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Original Article

Optimization of coffee oil extraction from spent coffee grounds using four solvents and prototype-scale extraction using circulation process

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ABSTRACT

The optimization of two parameters—espresso coffee oil extraction time and the ratio of dried spent coffee grounds (DSCG)-to-solvent—were conducted on DSCG employing four solvents. Extracted yields were investigated using response surface methodology. The two independent variables—ratio of DSCG-to-solvent (5.1–24.9 g/g) and extraction time (0.2–39.8 min)—were optimized in the batch mode. The predicted model was verified using actual experiments. The experimental yields achieved were 14.7 percent by weight (wt%; using hexane), 13.1 wt% (using anhydrous ethanol), 11.8 wt% (using hydrous ethanol), and 7.5 wt% (using methanol). Prototype extraction was tested using a circulation process. Approximately 11.8 wt% oil yield of prototype extraction could be obtained from DSCG under the optimal conditions of 30.4 min extraction time and 22.5 g/g ratio of DSCG-to-hexane from the laboratory-scale results. In this study, the miscella (the solution of coffee oil dissolved in the solvent) from up to six successive extractions was investigated to determine the optimal oil extraction process. The repeated miscella from each successive extraction showed high efficiency and stability of coffee oil yield similar to that obtained using fresh hexane.

Introduction

Organic residues from brewed coffee—the so-called spent coffee grounds (SCG)—have an oil content of approximately 10–15 percent by weight (wt%), depending on the coffee variety (Jenkins et al., 2014) and have a promising feedstock amount for the production of biodiesel (Caetano et al., 2012; Al-Hamamre et al., 2012; Vardon et al., 2013). Approximately 8 million t of coffee produced globally each year, roughly 1.3 billion L of biodiesel from coffee oil could be added to the world fuel supply (Jenkins et al., 2014; Campos-Vega et al., 2015). SCG is the residue obtained after brewing and requires a good waste management plan. Nestlé, a Swiss company and the maker of the world's first instant coffee in 1938 under the brand name NESCAFÉ, founded a large-scale production line for coffee extraction and spray drying of coffee beans (Nestlé, 2015a) and SCG from the brewed coffee process has been used as a renewable fuel resource (Nestlé, 2015b). Instant coffee is prepared from instant coffee powder (ICP) mixed with hot water or steam and accounts for almost half of the world's coffee consumption (Mussatto et al., 2011). After coffee extraction with hot water, the solid waste of the SCG amounts to 2 kg per kg of ICP (Pfluger, 1975). Generally, SCG from the coffee industry are used as a renewable energy resource, and this waste is collected by specialized agencies and sold for various purposes such as composting, gardening, bioenergy production and mushroom growing (Campos-Vega et al., 2015). After brewing, SCG from both instant and roasted coffee can be dumped, and the organic waste decomposes partly to methane, contributing more to climatic changes (Mussatto et al., 2011). In Thailand, coffee beans have been used to produce instant coffee, roasted coffee, ground coffee and canned coffee (Pongsiri, 2013). Increasing domestic consumption has led to an increase in SCG as well (Hansen et al., 2006; Campos-Vega et al., 2015). SCG contains 10–15 wt% oil content, depending on coffee varieties (Speer and Kölling-Speer, 2006; Jenkins et al., 2014). In the coffee oil extraction from SCG using a Soxhlet extractor, Al-Hamamre et al. (2012) studied oil extraction from dried spent coffee grounds (DSCG) for biodiesel production; 60 g DSCG and 250 mL solvent were kept for different time spans to determine the yield. Caetano et al. (2012) studied the effect of different solvents on coffee oil yield using a Soxhlet extractor for 2.5–9.5 h. Their results showed that 21.5 wt% of oil was obtained after 3 h extraction time with a mixed solvent (hexane:isopropanol, 50:50 by volume),

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while the use of isopropanol solvent alone resulted in 21.0 wt% of oil within 6.8 h. The use of pure isopropanol incurs a higher operating cost than the use of mixed solvent (Caetano et al., 2012).

To the best of the authors' knowledge, little research has directly focused on the optimization of coffee oil extraction from DSCG using the four solvents: hexane, anhydrous ethanol, hydrous ethanol and methanol applied in the currents study. Moreover, the prototype-scale was tested using solvent-circulated extraction instead of batch-type modes. Therefore, the main objective of this study was to demonstrate the optimization of coffee oil yields from each solvent type using response surface methodology (RSM) using two parameters—the extraction time and the ratio of DSCG-to-solvent.

Materials and methods

Materials

As shown in Fig. 1, the organic residues—the spent coffee grounds (SCG), the dried spent coffee grounds (DSCG), and the defatted spent coffee ground (DFSCG)—are normally considered as waste. The current study used SCG of the Arabica form from espresso brewing with a moisture content of approximately 66%. The SCG should be dried before oil extraction, as a high moisture content causes spoilage due to decomposition (Rocha et al., 2014). Thus, freshly brewed SCG should go through a drying process to inhibit microbial growth by applying a drying oven method at 105 °C for 24 h. The DSCG were used as the raw material for the solvent extraction process. The purities of the commercial grade solvents were: 29.7 wt% hexane, 99.9 percent by volume (vol%) ethanol (anhydrous ethanol), 95 vol% ethanol (hydrous ethanol) and 99.9 wt% methanol. Table 1 shows the physical and chemical compositions of DSCG and DFSCG, while the properties and price of the four solvents are shown in Table 2.

Laboratory scale of oil extraction from spent coffee grounds with the four solvents

In all experiments, the solvent extractions were batch processed at a laboratory scale. In the extraction, 10 g of DSCG was loaded into a 250 mL flask resting on a digital analytical balance (AL204; Mettler-Toledo; Küsnacht, Switzerland) with an accuracy of 0.0001 g. The required ratios of DSCG-to-solvent were then dosed into the flask and wrapped with aluminum foil to prevent solvent evaporation. The DSCG and solvent were constantly mixed using a 500 rpm magnetic bar at room temperature (approximately 30 °C). The solvent flowed over the surfaces of the DSCG and seeped through the pores during the designated extraction time. Subsequently, each mixture was filtered through filter paper (Genuine Whatman No. 1; W. & R. Balston Ltd.; Kent, UK) to separate the defatted spent coffee grounds (DFSCG) and the miscella (the solution of coffee oil dissolved in the solvent). The solvent was then separated from the coffee oil using a simple distillation process. To ensure that no residual solvent remained, the oil was further heated at 104 °C for 6 h (Abdullah and Koc, 2013). The procedures for coffee oil extraction were repeated for each of the four solvents.

Table 1
Physical and chemical compositions of dried spent coffee grounds (DSCG) and (defatted spent coffee ground) DFSCG.

Property	DSCG	DFSCG
Average particle size (μm)	237.20	216.20
Higher heating value (MJ/kg)	23.10	20.40
Element (weight %, wt%)		
Carbon	52.95	48.34
Hydrogen	6.76	6.17
Nitrogen	2.10	2.39
Sulfur	0.12	0.09
Oxygen	38.07	43.01
Ash (wt%)	1.59	1.86
Protein (wt%)	14.39	14.80
Crude fiber (wt%)	21.43	20.44
Total sugar (wt%)	14.09	12.93

wt% = percent by weight.

Analytical methods

Physical and chemical composition analysis involved DSCG and DFSCG for: carbon (C), hydrogen (H), nitrogen (N), sulfur (S) and oxygen (O) using a CHNS-O analyzer (CE Instruments Flash EA 1112 Series; ThermoQuest; Milan, Italy). The average particle size distribution was estimated using a laser particle size analyzer (LS 230; Beckman Coulter; Brea, CA, USA). The percentages of nutritional compositions were determined for the ash using the gravimetric method (method 942.05; Association of Official Analytical Chemists, 2000), protein, using the Kjeldahl method (method 991.20; Association of Official Analytical Chemists, 2000) and crude fiber using a fiber analyzer (ANKOM200; ANKOM Technology, New York, NY, USA). The total sugar was tested using the Lane-Eynon general volumetric method (method 923.09; Association of Official Analytical Chemists, 2000). The fatty acid profiles were analyzed using a gas chromatograph-flame ionization detector (GC 6890; Hewlett Packard; Santa Clara, CA, USA). The higher heating value (HHV) of the products from the DSCG, DFSCG and oil were measured using a calorimeter (C5000 control; IKA GmbH; Stauffen, Germany). A thin layer chromatograph with flame ionization detection (IATROSCAN MK-65; Mishubishi Kagahu latron Inc.; Tokyo, Japan) was used to analyze the purity of the ester, triglyceride, diglyceride, monoglyceride and free fatty acid (FFA) in the oil.

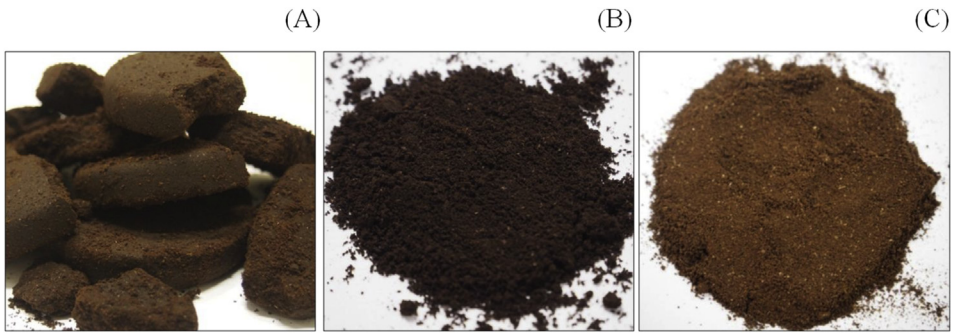

Fig. 1. Products in the process: (A) freshly brewed spent coffee grounds; (B) dried spent coffee grounds; (C) defatted spent coffee grounds, after oil extraction.

Table 2
Property and price of the four solvents used.

Property	Hexane	Anhydrous ethanol	Hydrous ethanol	Methanol
Purity[a] (%, minimum)	29.7 (wt%)	99.9 (vol%)	95.0 (vol%)	99.9 (wt%)
Density @30 °C (kg/L)	0.664	0.784	0.800	0.782
Boiling point (°C)	62.40	78.50	78.15	64.70
Price (USD/kg)	1.54[a]	0.46[b]	0.41[b]	0.37[c]

wt% = percent by weight, vol% = percent by volume.
[a] Sourced: Chemical Intelligence (2015).
[b] Sourced: Bank of Thailand (2015).
[c] Sourced: Methanol Market Services Asia (2015).

Table 3
Coding of independent variables for coffee oil extraction.

Independent variable	Coded independent variable				
	−1.414	−1	0	+1	+1.414
R = Ratio of DSCG[a]-to-solvent (g/g)	5.1	8	15	22	24.9
T = Extraction time (min)	0.2	6	20	34	39.8

[a] Dried spent coffee grounds.

In the analytical methods applied to extract oil, the oil yield was calculated using Equation (1) from Obruca et al. (2014):

$$Y = (W_o/W_d) \times 100 \quad (1)$$

where Y (wt%) is the coffee oil yield; W_o (grams) is the weight of extracted oil; and W_d (grams) is the weight of DSCG.

Experimental design for oil extraction

RSM using a 5-level and 2-factor CCD was used to optimize the coffee oil extraction. Multiple regression analysis was employed to derive a second-order polynomial equation to predict the coffee oil yield (Y). A general second-order polynomial equation was used, as shown in Equation (2) from Ranic et al. (2014):

Table 6
Analysis of variance of predicted model for coffee oil extraction.

Source	SS	MS	F_0	F_{signif}	DOF
For hexane					
Regression	13.66	2.731	15.18	0.00237	5
Residual	1.079	0.180			6
LOF Error	1.077	0.359	559.8274	0.000128	3
Pure Error	0.00192	0.000642			3
Total	14.74				11
For anhydrous ethanol					
Regression	14.95	2.990	11.14	0.00538	5
Residual	1.610	0.268			6
LOF Error	1.603	0.534	239.5678	0.000454	3
Pure Error	0.00669	0.00223			3
Total	16.56				11
For hydrous ethanol					
Regression	29.02	5.803	23.26	0.000733	5
Residual	1.497	0.250			6
LOF Error	0.974	0.325	1.8623	0.311	3
Pure Error	0.523	0.174			3
Total	30.51				11
For methanol					
Regression	20.24	4.048	5.424	0.03136	5
Residual	4.477	0.746			6
LOF Error	4.424	1.475	82.4973	0.00222	3
Pure Error	0.05362	0.01787			3
Total	24.72				11

LOF = lack of fit; SS = sum of squares; MS = mean sum of squares.

Table 4
Experimental design matrix and results for coffee oil extraction.

Run	R (g/g)	T (min)	Yield of coffee oil, Y (wt %)			
			Hexane	Anhydrous ethanol	Hydrous ethanol	Methanol
1	22.0	34.0	14.5700	12.9880	11.1277	8.1410
2	8.0	34.0	12.6580	11.3193	10.8025	4.4457
3	15.0	20.0	14.0237	12.3297	11.2660	6.4753
4	22.0	6.0	14.4017	11.8473	11.1230	7.0665
5	24.9	20.0	14.6323	12.8570	11.2040	6.5310
6	15.0	20.0	14.0283	12.3700	10.2660	6.3433
7	8.0	6.0	12.4070	10.4837	6.9047	4.0377
8	15.0	39.8	14.1125	12.6163	11.2660	4.3570
9	15.0	20.0	13.9887	12.2723	10.7830	6.2563
10	5.1	20.0	10.7623	9.3073	7.4247	2.9250
11	15.0	0.2	12.6600	9.7380	7.6667	6.6507
12	15.0	20.0	13.9773	12.2740	10.5970	6.1607

Table 5
Coefficient value and p-value for the predicted model.

Term	Hexane		Anhydrous ethanol		Hydrous ethanol		Methanol	
	C value	p-value	C value	p-value	C value	p-value	C value	p-value
β_0	8.130	0.000262	6.207	0.00307	−0.04543	0.972	0.853	0.707
β_1	0.06449	0.231	0.131	0.06835	0.327	0.00121	−0.000235	0.998
β_2	0.500	0.00452	0.423	0.02232	0.716	0.00173	0.538	0.05888
β_3	−0.000981	0.295	−0.00222	0.07796	−0.00245	0.05072	−0.00102	0.578
β_4	−0.000211	0.926	0.000778	0.778	−0.00993	0.00801	0.00170	0.713
β_5	−0.01095	0.01856	−0.00984	0.05667	−0.01137	0.03030	−0.01202	0.135
R^2	0.927		0.903		0.951		0.819	
$R^2_{adjusted}$	0.866		0.822		0.910		0.668	

R^2 = coefficient of determination; $R^2_{adjusted}$ = adjusted coefficient of determination; β_0, β_i, β_{ii}, and β_{ij} = intercept, linear, quadratic and interaction constant coefficients, respectively.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1} \sum_{j=i+1} \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

where Y is the response; x_i and x_j are the uncoded independent variables; β_0, β_i, β_{ii}, and β_{ij} are the intercept, linear, quadratic and interaction constant coefficients, respectively; k is the number of variables; and ε is the error.

The two independent variables—the ratio of DSCG-to-solvent (R) and the extraction time (T)—were first studied in batch mode. Table 3 shows the five code levels for the varying ranges of independent variables, which were coded as −1.414, −1, 0, +1, and +1.414. The coded independent variables were repeated for the four solvents (hexane, anhydrous ethanol, hydrous ethanol and methanol).

Results and discussion

Experimental results for coffee oil extraction

Fig. 1 shows the products from the coffee oil extraction process. The physical characteristics of the DSCG were a very fine, dark-brown powder. After oil extraction, the color in the DFSCG had changed to pale brown. According to Table 1, 21.43 wt% and 20.44 wt% of crude fiber were evaluated in the DSCG and DFSCG, respectively. The protein content was 14.39 wt% and 14.80 wt% in DSCG and DFSCG, respectively. Thus, the protein content in the latter was a little higher because the residual oil in the DSCG had been removed. In conclusion the protein content for use as a nutritious animal feed increased after oil extraction because the total weight of the DFSCG decreased after the oil in the DSCG was removed by the solvent extraction. Thus, the protein content in DFSCG increased more than the protein content in DSCG when compared on an equal weight basis of DSCG. These results were similar to those described by Berk (1992) where the concentration of protein increased when most of the soluble non-protein constituents had been removed. The products before and after oil extraction from the DSCG and DFSCG have been traded as nutritional products for animal feed (Bouafou et al., 2011). The remaining fuel energy density of the waste coffee grounds was studied as a biomass energy resource and for solid-fuel. Approximately 23.1 MJ/kg of HHV from DSCG, and 20.4 MJ/kg from DFSCG, were derived. The HHV slightly reduced by approximately 11.65% after the oil in the DSCG had been extracted. The ash from SCG burning provides a

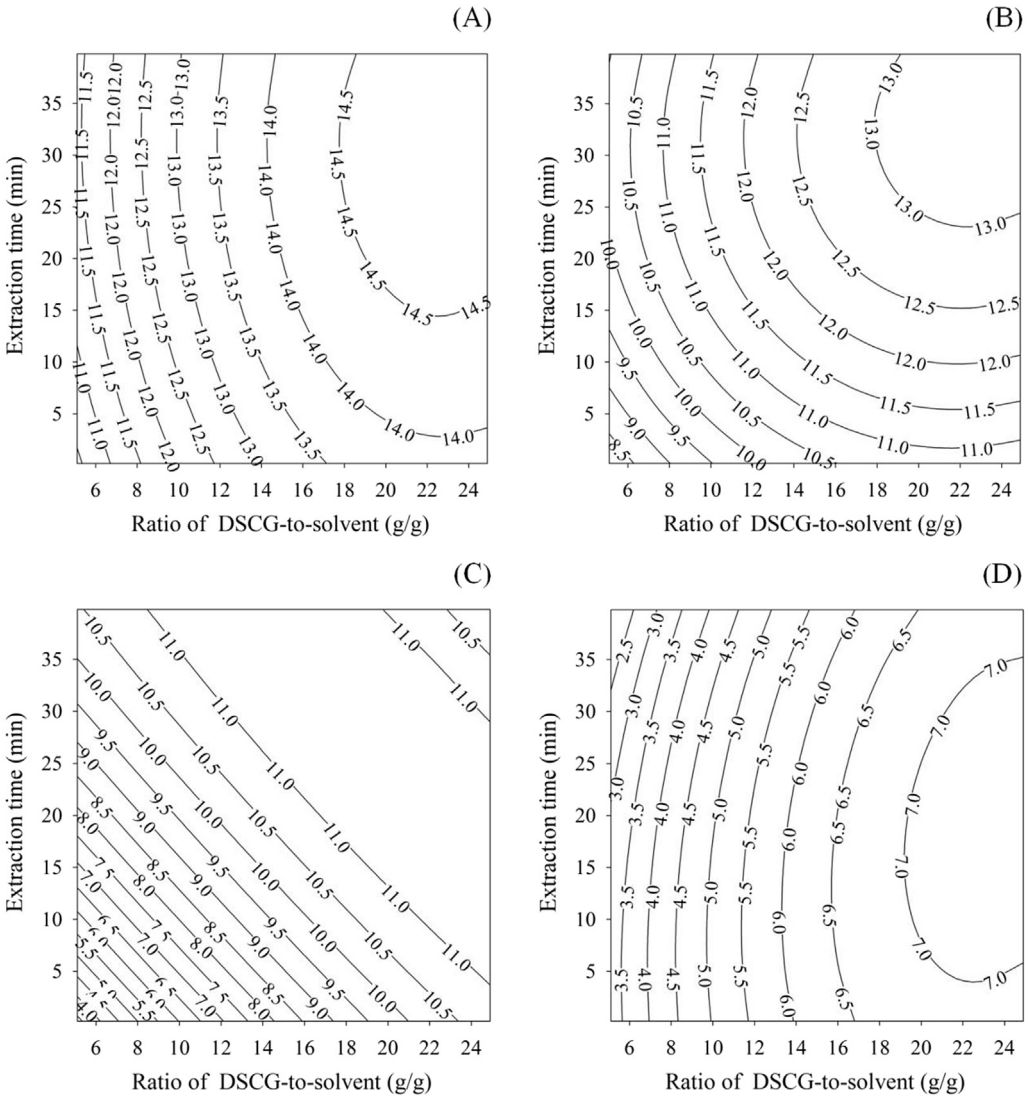

Fig. 2. Contour plots of ratio of dried spent coffee grounds (DSCG)-to-solvent and extraction time on the coffee oil yield at 30 °C: (A) hexane; (B) anhydrous ethanol; (C) hydrous ethanol; (D) methanol.

Table 7
Optimal conditions and coffee oil composition from dried spent coffee grounds (DSCG) using four different solvents.

Parameter and composition	Hexane	Anhydrous ethanol	Hydrous ethanol	Methanol
Extraction time (min)	30.4	33.5	25.5	19.6
Ratio of DSCG-to-solvent (g/g)	22.5	22.8	20.3	23.8
Predicted oil yield	14.7	13.2	11.4	7.2
Verified oil yield	14.7	13.1	11.8	7.5
Free fatty acid (wt%)	0.412	1.406	1.296	0.872
Triglyceride (wt%)	81.156	9.562	9.487	8.044
Diglyceride (wt%)	5.926	0.618	0.816	0.751
Monoglyceride (wt%)	11.428	85.183	84.894	90.148
Ester (wt%)	1.078	3.230	3.507	0.185

wt% = percent by weight.

good ingredient for fertilizers as it has high levels of calcium, phosphorous and magnesium (Silva et al., 1998). The HHV of both DSCG and DFSCG are similar to coal and are higher than wood and other softwood biomass, (approximately 19 MJ/kg HHV) according to Demirbaş (1997). The ash contents of 1.59 wt% (after DSCG burning) and 1.86 wt% (after DFSCG burning) were noted. These ash contents were lower than from burning woody biomass such as tea waste, wood bark, wheat straw, tobacco stalk, tobacco leaf and olive husk (Demirbaş, 1997). The experimental design matrix and the laboratory scale results using the four solvents (hexane, anhydrous ethanol, hydrous ethanol and methanol) in this study are shown in Table 4.

Predicted model and statistical analysis for coffee oil extraction

Regression analyses (detailed in Table 5) were performed to fit the response models with the results of 12 experiments using multiple regression add in tool for the Excel software package (version 2013; Microsoft Corp.; Redmond, WA, USA). The results showed that the relationship between the coffee oil yield (*Y*) and

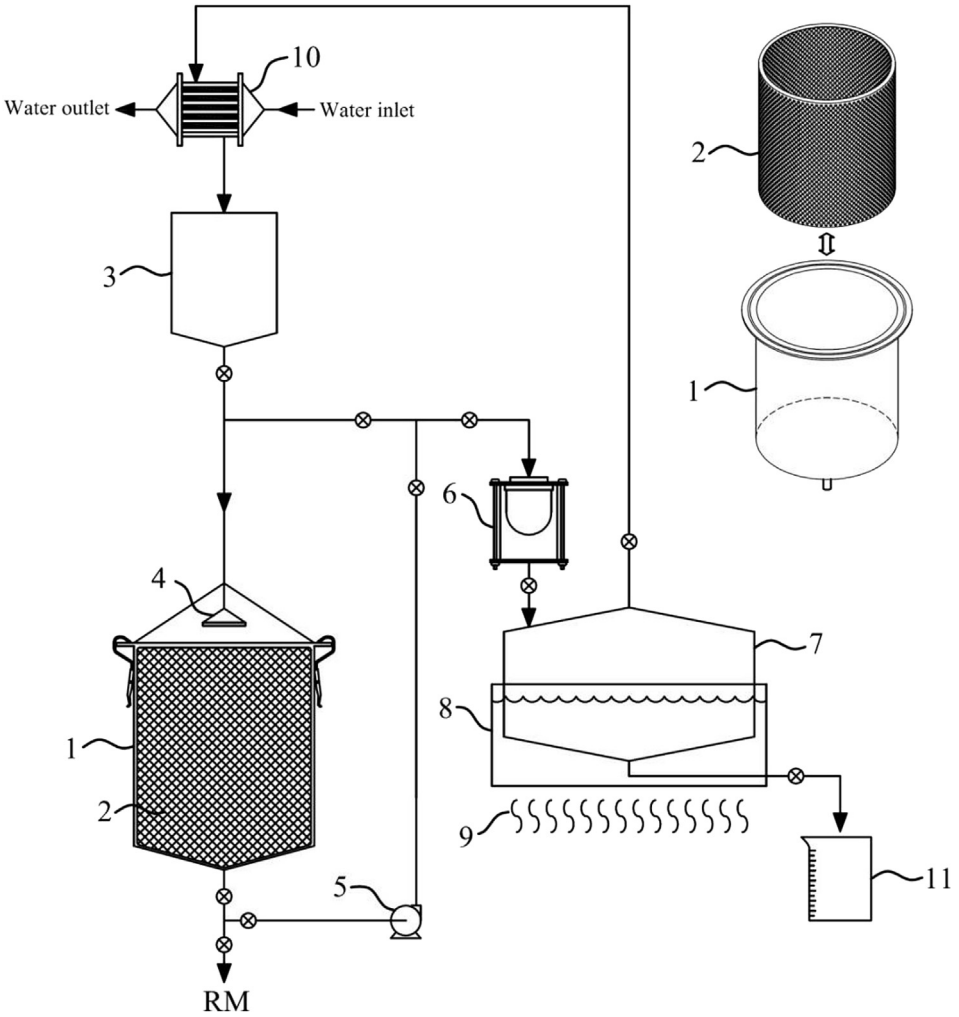

Fig. 3. Schematic diagram of prototype-scale coffee oil extraction. (1: extraction tank, 2: stainless steel sieve, 3: solvent tank, 4: spray head, 5: circulating pump, 6: ceramic membrane filter, 7: distillation tank, 8: hot water tank, 9: heat source, 10: shell and tube heat exchanger, and 11: coffee oil beaker).

the two independent variables—the ratio of DSCG-to-solvent (R), and the extraction time (T)—was best obtained using a quadratic model for all four solvents, as Expressed in Equation (3).

$$Y = \beta_0 + \beta_1 T + \beta_2 R + \beta_3 T^2 + \beta_4 TR + \beta_5 R^2 \quad (3)$$

where Y is the coffee oil yield; R is the ratio of DSCG-to-solvent; T is the extraction time; and β is the coefficient value.

The goodness of fit of this predicted model was defined by the coefficient of determination (R^2), the adjusted coefficient of determination ($R^2_{adjusted}$), the probability of error value (p-value) and the coefficient values of the model, as listed in Table 5. The p-value to test statistical significance of each term in the quadratic model was obtained. The smallest p-value of each term for the four solvents indicates the highest significant contribution. The lowest p-values of 0.00452, 0.02232, and 0.05888 occurred in the term $\beta_2 R$, when hexane, anhydrous ethanol and methanol, were used, respectively. Consequently, the ratio of DSCG-to-solvent was significantly higher than the extraction time for these three solvents, whereas for hydrous ethanol the lowest p-value of 0.00121 occurred in the term $\beta_1 T$. The analysis of variance of the predicted model for the four solvents is shown in Table 6.

Contour plots, optimization and verification of coffee oil yield in laboratory-scale

Fig. 2A–D shows the relationship between the dependent (yield) and independent variables (ratio of DSCG-to-solvent and extraction time), which are presented using contour plots for hexane, anhydrous ethanol, hydrous ethanol and methanol, respectively. The optimum conditions of the oil yield were evaluated using the solver add in the Excel software package (version 2013; Microsoft Corp.; Redmond, WA, USA) and are shown in Table 7. The optimum conditions for the four solvents obtained from the model were employed to verify the model yield results with the actual experiment yield results. The oil yield results from the actual experiment were: 14.7 wt%, 13.1 wt%, 11.8 wt%, and 7.5 wt % for hexane, anhydrous ethanol, hydrous ethanol and methanol, respectively.

Consideration of solvent type for coffee oil extraction

The highest oil yield from the extraction process was the prime consideration of selection among the four solvents. The maximum oil yield of 14.7 wt% was achieved using hexane under the optimal conditions of 30.4 min of extraction time and 22.5 g/g of ratio of DSCG-to-solvent. The next highest oil yield of 13.1 wt% was obtained when anhydrous ethanol was used. If the highest yield were the only value of interest then ethanol, either anhydrous or hydrous, is a reasonable choice because the price of either form is approximately one-third of the hexane price, as shown in Table 2. When the environmental effects are considered, anhydrous ethanol and hydrous ethanol are favorable. Ethanol is organic and hence more environmentally friendly than toxic solvents such as hexane and methanol, and it is mainly derived from a crop fermentation process (Intarat et al., 2014). In Thailand, ethanol is mostly produced from cassava and molasses and it does not need to be imported (Somnuk et al., 2016). Removing the residual water in ethanol using a molecular sieve to improve its purity incurs an incremental cost (Wei-Cheng et al., 2014). The cost of anhydrous ethanol production is higher than that for hydrous ethanol; however, the oil yields from the former (13.1 wt% yield) and the latter (11.8 wt%) are not much different. Anhydrous ethanol thus is not as cost-effective as hydrous ethanol in this context.

Table 8
Composition of fatty acid profile in the extracted coffee oil with hexane.

Common name	Fatty acid	Content (wt%)
Caprylic acid	C8:0	0.01
Nonanoic acid	C9:0	0.00
Capric acid	C10:0	0.01
Lauric acid	C12:0	0.02
Myristic acid	C14:0	0.09
Pentadecanoic acid	C15:0	0.00
Palmitic acid	C16:0	34.44
Palmitoleic acid	C16:1	0.04
Stearic acid	C18:0	0.00
Oleic acid	C18:1	7.74
Linoleic acid	C18:2	43.12
Alpha linolenic acid	C18:3	1.18
Arachidic acid	C20:0	2.83
Paullinic acid	C20:1	0.27
Behenic acid	C22:0	0.59
Erucic acid	C22:1	0.19
Lignoceric acid	C24:0	0.29
Nervonic acid	C24:1	0.01

wt% = percent by weight.

The FFA content in oil is used as an index to determine the quality of extracted oil. Unfortunately, more than 1 wt% of FFA content in the extracted oil was measured when either form of ethanol was used, as shown in Table 7. The high FFA contents in the coffee oil when ethanol was used could be reduced to less than 1 wt% through acid-catalyzed esterification, followed by base-catalyzed transesterification (Abbaszaadeh et al., 2012; Somnuk et al., 2013, 2015). This process would increase the operating time, chemical cost and wages. Thus, as coffee oil containing less than 1 wt% FFA content was extracted from DSCG when hexane was used, hexane should be chosen in terms of achieving the highest yield and a higher quality of extracted oil.

Table 9
Characterization of coffee oil extraction employing hexane.

Property	Coffee oil
Mean molecular weight (g/mol)	636.3
Free fatty acid (wt%)	0.412
Triglyceride (wt%)	81.156
Diglyceride (wt%)	5.926
Monoglyceride (wt%)	11.428
Ester (wt%)	1.078
Higher heating value (kJ/kg)	38377.3
Density at 30 °C (kg/L)	0.925

wt% = weight %.

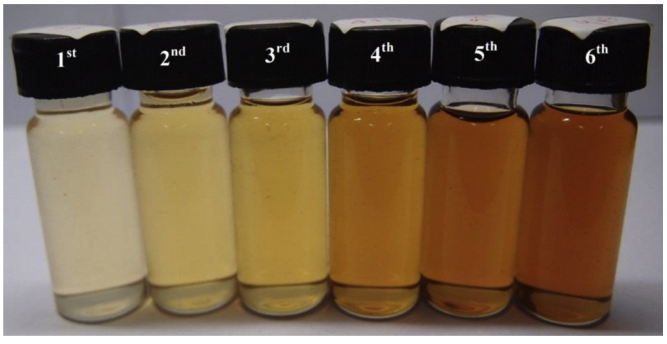

Fig. 4. Color intensity of miscella after coffee oil extraction after each of six cycles of extraction.

Oil extraction from spent coffee grounds using prototype scale

Methods of oil extraction from spent coffee grounds using prototype scale

Fig. 3 shows a schematic diagram at a prototype scale for coffee oil extraction employing a circulation process. The optimal conditions for hexane from the laboratory-scale results were applied to the prototype extraction. Approximately 1200 g DSCG were placed into a stainless steel sieve (approximately 45 L, equipped with a #500 sieve with a sieve size of 25 μm), which was installed inside a cylindrical extraction tank 360 mm in diameter and 450 mm in height. The lid of the tank was closed and locked. The fresh hexane from a solvent tank was then sprayed onto the DSCG. The pump was turned on to circulate the hexane in the tank, and the timer immediately initiated. Hexane was kept circulating through the sieve until the completion of oil extraction (30.4 min, as obtained from the lab-scale optimization results.) The miscella component was then sucked from the bottom of the extraction tank into the distillation tank. Most of the DFSCG had already been filtered through the stainless steel sieve, but smaller particles suspended in the miscella were further filtered through a 10 μm ceramic membrane. The clearer, yellow miscella component was pumped into the distillation tank, which was sitting in a hot water tank, to separate the solvent from the coffee oil using a simple distillation method. In the solvent recovery process, hexane vapor from the distillation process was condensed using a water-cooled shell and tube heat exchanger.

Results of oil extraction from dried spent coffee grounds using prototype scale

From the prototype scale results, approximately 11.8 wt% coffee oil yield could be extracted from DSCG under the optimal conditions of 30.4 min extraction time and 22.5 g/g ratio of DSCG-to-hexane. Tables 8 and 9 show the composition of the fatty acid profile and characterization in the extracted coffee oil with hexane, respectively. To investigate an alternative to using fresh hexane for each new extraction, the effect of miscella reuse was examined in a multiple extraction procedure, coined here as repeated miscella (RM) or the solution of accumulated coffee oil dissolved in the miscella. RM was repeatedly used to extract oil from subsequent batches of fresh DSCG. Up to six RM cycles were investigated. The RM was drained from the bottom of the extraction tank, the DSCG

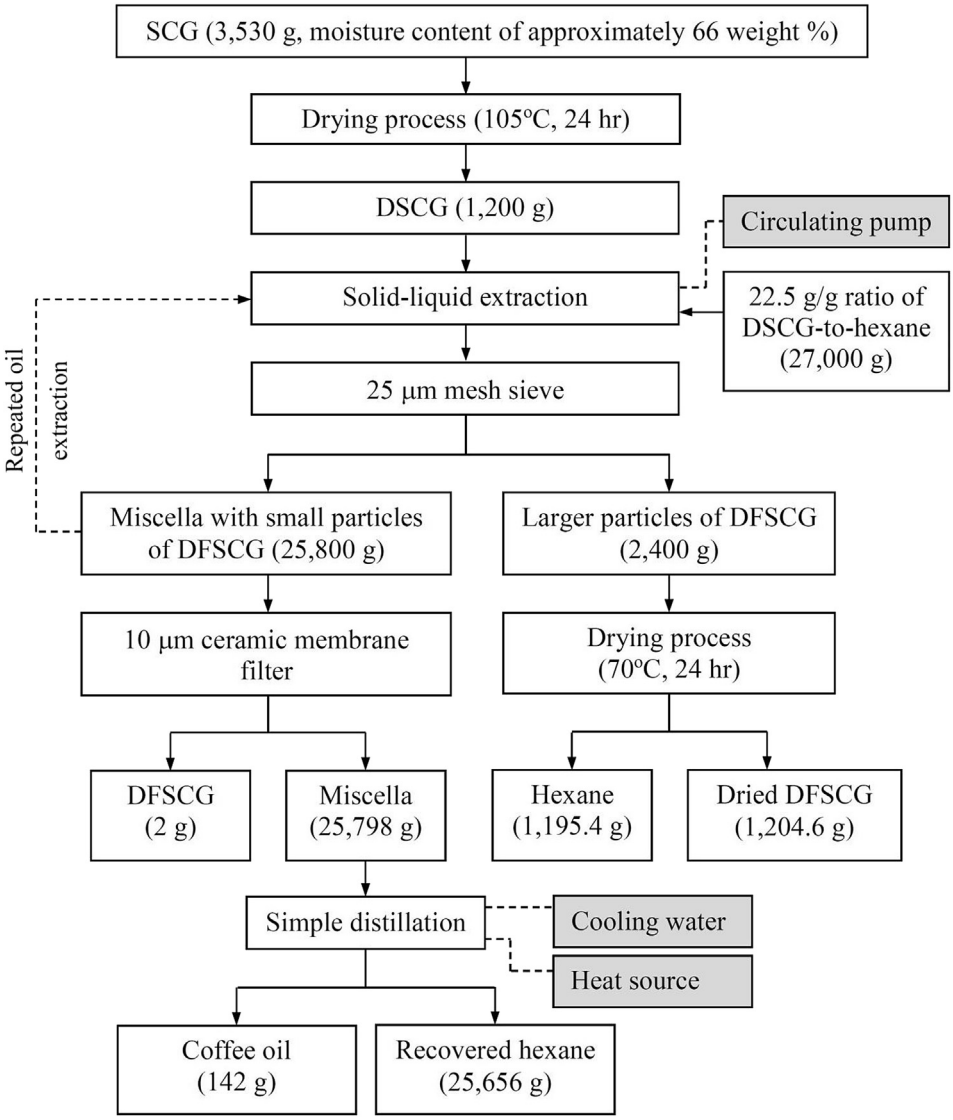

Fig. 5. Material balance of coffee oil extraction from spent coffee grounds (SCG, moisture content approximately 66 wt%), dried spent coffee grounds (DSCG), defatted spent coffee grounds (DFSCG).

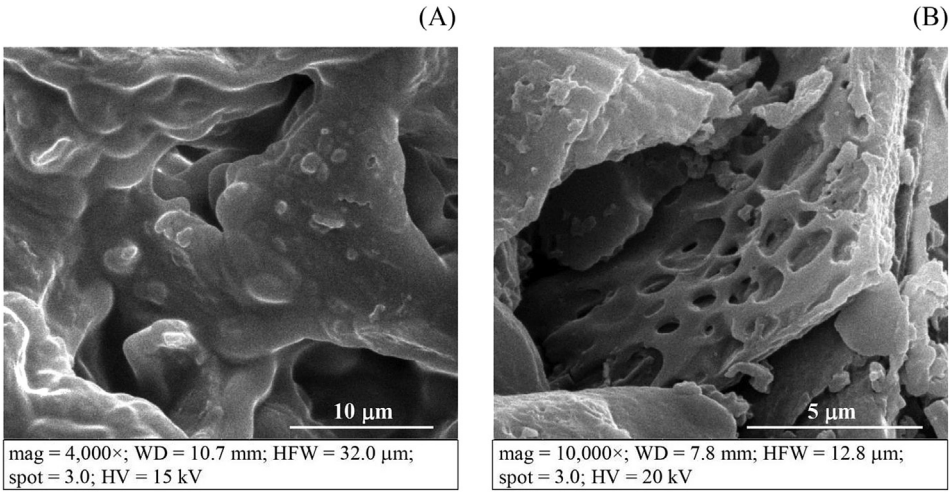

Fig. 6. Scanning electron microscopy images: (A) dried spent coffee grounds; (B) defatted spent coffee grounds (mag = machine magnification; WD = working distance; HFW = horizontal field width; spot = qualitative measure of beam current; HV = accelerating voltage).

was dumped, and fresh DSCG was then loaded (Fig. 3). The coffee oil in the new batch of DSCG was extracted using this RM. The concentration of oil in the RM thus increased after each oil extraction. Fig. 4 shows the color intensity of the RM, increasing with each cycle of extraction, indicating that the concentration of oil in the RM was incrementally increased. The concentrated oil in the RM was finally separated using simple distillation. The coffee oil in the miscella from the first cycle through to the sixth was: 0.55 wt% per g miscella, 0.99 wt% per g miscella, 1.51 wt% per g miscella, 1.98 wt% per g miscella, 2.46 wt% per g miscella and 2.93 wt% per g miscella. Clearly, the viscosity of the RM also incrementally increased and impeded filtering, but it was still tolerable throughout. The effort to reuse the RM was proven worthwhile; less hexane was needed and the energy expended to extract the oil was reduced.

Material balance of prototype-scale of coffee oil extraction

Fig. 5 shows the material balance of the coffee oil extraction using the circulation process. The final products after distillation process were: a coffee oil yield of 142 g (approximately 11.8 wt%) and recovered hexane of 25,656 g (approximately 95 wt% hexane was recovered, compared with the 27,000 g initial hexane content).

Scanning electron microscopy images

Fig. 6 shows the scanning electron microscopy (SEM) images of DSCG and DFSCG. DFSCG appear to have smoother surfaces than DSCG, as shown in Fig. 6B. The oil drops appeared to be extracted from the inside of the DFSCG pores; this oil was drawn toward the exterior of the DFSCG particles under solvent extraction.

The oil extracted from SCG can be used as a promising feedstock for renewable energy resources. The SCG as liquid fuel and solid fuel can be used as a raw material for biodiesel production and as a biomass source. In the laboratory-scale oil extraction, the experimental yields using hexane, anhydrous ethanol, hydrous ethanol and methanol were: 14.7 wt%, 13.1 wt%, 11.8 wt% and 7.5 wt%, respectively. The best-performing hexane solvent was selected to extract 1200 g DSCG in a prototype-scale extraction employing a circulation process. An oil yield of 11.8 wt% was extracted under the optimal conditions of 30.4 min extraction time and 22.5 g/g ratio of DSCG-to-hexane. The RM could be reused to extract the oil in fresh DSCG in subsequent extraction batches. Therefore, the hexane content and energy consumption decreased when RM was used in the prototype-scale extraction using a circulation process.

Conflicts of interest

The authors declare that there are no conflicts of interest.

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