OPTIMIZATION OF SOLID-TO-SOLVENT RATIO AND TIME FOR OIL EXTRACTION PROCESS FROM SPENT COFFEE GROUNDS USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

In this study, the optimization of two parameters: solid-to-solvent ratio and extraction time of the oil extraction process from the dried spent coffee grounds (DSCG) after brewing coffee were investigated by using the response surface methodology (RSM). The results showed that the 14.75 wt.% of calculated yield of coffee oil from the predicted model was obtained, when the optimal condition: the 1:22.5 g/g of mass ratio of DSCG-to-hexane and 30.4 min of extraction time under the 30 °C of room temperature was used. The model was verified by the experiment, the 14.68 wt.% experimental yield of coffee oil was achieved after passing the extraction process under the optimal condition. Moreover, the composition of coffee oil after eliminate the residual hexane were analyzed by the thin layer chromatograph with flame ionization detection (TLC/FID). The 81.156 wt.% triglyceride, 5.926 wt.% diglyceride, 11.428 wt.% monoglyceride, 1.078 wt.% ester, and 0.412 wt.% free fatty acid were found.

Keywords: spent coffee grounds, coffee oil, optimization, response surface methodology.

INTRODUCTION

The coffee grounds are consumed in coffee shop, homes, bars and restaurants, large factories of instant coffee (Wikipedia Contributors 2015). Consequently, the coffee beverage is consumed worldwide. In Thailand, the coffee beans were used to produce the instant coffee, roasted and ground coffee, and canned coffee (Pongsiri 2013). Therefore, the increasing domestic consumption of coffee bean is affected on the amount of coffee grounds increased as well. Many organic residues, spent coffee grounds (SCG), after brewing coffee of both instant coffee and roasted coffee processes were dumped to the garbage, where the other organic waste may be decomposed to methane, and thus encourage to climatic change (Mussatto *et al.* 2011), (Hansen *et al.* 2006). The SCG can be extracted the coffee oil using solvent extraction method. Thus, the SCG have the potential feedstock to produce the biodiesel from the coffee oil. The SCG containing the 7-17 wt.% oils content, depending on coffee varieties (Speer & Kölling-speer 2006). Moreover, the yield of coffee oil from SCG depends on the various parameters: moisture content, particle size, amount of solvent, type of solvent, extraction method, and extraction time (Al-Hamamre *et al.* 2012), (Caetano *et al.* 2012). Table I shows the various conditions of coffee oil extraction from SCG. The hexane solvent extraction is most commonly used for extracting the coffee oil by Soxhlet extractor. Moreover, the 12-16% oil yield from SCG was achieved in the extraction process, when Soxhlet and ultrasound extractions were used. Certainly, the ratio of solvent-to-SCG has a high significance of oil yield; however, the researchers have not yet studied the variation of this parameter. For instance, Al-Hamamre *et al*. studied the oil extraction from the dried spent coffee grounds (DSCG) as a renewable source for biodiesel production, the 60 g dried sample and 250 ml solvent (or the ratio of solvent-to-SCG is equal to 1:4.2 g/mL), were fixed at the different time spans to determine the yield. Caetano *et al*. studied the effect of different solvents on the coffee oil yield by using Soxhlet extractor with the condition: 2.5 to 9.5 h of extraction time, the 10 g of DSCG and 200 mL of solvent were fixed. Therefore, the optimal condition of coffee oil extraction should be studied to reduce the extraction costs, such as type of solvent, ratio of DSCG-to-solvent, and extraction time. This present study was undertaken to evaluate and optimize the highest yield of coffee oil for the solvent extraction process from DSCG, when the two parameters: ratios of DSCG-to-hexane and extraction time were studied by using the response surface methodology (RSM).

MATERIALS AND METHODS

Materials

In the Espresso coffee shops, many organic residues, spent coffee grounds (SCG) from brewing coffee were dumped to the garbage, as shown in Figure-1. The SCG were obtained from local coffee shop in Prince of Songkla University (PSU), which Arabica coffee grounds were used to brew the Espresso. An approximately 66% of moisture content in the SCG were measured by the drying oven method at 105 °C and 24 h (Abdullah & Bulent Koc 2013). The SCG must be dried to eliminate the moisture content before introduced into the extraction process. Moreover, the SCG drying process will prevent the spoilage and microbial growth (Abdullah & Bulent Koc 2013). The commercial grade of hexane solvent was used to extract the coffee oil from the dried spent coffee grounds (DSCG). All chemicals were analytical grade to analysis the compositions of coffee oil by the thin layer chromatograph with flame ionization detection (TLC/FID).

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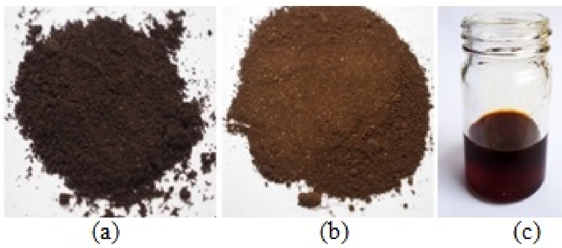

Figure-1. (a) spent coffee grounds, (b) oil-free spent coffee grounds, and (c) coffee oil.

Methods

Extraction Procedures

After complete the SCG drying process, the 10 g of DSCG and the required mass ratios of DSCG-to-hexane were loaded into the 250 mL flask for weighing by the Mettler-Toledo digital analytical balance. All experiments, the solvent extraction were operated in the batch process. The mass ratios of DSCG-to-hexane were varied under the experimental design matrixes. The magnetic bar was instantaneously put into the flask to mix the DSCG with hexane. In the coffee oil extraction process, the flask was placed on the digital magnetic stir plate to mix the DSCG with solvent by the magnetic stirrer at 30 °C of room temperature, and immediately start the timer to monitor the extraction time. After complete extraction oil, the oil-free spent coffee grounds (OFSCG) will rapidly fall to the bottom of the flask, and the solutions of hexane-to-coffee oil are on the top layer. The suspensions of OFSCG in the solutions were filtered by the filter paper (W. & R. Balston Ltd. Genuine Whatman No. 1) to separate the OFSCG and solutions. In the hexane distillation process, the solutions of hexane-to-coffee oils were stilled by the simple distillation process to remove the most hexane. In the final process, the coffee oil from the distillation process will be heated by the electrical oven at 104 °C for 6 h, to remove the residual hexane, which might also be left in the coffee oil (Abdullah & Bulent Koc 2013).

Analysis Method

Two independent parameters: mass ratios of DSCG-to-hexane and extraction time, were studied to optimize the coffee oil yield from DSCG. The yield of coffee oil was determined with equation (1). The TLC/FID (model: IATROSCAN MK-65; Mishubishi Kagahu Latron Inc.; Tokyo, Japan) was used to analyze the percentage of tri-, di-, mono-glycerides, ester, and free fatty acid (FFA) in the coffee oils.

Experimental Design

The experimental design of the solid-liquid extraction process was aimed to optimize the effects of the ratios of DSCG-to-hexane (*R*), and extraction time (*T*) on the coffee oil yield by the response surface methodology (RSM) approach. Twelve experiments were designed for 5-coded-level (-1.414, -1, 0, +1, +1.414) followed by the coded level, as shown in Table-2.

$$Y = (W_0 / W_d) \times 100 \tag{1}$$

Where Y is the coffee oil yield, W_0 (g) is weight of extracted oil and W_d (g) is weight of DSCG.

RESULTS AND DISCUSSION

Experimental Results

Table-3 is the experimental design matrixes of 2-independent-variable: DSCG-to-hexane (g/g), extraction time (min), and 1-dependent-variable: coffee oil yield (wt.%) of the solid-liquid extraction process by solvent.

Response Surface Model and Statistical Analysis

The coefficient values and statistical analysis of response surface model were analyzed by using the multiple regression equation to fit a second-order polynomial model, as shown in Table-4.

Table-1. Conditions of coffee oil extraction from SCG.

Time (min)	**Type of solvent**	**Yield** (wt.%)	**DSCG-to-solvent** (g/mL)	**Extraction method**	**References**
45	hexane	12.00	1:4	Ultrasound	(Rocha *et al.* 2014)
30	pentane	15.18	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
30	hexane	15.28	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
30	toluene	14.32	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
40	chloroform	11.15	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
30	acetone	12.92	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
50	isopropanol	10.92	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
40	ethanol	11.90	1:4.2	Soxhlet	(Al-Hamamre *et al.* 2012)
180	50:50 (hexane: 2-propanol)	21.50	1:20	Soxhlet	(Caetano *et al.* 2012)
408	isopropanol	21.00	1:20	Soxhlet	(Caetano *et al.* 2012)
150	80:20 (hexane:2-propanol)	19.50	1:20	Soxhlet	(Caetano *et al.* 2012)
570	n-octane	26.00	1:20	Soxhlet	(Caetano *et al.* 2012)
N/A	hexane	16.00	1:20	Soxhlet	(Caetano *et al.* 2012)
N/A	ethanol	16.00	1:20	Soxhlet	(Caetano *et al.* 2012)
N/A	60:40 (hexane:2-propanol)	17.00	1:20	Soxhlet	(Caetano *et al.* 2012)
N/A	70:30 (hexane:2-propanol)	21.00	1:20	Soxhlet	(Caetano *et al.* 2012)
N/A	heptane	18.00	1:20	Soxhlet	(Caetano *et al.* 2012)

N/A: the extraction time was varied in the range between 150 and 570 min

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Table-2. Coded level of independent variables.

Independent variable	Coded level				
	-1.414	-1	0	+1	+1.414
R : Ratios of DSCG-to-hexane (g/g)	1:5.1	1:8	1:15	1:22	1:24.9
T : Extraction time (min)	0.2	6	20	34	39.8

In Table-4, the positive sign of coefficient values of each parameter mean the coffee oil yield has increased. In contrast, the negative sign of coefficient values mean the decreasing of oil yield. All analysis of results were conducted with ‘‘Excel’’ solver from Microsoft Excel add-in tool to solve the optimization of equation (2). It was found that the predicted model of the relationship between the coffee oil yield and two independent variables were classified under the polynomial equation. The model of the two responses is expressed in equation (2) to obtain the highest yield of coffee oil from the solid-liquid extraction process. This model was tested for its ability to describe the experimental results and to prove for statistical significance by the *t*-test at the confidence level of 95%.

$$Y = \beta_0 + \beta_1 T + \beta_2 R + \beta_3 T^2 + \beta_4 TR + \beta_5 R^2 \quad (2)$$

where Y: coffee oil yield, R: ratio of DSCG-to-hexane, T: extraction time, and β: coefficient value.

Figure-2 shows the contour plot of the relationship between the extraction time and the mass ratio of DSCG-to-hexane on the coffee oil yield under the 30 °C room temperature. To investigate the optimal condition for the highest coffee oil yield, therefore, the Excel solver was used to solve the equation 2. The results showed that the 14.75 wt.% calculated yield of coffee oil was achieved, when the optimal condition: the 1:22.5 g/g of mass ratio of DSCG-to-hexane and 30.4 min of extraction time at the 30 °C was used. The yield of calculated coffee oil under the optimal condition can be proved by the experiment. The results from the experiment showed that the 14.68 wt.% actual experimental yield of coffee oil can be extracted from DSCG under the optimal condition, which the experimental yield are close to the calculated coffee oil yield. Moreover, the compositions of coffee oil after eliminate the residual hexane were analyzed by TLC/FID, 81.156 wt.% TG, 5.926 wt.% DG, 11.428 wt.% MG, 1.078 wt.% ester, and 0.412 wt.% FFA were found. The major problem of biodiesel production from vegetable oil is the FFA, which the FFA is transesterification reaction was used. As the results, the ester conversion was reduced by the saponification reaction (Somnuk *et al.* 2013). However, the coffee oil has the FFA content lower than 0.5 wt.%. Consequently, the coffee oil will be produced the biodiesel by using a base-catalyzed direct transesterification reaction.

CONCLUSIONS

The aim of this study was to optimize the highest yield of coffee oil for the solvent extraction process from SCG after brewing coffee as a potential alternative raw material for biodiesel from coffee oil. In the part of oil extraction process, the results of this research demonstrates that the 14.68 wt.% actual experimental yield of coffee oil can be extracted from dried spent coffee grounds (DSCG) under the optimal condition: the 1:22.5 g/g of mass ratio of DSCG-to-hexane and 30.4 min of extraction time under the 30 °C of room temperature. Increasing of mass ratio of DSCG-to-hexane will enhances the diffusion of solid and solvent, and improves the transport of the solvent while mixture flow through the pores and surface area of DSCG in the oil extraction process. Therefore, the organic residues, the SCG have the potential raw material to use for biodiesel production from the coffee oil and to use for combustion energy from OFSCG.

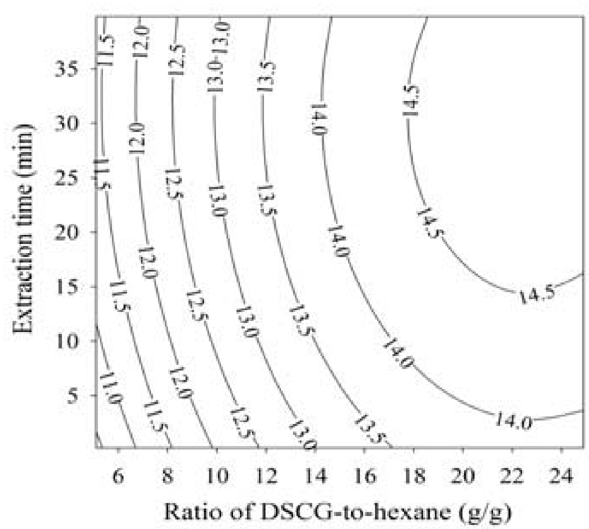

Figure-2. Contour plot of extraction time and mass ratio of DSCG-to-hexane on the yield of coffee oil under the room temperature of 30 °C.

Table-3. Experimental design matrix.

Run	**Ratio of DSCG-to-hexane, *R*** (g/g)	**Extraction time, *T*** (min)	**Yield of coffee oil, *Y* (wt.%)**
1	1: 22.0	34.0	14.5700
2	1: 8.0	34.0	12.6580
3	1: 15.0	20.0	14.0237
4	1: 22.0	6.0	14.4017
5	1: 24.9	20.0	14.6323
6	1: 15.0	20.0	14.0283
7	1: 8.0	6.0	12.4070
8	1: 15.0	39.8	14.1125
9	1: 15.0	20.0	13.9887
10	1: 5.1	20.0	10.7623
11	1: 15.0	0.2	12.6600
12	1: 15.0	20.0	13.9773

Table-4. Coefficient values of response surface model.

Coefficient	β_0	β_1	β_2	β_3	β_4	β_5
Value	8.130	0.06448	0.500	-0.00098	-0.00021	-0.01096
p-value	0.000263	0.231	0.00453	0.296	0.926	0.01861

(R^2 = 0.927, and $R^2_{adjusted}$ = 0.866)

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