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Optimization of Polysaccharides Extraction from Spent Coffee Grounds (SCGs) by Pressurized Hot Water Extraction †

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Abstract: Spent coffee grounds (SCGs) are a by-product of the food industry, which contains a rich source of polysaccharides. This research was to study the extraction of polysaccharides from SCGs by environmentally friendly technic of pressurized hot water. The process optimization was investigated by response surface methodology (RSM) to produce the highest extraction yield with a different temperature of 80–120 °C, a pressure of 4–12 bars, and an extracted time of 60–180 min. The pressurized hot water showed an efficient technique to recover polysaccharides from SCG.

Keywords: spent coffee grounds; pressurized hot water; polysaccharide extraction

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1. Introduction

The consumption of coffee in Thailand has gained immense popularity and reached 85,805 tons per year in 2018. Thailand has approximately 27,000 coffee shops countrywide (Department of Agricultural Extension of Thailand, 1 October 2018). Consequently, those coffee shops will produce large quantities of spent coffee grounds (SCGs) in the future. SCGs are the main residue obtained during the brewing process of coffee, 1 kg of ground coffee can produce 910 g of the dried SCGs on average. They contain polysaccharide (45–47%), oil or lipids (9–16%), protein (13–17%), phenolic compounds (1.7–3.5%), caffeine (0.5–1.2%), and other minerals (1.6%) [1], depending on the coffee's species, roasting, grinding, and brewing process.

Pressurized hot liquid extraction (PLE) is regarded as a green and efficient technique to extract solid and semi-solid samples with liquid solvents [2,3]. This technique provided a higher efficiency to the extraction process by using conventional solvents such as water, ethanol and hexane at elevated temperatures and pressures. The PLE with liquid solvent has been used to extract bioactive compounds, especially phenolic compounds, from several plant materials [4,5]. The antioxidative polyphenolics were successfully extracted from SCGs by PLE, ethanol and water [6]. This study aims to recover the polysaccharide from SCGs residues by pressurized hot water (PHW) and to convert the food agricultural waste into value-added products. The response surface methodology (RSM) was applied to find out the optimal condition to produce the highest extraction yield.

2. Materials and Methods

2.1. Preparation of SCGs Sample

The SCGs used in this study was supplied by Starbucks, V Market branch, Ladkrabang, Bangkok, Thailand. The SCGs sample were dried at 40 °C for 24 h until constant weight was observed. They were kept in a desiccator until utilized in the experiments.

2.2. Pressurized Hot Water Extraction

The pressurized hot water extraction was performed in a 500-cm^3 batch reactor (Parr Instrument Company, USA). An approximately 20 ± 0.1000 g of dried SCGs and 300 mL of distilled water were directly loaded into the reactor. Then, the reactor was heated according to the conditions as mentioned in Table 2. The reactor was cooled to room temperature by tap water to stop the reaction. The obtained extract was filtered using Whatman paper filters No.1 and stored at 4 °C for recovery of polysaccharides.

2.3. Recovery of Polysaccharides

The polysaccharides were recovered by mixing the hydrolysate with 2-fold volume of absolute ethanol and kept overnight at 4 °C. Subsequently, the mixture was centrifuged at 5000 rpm for 10 min. The precipitate was dried in an oven at 40 °C for 24 h until constant weight was observed. The polysaccharides extract was kept in a desiccator at room temperature for further analysis. The recovery yield of polysaccharides was calculated by Equation (1).

$$\%\ \text{yield} = (\text{polysaccharides extract (g)}/\text{SCGs weight (g)}) \times 100 \quad (1)$$

2.4. Experimental Design and Data Analysis

In this study, the effect of independent variables, temperature (A), pressure (B) and extraction time (C), on polysaccharides yield was investigated by the central composite design (CCD) method and response surface method (RSM) was applied to locate the optimal condition. The highest yield of polysaccarides was the indicator. Table 1 presents ranges and center point values of independent variables. Design-Expert® software (Stat-Ease, Inc., MN, USA) was employed to design the experiment and to carried out the analysis of variance (ANOVA), regression model, and statistical analysis accordingly.

Table 1. Independent variables and their levels for experimental design of SCGs extraction by pressurized hot water extraction.

Independent Variables	Code Units	Coded Levels				
		−α	−1	0	1	+α
Temperature (°C)	A	70	80	100	120	130
Pressure (bar)	B	2	4	8	12	14
Extraction time (min)	C	30	60	120	180	210

2.5. Determination of Phenolic Compounds

The total phenolic compounds of the highest extraction yield were analyzed by the Folin-Ciocalteu spectrophotometric method [7]. The absorbance of samples was measured by a UV-visible spectrophotometer (PharmaSpec, UV-1700) at 730 nm. The results were compared to a standard curve of gallic acid equivalents to calculate the total phenolics compounds.

3. Results and Discussion

3.1. Data Analysis and Response Surface Model Building

Table 2 shows the experimental results of 30 runs base on CCD design, comprised of duplicates of eight factorial points, six axial points, and a central point. The %yield of polysaccharides was considered as the response (Y). The responses were statistically analyzed and observed to fit with the quadratic regression model as shown in equation (2).

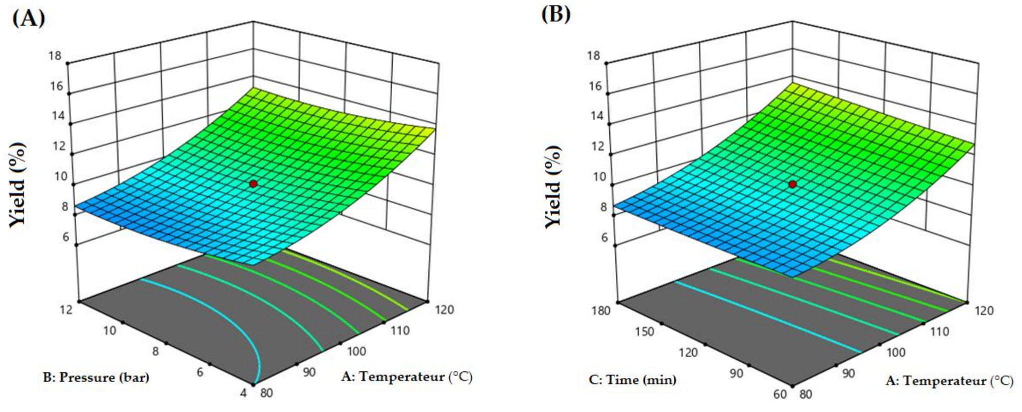
Table 2. Central composite design matrix and the response values for the extraction yield of polysaccharide.

Run	Actual Process Variables			%Yield
	Temperature (°C)	Pressure (bar)	Extraction Time (min)	
1	100	2	120	11.5100
2	100	2	120	11.5550
3	80	4	60	10.1562
4	80	4	60	9.1000
5	80	4	180	9.4061
6	80	4	180	9.4061
7	120	4	60	12.2650
8	120	4	60	15.2737
9	120	4	180	14.1212
10	120	4	180	13.2937
11	100	8	30	10.6625
12	100	8	30	9.8187
13	70	8	120	8.6712
14	70	8	120	8.2537
15	100	8	120	9.9675
16	100	8	120	10.1650
17	130	8	120	14.9100
18	130	8	120	16.1700
19	100	8	210	10.7050
20	100	8	210	9.9962
21	80	12	60	7.3437
22	80	12	60	9.6975
23	120	12	60	10.6075
24	120	12	60	13.7875
25	80	12	180	9.3637
26	80	12	180	8.5587
27	120	12	180	13.6300
28	120	12	180	14.8700
29	100	14	120	11.4375
30	100	14	120	9.5612

$$Y\ (\%) = 24.85923 - 0.312912A - 0.692658B - 0.029169C + 0.000817AB + 0.000185AC + 0.001446BC + 0.001982A^2 + 0.022171B^2 + 9.60789 \times 10^{-6}C^2 \quad (2)$$

where; A, B, and C are coded factors, whereby A is Temperature, B is pressure, and C is Extraction time

The result of ANOVA shows in Table 3. The model has significance with a p-value of <0.0001, which indicates that the model provides an accurate description of the experimental data. Concerning the operating parameter, only temperature(A) has a p-value less than 0.0001. So, it is the main factor that influences the yield of polysaccharides. As presents in Figure 1, the %yield increased with the raising of temperature while it has a bit change with the increase of pressure and extraction time.



(**a**) (**b**)

Figure 1. Response surface plot demonstrating the effect of (**a**) temperature and pressure and (**b**) temperature and extraction time on %yield of polysaccharides.

Table 3. The analysis of variance (ANOVA) table for the response surface quadratic model of %yield of polysaccharides.

Source	Sum of Squares	df	Mean Square	*p*-Value
Model	140.62	9	15.62	<0.0001
A (Temperature)	125.66	1	125.66	<0.0001
B (Pressure)	2.73	1	2.73	0.1033
C (Extraction Time)	0.9019	1	0.9019	0.3384
AB	0.0684	1	0.0684	0.7898
AC	0.7850	1	0.7850	0.3710
BC	1.93	1	1.93	0.1670
A^2	6.91	1	6.91	0.0133
B^2	1.38	1	1.38	0.2384
C^2	0.0132	1	0.0132	0.9069

3.2. Process Optimization by RSM

To maximize the responses, the optimal condition for SCGs extraction with pressurized hot water was carried out with the assistance of the optimization function embedded in the Design-Expert® software. The optimum conditions obtained were as follows: temperature of 120 °C, pressure of 4 bar, and extraction time of 60 min, which correspond to the predicted %yield 13.782. Comparing to the actual value of 13.71 ± 53%, which obtained from the experimental runs under the suggested optimal conditions in triplicate, indicates that these optimal parameters are valid for this study. In addition, the total phenolic content from obtained polysaccharide was 11.13 ± 1.33 mg gallic acid equivalent (GAE)/g dry SCG.

Table 4. Optimal conditions for SCGs extraction with pressurized hot water and the predicted and the actual values of response.

Operating Parameter	Optimal Condition	%Yield	
		Predicted Value	Actual Value
Temperature (°C)	120		
Pressure (bar)	4	13.782	13.71 ± 53
Extraction time (min)	60		

4. Conclusions

The polysaccharides from Spent coffee grounds (SCGs) have been successfully extracted by pressurized hot water in this study. The temperature was observed as the main influence on the increasing of %polysaccharides yield. The process optimization was temperature of 120 °C, pressure of 4 bar, and extraction time of 60 min, was investigated by RSM method. Under this optimal condition, the highest extraction yield was 13.71 ± 53% and the total phenolic content was 11.13 ± 1.33 mg gallic acid equivalent (GAE)/g dry SCG.

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