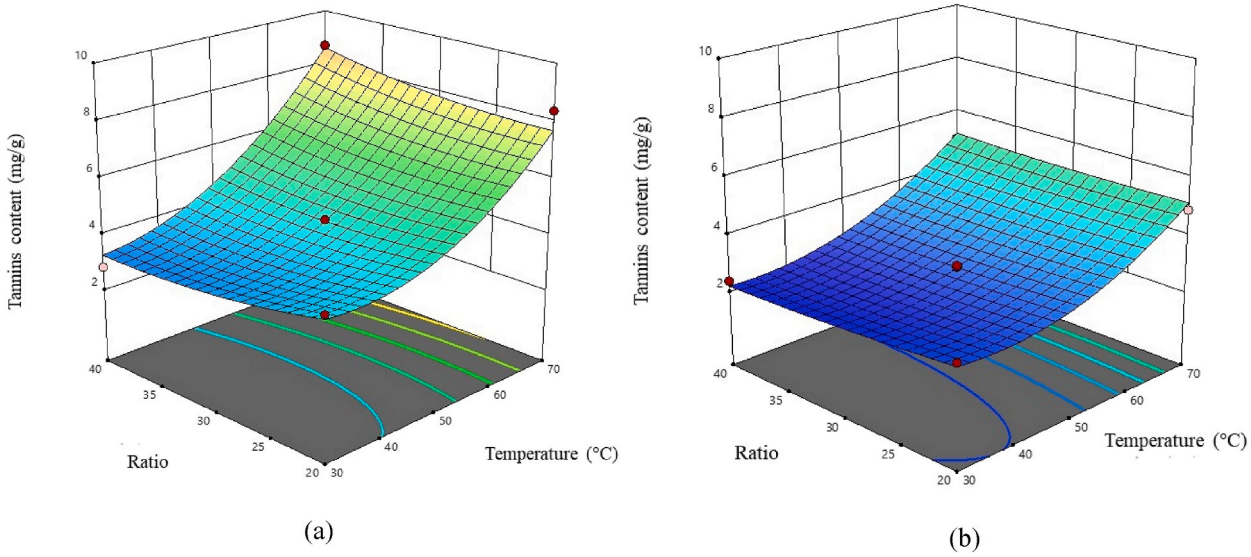


Fig. 2. The influence of different temperatures and ratios of solid to solvent upon tannin content levels when using: (a) water (b) ethanol.

acetic acid in water (A) while the second was methanol (B). Elution was then programmed in the format of t (min), B%: (0, 22), (25, 36), (55, 52), (90, 63), (115, 70), (135, 75), (150, 80), (155, 80), (160, 22), and (170, 22). During this process, the flow rate of the solvent remained constant at 0.75 mL/min, while the volume injected was 10 μL.

LC-MC/MS was used in combination with a source of electrospray ionization, with the positive mode used for the ion spray. The gas used for nebulization and the removal of the solvent was nitrogen, flowing at 1.5 L/min. Scanning of the mass range was performed from *m/z* 50 to 2000.

3. Results and discussion

3.1. Use of RSM to optimize tannin content

The CCD was used to design 26 experimental runs, whereupon the process performed five replicates in order to obtain an estimate for the pure error sum of squares. Table 2 indicates the tannin content levels recorded for each of the experiments. The tannin content of dried coconut coir ranges from 2.37 ± 0.08 to 9.495 ± 0.525 mg/g.

The fitted model was subjected to variance analysis. On the basis of the date presented in Table 2, the following values are obtained for the model: $F = 71.36$, $P < 0.0001$, $R^2 = 0.9825$, and adj. $R^2 = 0.9687$, while the 'lack-of-fit' term can be considered significant at $p < 0.0001$. Fig. 1 shows the experimental values along with the predictions, with the resulting data suggesting that the model is capable of making appropriate response predictions. ANOVA was used to establish the ideal parameters for the process. Table 3 presents the findings from the regression model, demonstrating that the factors of the terms A, C, AB, AC, and A^2 have p-values <0.05. The software was then used to process the experimental data, while the relationship between the dependent and independent variables was determined through the quaternion quadratic polynomial equation. This was derived from Eq. (2) (for water) and Eq. (3) (for ethanol)

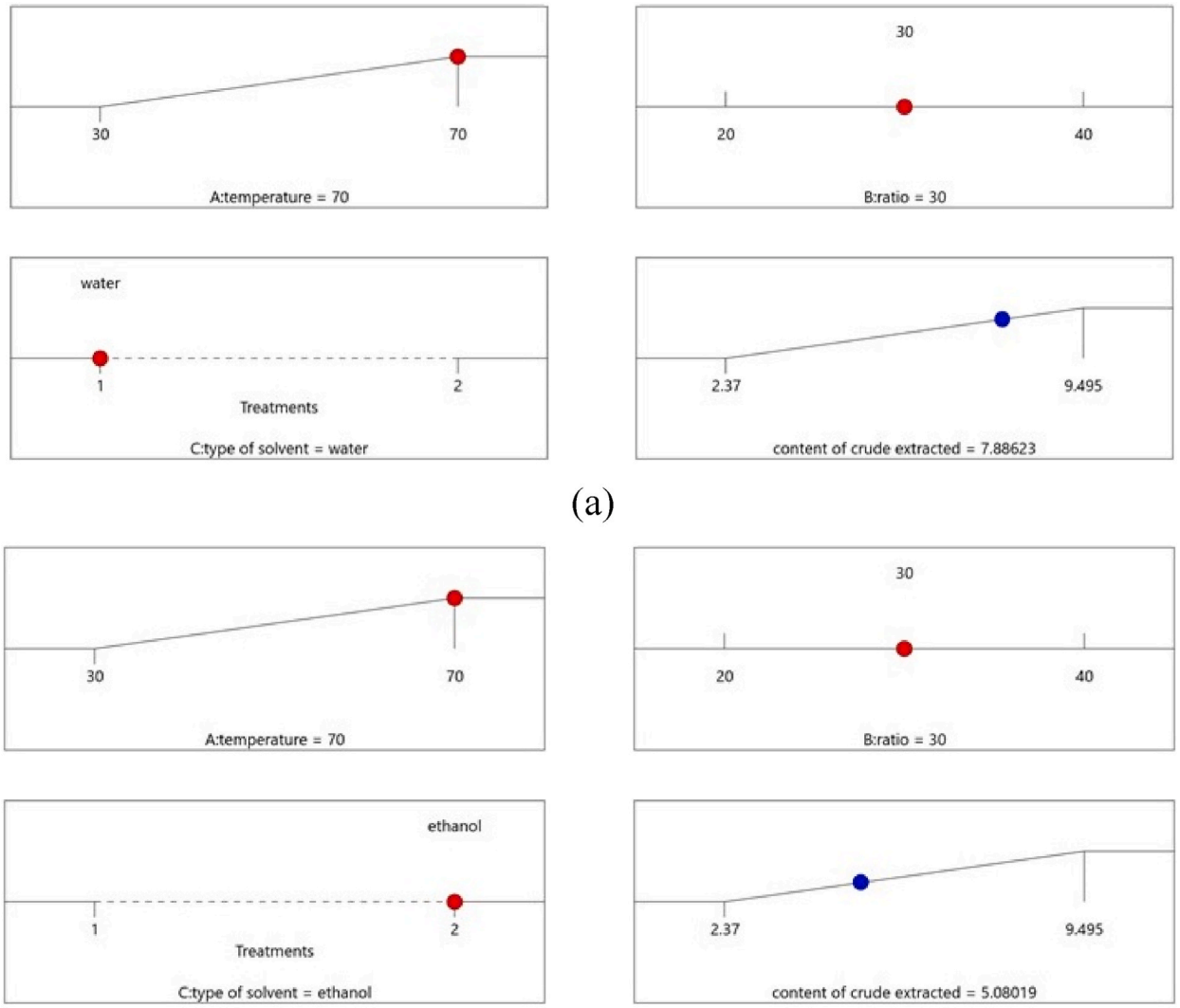


Fig. 3. Optimal conditions for coconut coir extraction: (a) water; (b) ethanol.

as shown below.

$$Tannin\ content\left(\frac{mg}{g}\right) = 12.85099 - 0.391580A - 0.136051B + 0.003196AB + 0.005215A^2 - 0.001689B^2 - 0.000069A^2B + 0.000092AB^2 \quad (2)$$

$$Tannin\ content\left(\frac{mg}{g}\right) = 8.66435 - 0.266058A - 0.055012B + 0.001308AB + 0.004100A^2 - 0.002251B^2 - 0.000069A^2B + 0.000092AB^2 \quad (3)$$

When water or ethanol is used as the solvent, a response surface plot, which can be observed in Fig. 2 (a) and (b), shows how the temperature and ratio will interact, thus determining the levels of tannin content. It is clear from the response surface plot that temperature is the factor which is most influential in terms of the effect upon tannin content. High temperatures result in higher tannin contents than those at low temperatures. The extraction conditions, particularly the temperature, can considerably affect the extractability of tannins. However, most tannins are water-soluble [37]. At the highest temperature of the water bath (78 °C), the tannin contents are 9.495 ± 0.525 and 6.825 ± 0.425 mg/g dry coconut coir when water and ethanol are used as solvents,

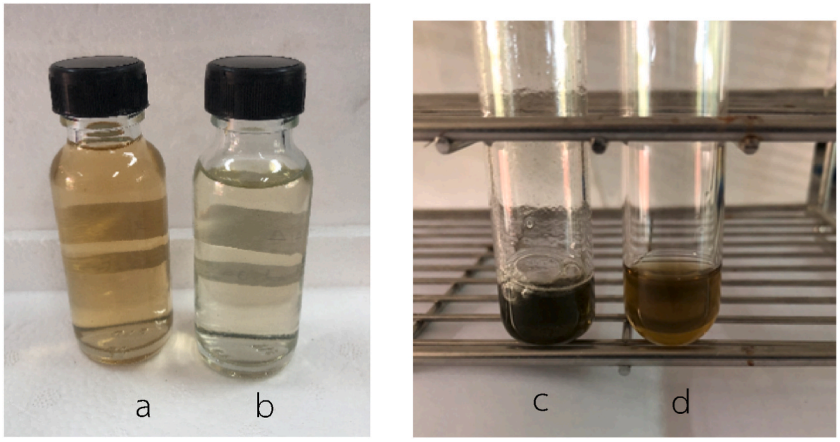


Fig. 4. Crude tannins extracted from coconut coir when using (a) water (b) and ethanol as the solvent; 1% $FeCl_3$ added to the (c) water-extracted and (d) ethanol-extracted samples.

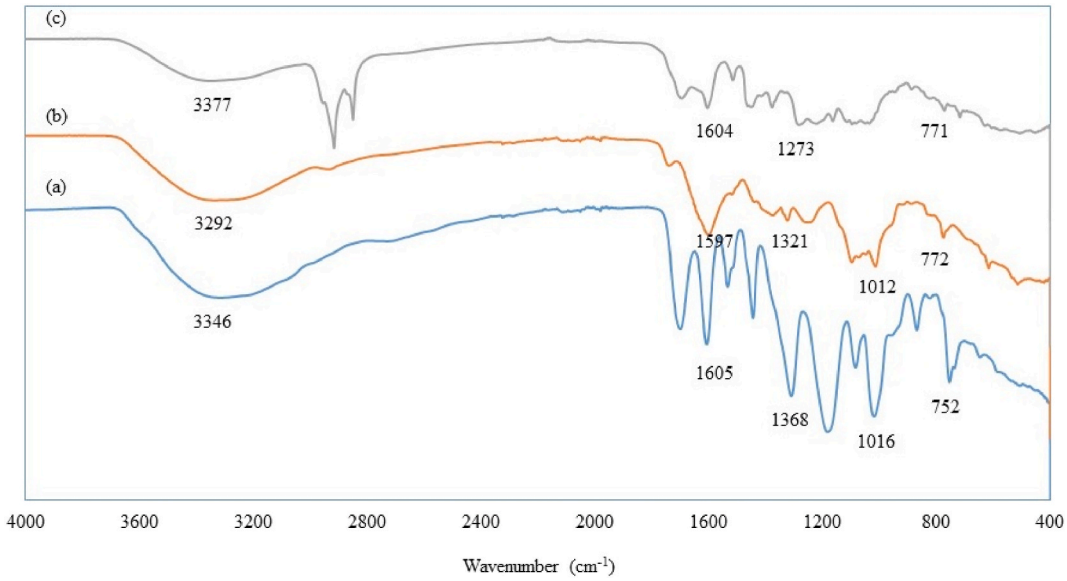


Fig. 5. Fourier-transform infrared spectra for (a) tannic acid, (b) water-extracted and (c) ethanol-extracted samples.

respectively. Increasing the extraction temperature increases the diffusion coefficient [24]. Some studies have shown that higher temperatures can break the cell walls and release polyphenols, resulting in a high phenolic acid yield [38]. Based on tannin extraction studies, when several solvents were examined, water extracted the crude more easily than ethanol under the same conditions [39]. The extract yield increases with solvent polarity [13]. The solvent polarity is an important parameter affecting the process of extraction, and therefore it could be concluded that in the case of polyphenolic compound extraction from pistachio, it would be appropriate to use water [20]. Water resulted in a higher extraction yield than that obtained using ethanol; moreover, water also permitted the extraction of greater levels of gallic acid content, as well as corilagin and ellagic acid than would be the case when ethanol was used [13].

Although there is no influence upon tannin content exerted by the ratio of solid to solvent, the term B is not significant to the response; we consider that the amount of solvent used in this experiment was adequate for extraction. However, an extremely low ratio cannot be used because the dried coconut coir completely absorbs the solvent.

The optimized conditions for maximizing the tannin content using water are determined and are shown in Fig. 3 (a); the maximum tannin content according to the software prediction was 7.88623 mg/g under optimal conditions of a temperature of 70 °C (A) and a ratio of solid to solvent of 1:30 (B). In the case of ethanol, the maximum tannin content predicted by the software was 5.08019 mg/g (Fig. 3 (b)); at the same optimum parameter of water, the desirability values for water and ethanol were 0.880 and 0.617, respectively. The equation predictions were validated by further experimental procedures which were carried out under the same specific conditions. In this case, the levels of tannin content were assessed to be 8.117 and 4.517 mg/g when using water and ethanol, respectively. The errors between the software predictions and experimental results were 2.93 and 11.08% for water and ethanol, respectively.

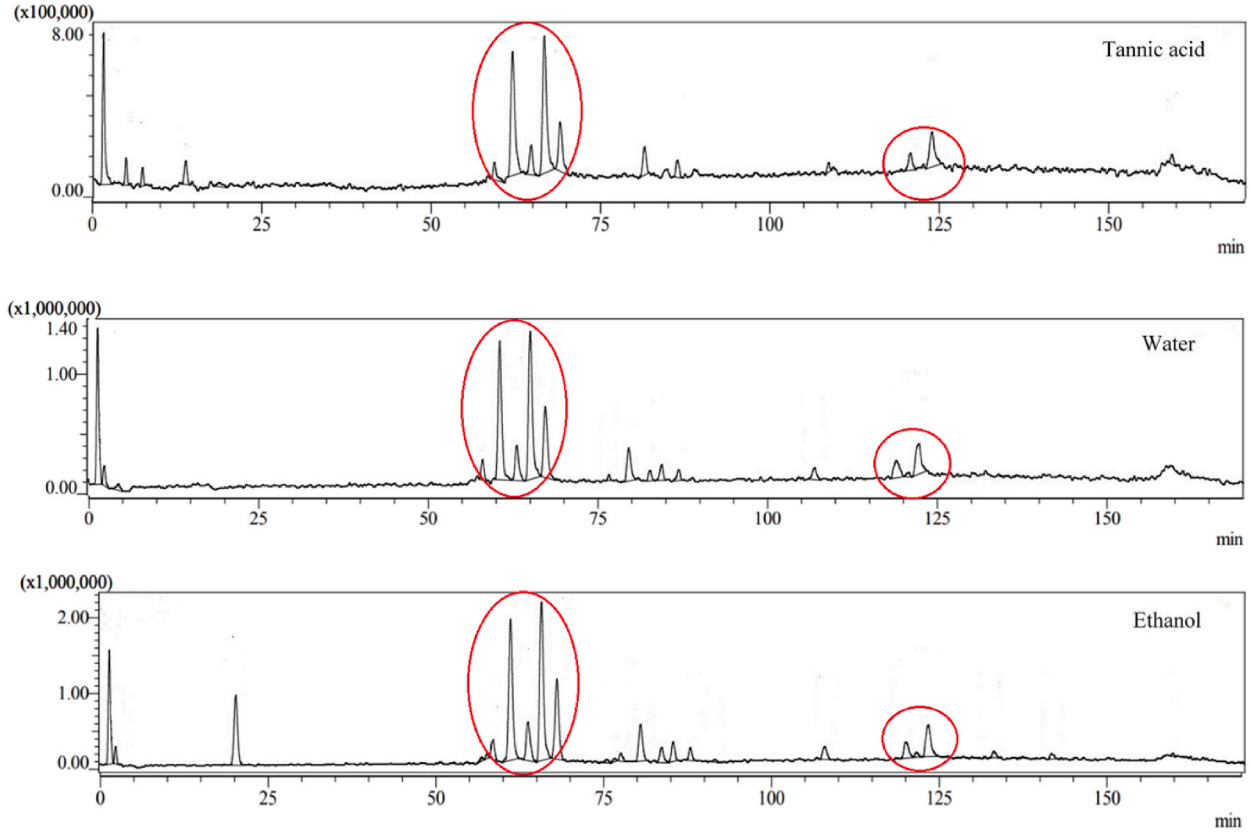


Fig. 6. The profiles obtained from liquid chromatography with tandem mass spectrometry for the crude extracts using water and ethanol, in comparison to a sample of tannic acid.

3.2. Testing of tannins

The products obtained after extraction are shown in Fig. 4. The coconut coir extract is brownish in water and green in ethanol. The extracted samples were analyzed for the presence of tannins in 1% $FeCl_3$. When 1% $FeCl_3$ was added to the extracts obtained using water and ethanol, there was a change observed in the extract color as can be seen in Fig. 4 (c) and (d), whereby the original brown was altered to a black-green color. These findings demonstrate the presence of condensed tannins in the sample extracts. These results are similar to those of previous studies [15,39]. Water can extract procyanidins and flavanols [40], and ethanol can extract anthocyanins, flavanols, and free phenolic acids [41]. Water can extract condensed tannin from the bark owing to the presence of hydroxyl groups [39].

3.3. Characteristics of tannins

3.3.1. Functional group analysis

The use of FT-IR allowed analysis of the tannin functional groups following extraction from the coconut coir. The spectrum of tannic acid or tannins (Fig. 5(a)) is compared with those obtained for the crude extract with water (Fig. 5. (b)) and ethanol (Fig. 5. (c)) as the solvents [42]. All spectra in Fig. 5 appeared within the relatively wide range of 3700 to 3000 cm^{-1} associated with the OH stretching band which shows that polymerization has taken place. Condensed tannins exhibit various degrees of polymerization [6]. Further bands which indicate the stretching vibration of the OH groups of the phenolic structures are identified at 3,346, 3,392, and 3377 cm^{-1}, respectively.

From 1620 to 1400 cm^{-1} is the spectrum region associated with vibrations of the C═C bonds of the aromatic rings. Within the spectrum, condensed tannins may be found in the shape band between 1620 and 1604 cm^{-1} [43]. This result is observed in the spectra of tannic acid and crude extracted using ethanol at 1605 and 1604 cm^{-1}, respectively.

The stretching vibration of C–O can be observed in the region from 1350 to 1100 cm^{-1}, while the band frequency associated with aromatic ring substitution is found to be approximately 900–750 cm^{-1} [44]. These bands are observed for the three samples.

3.3.2. Comparison of tannin spectra

Tannins were extracted using water and ethanol as solvents following the process of evaporation in a vacuum at a temperature of

40 °C until a completely dry state was achieved. The resulting residue was subsequently dissolved using a deionized water filter (0.22 μm filter) prior to injection into the LC–MS/MS device. The MS spectra of water and ethanol are similar to those of tannic acid, with peak group retention times between 50 and 75 min, and also approaching 125 min (Fig. 6).

4. Conclusion

In this experiment, three factors for solvent extraction of coconut coir were investigated using RSM: temperature, ratio of solid to solvent, and the type of solvent involved. It was determined that the tannin content level would be governed by both temperature and solvent type. For the model that considered water, the maximum tannin content was predicted to arise when the temperature was set to 70 °C and the ratio of solid to solvent was 1:30; thus, water is regarded as the optimal solvent for extraction. The FT-IR spectra of water and ethanol revealed condensed tannins in a broad range of 3700–3000 cm^{-1}. In terms of LC–MS/MS, the MS spectra of water, ethanol, and tannic acid were comparable.

Declarations

Author contribution statement

Rinlada Sirisangsawang: Performed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.
Natacha Phetyim: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper

Acknowledgment

The researchers wish to extend sincere thanks to the Rajamangala University of Technology Thanyaburi for their assistance in providing the necessary equipment required to successfully carry out this research study.

References

[1] M. Arivalagan, T.K. Roy, A.M. Yasmeen, K.C. Pavithra, P.N. Jwala, K.S. Shivasankara, S.R. Kanade, Extraction of phenolic compounds with antioxidant potential from coconut (Cocos nucifera L.) testa and identification of phenolic acids and flavonoids using UPLC coupled with TQD-MS/MS, LWT (Lebensm.-Wiss. & Technol.) 92 (2018) 116–126.
[2] A.G. Adeniyi, D.V. Onifade, J.O. Ighalo, A.S. Adeoye, A review of coir fiber reinforced polymer composites, Compos. B Eng. 176 (2019), 107305.
[3] B. Ali, A. Hawreen, N.B. Kahla, M.T. Amir, M. Azab, A. Raza, A critical review on the utilization of coir (coconut fiber) in cementitious materials, Construct. Build. Mater. 351 (2022), 128957.
[4] Y.C. Sharma, S.N. Upadhyay, Removal of a cationic dye from wastewaters by adsorption on activated carbon developed from coconut coir, Energy Fuels 23 (6) (2009) 2983–2988.
[5] M.Z.I. Arina, Y. Harisun, Effect of extraction temperature on tannin content and antioxidant activity of *Quercus infectoria* (Manjakani), Biocatal. Agric. Biotechnol. 19 (2019), 101104.
[6] M.J. Kasim, M.H. Hussin, A. Achmad, N.H. Dahon, T.K. Suan, H.S. Hamdan, Determination of total phenol, condensed tannin and flavonoid contents and antioxidant activity of Uncaria gambir extracts, Majalah Farmasi Indonesia 22 (1) (2011) 50–59.
[7] J. Hayat, M. Akodad, A. Moumen, M. Baghour, A. Skalli, S. Ezrari, S. Belmelha, Phytochemical screening, polyphenols, flavonoids and tannin content, antioxidant activities and FTIR characterization of *Marrubium vulgare* L. from 2 different localities of Northeast of Morocco, Heliyon 6 (2020), e05609.
[8] G. Maisetta, G. Batoni, P. Caboni, S. Esin, A.C. Rinaldi, P. Zucca, Tannin profile, antioxidant properties, and antimicrobial activity of extracts from two Mediterranean species of parasitic plant Cytinus, BMC Compl. Alternative Med. 19 (1) (2019) 1–11.
[9] F.L. Morbeck, R.C.C. Lelis, M.V.E. Schueler, W.A. Santos, D.A. Sampaio, B.C.D. Silva, R.D.M. Morais, G.M. Santana, Extraction and Evaluation of Tannin from Green Coconut Mesocarp, Matéria, Rio de Janeiro), 2019, p. 24.
[10] X.H. Luo, R.L. Bai, D.S. Zhen, Z.B. Yang, D.N. Huang, H.L. Mao, X.F. Li, H.T. Zou, Y. Xiang, K.L. Liu, Z.G. Wen, C. Fu, Response surface optimization of the enzyme-based ultrasound-assisted extraction of acorn tannins and their corrosion inhibition properties, Ind. Crop. Prod. 129 (2019) 405–413.
[11] K. Khanbabaee, T. van Ree, Tannins: classification and definition, Nat. Prod. Rep. 18 (6) (2001) 641–649.
[12] A.E. Hagerman, Y. Zhao, S. Johnson, Methods for Determination of Condensed and Hydrolyzable Tannins, 1997.
[13] M. Markom, M. Hasan, W.R.W. Daud, H. Singh, J.M. Jahim, Extraction of hydrolysable tannins from *Phyllanthus niruri Linn.*: effects of solvents and extraction methods, Separ. Purif. Technol. 52 (2007) 487–496.
[14] T. Wu, W. Hongling, J. Shiwen, L. Dandan, W. Fei, Optimization of extraction of tannins from banana peel using response surface methodology, Appl. Mech. Mater. 678 (2014) 566–571.
[15] N. Rhazi, H. Hannache, M. Oumam, A. Sesbou, B. Charrier, A. Pizzi, F. Charrier-El Bouhtoury, Green extraction process of tannins obtained from Moroccan *Acacia nollissima* barks by microwave: modeling and optimization of the process using the response surface methodology RSM, Arab. J. Chem. 12 (2019) 2668–2684.
[16] F. Abilleira, P. Varela, A. Cancela, X. Alvarez, A. Sanchez, E. Valero, Tannins extraction from *Pinus pinaster* and *Acacia dealbata* bark with applications in the industry, Ind. Crop. Prod. 164 (2021), 113394.
[17] M.R. Pansera, G.A. Iob, , A.C. A-S, M. Rossato, L. Atti-S, E. Cassel, Extraction of Tannin by *Acacia mearnsii* with supercritical fluids, Braz. Arch. Biol. Technol. 47 (6) (2004) 995–998.
[18] H. Rahman, F.A. Arini, V. Utomo, Tannin extraction of tea leaves by ultrasonic method: comparison with the conventional method, 85, Jurnal Teknologi 8 (1) (2020) 94.
[19] T. Neimsuwan, P. Hengniran, P. Siramon, V. Punsuvon, Tannin extraction of Rhizophora bark from residual charcoal production, J. Trop. For. Res. 1 (1) (2017) 36–50.
[20] A.H. Goli, M. Barzegar, M.A. Sahari, Antioxidant activity and total phenolic compounds of pistachio (Pistachia vera) hull extracts, Food Chem. 92 (3) (2005) 521–525.
[21] A.K. Das, M.Dn Islam, M.O. Faruk, D. Ashaduzzaman, R. Dungani, Review on tannins: extraction processes, applications and possibilities, South Afr. J. Bot. 135 (2020) 58–70.
[22] J. Yang, N. Li, C. Wang, T. Chang, H. Jiang, Ultrasound-homogenization-assisted extraction of polyphenols from coconut mesocarp: optimization study, Ultrason. Sonochem. 78 (2021), 105739.
[23] J.E. Cacace, G. Mazza, Extraction of anthocyanins and other phenolics from black currants with sulfured water, J. Agric. Food Chem. 50 (21) (2002) 5939–5946.

[24] M.A. Al-Farsi, C.Y. Lee, Optimization of phenolics and dietary fibre extraction from date seeds, Food Chem. 108 (3) (2008) 977–985.
[25] N.C. Predescu, C. Papuc, V. Nicorescu, , Gajaila, I. U. L. I. A. N. A., G.V. Goran, C.D. Petcu, Stefan, G. E. O. R. G. E. T. A., The influence of solid-to-solvent ratio and extraction method on total phenolic content, flavonoid content and antioxidant properties of some ethanolic plant extracts, Rev. Chim. (Bucharest) 67 (2016) 1922–1927.
[26] P.W. Tan, C.P. Tan, C.W. Ho, Antioxidant properties: effects of solid-to-solvent ratio on antioxidant compounds and capacities of Pegaga (Centella asiatica), Int. Food Res. J. 18 (2) (2011).
[27] C. Sun, Z. Wu, Z. Wang, H. Zhang, Effect of ethanol/water solvents on phenolic profiles and antioxidant properties of Beijing propolis extracts, Evid. base Compl. Alternative Med. 2015 (2015).
[28] T.R. Kukreja, D. Kumar, K. Prasad, R.C. Chauhan, S. Choe, P.P. Kundu, Optimisation of physical and mechanical properties of rubber compounds by Response Surface Methodology—Two component modelling using vegetable oil and carbon black, Eur. Polym. J. 38 (7) (2002) 1417–1422.
[29] Y. Singh, A. Sharma, G.K. Singh, A. Singla, N.K. Singh, Optimization of performance and emission parameters of direct injection diesel engine fuelled with pongamia methyl esters-response surface methodology approach, Ind. Crop. Prod. 126 (2018) 218–226.
[30] M. Mäkelä, Experimental design and response surface methodology in energy applications: a tutorial review, Energy Convers. Manag. 151 (2017) 630–640.
[31] S. Sukpancharoen, P. Hansirisawat, T.R. Srinophakun, Implementation of response surface to optimum biodiesel power plant derived from empty fruit bunch, J. Energy Resour. Technol. 144 (1) (2022), 012101.
[32] S. Sukpancharoen, T.R. Srinophakun, P. Aungkulanon, Grey wolf optimizer (GWO) with multi-objective optimization for biodiesel production from waste cooking oil using central composite design (CCD), Int. J. Mech. Eng. Rob. Res. 9 (8) (2020) 1219–1225.
[33] N. Thonglhueng, R. Sirisangsawang, S. Sukpancharoen, N. Phetyim, Optimization of iodine number of carbon black obtained from waste tire pyrolysis plant via response surface methodology, Heliyon 8 (12) (2022), e11971.
[34] Tanco Martin, Elisabeth Viles, Laura Ilzarbe, Maria Jesus Alvarez, Dissecting DoE Software," Six Sigma Forum Magazine, May 2008.
[35] John Cornley, Design of Experiments: Useful Statistical Tool in Assay Development or Vendor Disconnect!", , Drug Discovery World, Winter, 2009/2010.
[36] M.L. Price, L.G. Butler, Rapid visual estimation and spectrophotometric determination of tannin content of sorghum grain, J. Agric. Food Chem. 25 (6) (1977) 1268–1273.
[37] A. Scalbert, Quantitative methods for the estimation of tannins in plant tissues, in: Plant Polyphenols, Springer, Boston, MA, 1992, pp. 259–280.
[38] S. Wang, F. Chen, J. Wu, Z. Wang, X. Liao, X. Hu, Optimization of pectin extraction assisted by microwave from apple pomace using response surface methodology, J. Food Eng. 78 (2) (2007) 693–700.
[39] K. Moosophin, T. Wetthaison, L. Seeratchakot, W. Kokluecha, Tannin extraction from mangosteen peel for protein precipitation in wine, KKU Res. J. 15 (5) (2010) 377–385.
[40] P.N. Diouf, T. Stevanovic, A. Cloutier, Study on chemical composition antioxidant and anti-inflammatory activities of hot water extract from *Picea mariana* bark and its proanthocyanidin-rich fractions, Food Chem. 113 (2009) 897–902.
[41] S. Wang, K. Huang, Determination of flavonoids by high performance liquid chromatography and capillary electrophoresis, J. Chromatogr. 1032 (2004) 273–279.
[42] V. Viswanath, V.V. Leo, S. Sabna Prabha, C. Prabhakumari, V.P. Potty, M.S. Jisha, Thermal properties of tannin extracted from Anacardium occidentale L. using TGA and FT-IR spectroscopy, Nat. Prod. Res. 30 (2) (2015) 223–227.
[43] A. Ricci, K.J. Olejar, G.P. Parpinello, P.A. Kilmartin, A. Versari, Application of Fourier transform infrared (FTIR) spectroscopy in the characterization of tannins, Appl. Spectrosc. Rev. 50 (5) (2015) 407–442.
[44] C.S. Robb, S.E. Geldart, J.A. Seelenbinder, P.R. Brown, Analysis of green tea constituents by HPLC-FTIR, J. Liq. Chromatogr. Relat. Technol. 25 (5) (2002) 787–801.