

Supplementary material for “Optimization of ultrasound-assisted extraction of spent coffee grounds oil using response surface methodology”

Table S1. Comparison among studies results using common extraction technologies for SCG oil.

Matrix specifications	Optimum experimental conditions	Max. oil yield	Key findings (Oil quality parameter excluded)	Reference
<u>Ultrasound-Assisted Extraction (UAE)</u>				
Dried SCG (<i>C. Arabica</i> / <i>C. Robusta</i> blend) PS : 0.315-0.6 mm	US bath Single parameter optimization: L/S (10, 15, 20). <i>f</i> : 40 kHz; UP: 400 W; n-hexane; L/S: 20 mLg ⁻¹ ; T: RT; ED: ≈120 min.	≈14	A correlation was remarked between increasing solvent volume and oil yield.	[34]
Dried SCG (<i>C. Arabica</i>) PS: ND	US bath 2 ² factorial design: T (20-60 °C); L/S (2-4 mLg ⁻¹). <i>f</i> : 40 kHz; UP: 160 W; S: n-hexane; L/S: 4 mLg ⁻¹ ; T: 60 °C; ED: 45 min	12 ± 1	The ratio had a dominant influence over the temperature in terms of oil yield. Positive interaction effect (Ratio × Temp).	[33]
Dried SCG PS: 0.70 mm	US bath Solvents: n-hexane & petroleum benzene S: n-hexane; L/S: 2 mLg ⁻¹ ; T: RT; ED: 45 min	13.99 (83.83% of 16.7)	No optimization of UAE conditions except the solvent type. Under mild conditions and shorter time, UAE achieves good yield.	[35]
Wet SCG (<i>C. Arabica</i> / <i>C. Robusta</i> blend) PS: ND	US probe: 1.92-cm diameter ultrasound horn probe L/S (1-2 mLg ⁻¹); ED(5-30 min) <i>f</i> :20 kHz ; UP:210 W; S:n-hexane; L/S: 2 mLg ⁻¹ ; T:30°C; ED:30 min	≈13	Methanol and hexane were used as first and second phase solvents. Ultrasound-assisted two-phase oil extraction extracts 98% of available oil in 30 min.	[47]
Dried SCG (<i>C. Arabica</i>)	US probe: Q500-20 probe equipped with 1'' diameter tip. Single parameter optimization: L/S (2.5-5 mLg ⁻¹); UP (100- 175 w); ED (15-35 min) <i>f</i> :20 kHz ; UP:150 W; S:n-hexane; L/S: 4 mLg ⁻¹ ; T:-; ED:30 min	14.52	N-hexane was determined to be the most effective solvent among chloroform & methanol. UAE diminishes extraction duration and improves oil yield. Direct ultrasonication is more effective than sonication bath (Reference: Rocha study).	[43]
Dried SCG PS: ND	US probe: Q500-20 probe equipped with 1'' diameter tip. 2 ³ factorial design (BBD): UP (100-200 W); L/S (15-30 mLg ⁻¹); ED (25-50 min). <i>f</i> :20 kHz ; UP:160 W; S:n-hexane; L/S: 30 mLg ⁻¹ ; ED: 34 min	Pre: 17.75 Exp: 17.23	N-hexane extracts more coffee oil compared to chloroform & methanol. 13.5 % higher yield offered by ultrasonic probe compared to the standard method. Positive influence of all process parameters.	[40]
<u>Supercritical Fluid Extraction (SFE)</u>				
Dried SCG PS: 0.297 mm	2 ³ factorial design (BBD): T (40-60 °C); P (175-225 bar); ED (60-180 min). S: CO ₂ , P:200 bar, T:50°C, ED:120 min	12.14	Positive influence of all optimized parameters. Pressure has the greatest effect among them.	[48]
Dried SCG PS: 0.70 mm	2 ³ factorial design (Taguchi): T (40-60 °C); P (200-300 bar); Co-solvent (Water, ethanol, hexane); Co-solvent amount (0-18 ml/100 g)	16.4	98.14% of oil content was obtained by SFE. As temperature increases, CO ₂ density decreases affecting extraction performance.	[35]

	S: CO ₂ -Ethanol; P:250 bar; T40°C, Ethanol: 18 ml/100 g.			
Dried SCG PS:ND	Single parameter optimization: T (313 – 328 K); P (15-30 MPa) S: CO ₂ P:25 MPa; T:323 K; ED:180 min	15.4	SFE extracted up to 85% of the total amount of oil of SCG.	[24]
<u>Microwave-Assisted Extraction (MAE)</u>				
Dried SCG (<i>C. Arabica</i> / <i>C. Robusta</i> blend) PS : 0.315-0.6 mm	Single parameter optimization: L/S (10, 15, 20) S: n-hexane; L/S: 15 mLg ⁻¹ ; T: RT; ED: 30 min	17.96	MAE was found to be the most effective tool in extracting oil from SCG, among other techniques (SOX, MAC, and UAE).	[34]
Dried SCG PS: ND	Single parameter optimization: L/S (4.16-6.66 mLg ⁻¹); T(69-95°C); ED (10-32.5 min) S: n-hexane; L/S: 6.66 mLg ⁻¹ ; T: 95°C; ED: 10 min	11.54	MAE exhibits about 24- fold lower extraction time comparing to SE method. MAE uses less solvent per gram of oil produced.	[49]
Dried SCG PS: 0.70 mm	Single parameter optimization: MP (200-800 W); S (n-hexane & petroleum benzene) S: n-hexane; L/S: 2 mLg ⁻¹ ; MP: 800 W; T: RT; ED: 10 min	13.8 (82.63% of 16.7)	Increasing microwave power led to a significant increase in oil yield. The microwave irradiation tool offers a good yield of oil from SCG.	[35]
Dried SCG (<i>C. Arabica</i> / <i>C. Robusta</i> blend) PS : 0.297 mm	US bath 2 ³ factorial design (CCRD): T (26-59 °C); L/S (1-24.82 mLg ⁻¹); ED (10.3-115.7 min). UP: 140 W; S: n-hexane; L/S:16 mLg ⁻¹ ; T: 29 °C; ED: 56.6 min.	11.89 ± 0.17	Extraction factors were found to have a positive effect on oil yield. The influence of process variables on oil yield can be ranked as follow: Ratio > Time > Temp. Favorable interaction of all optimized factors over oil yield.	Current study

PS: Particle size; **ND:** not defined; *C. Arabica*: Coffee Arabica; *f*: Ultrasonic frequency; **UP:** Ultrasonic power based on amplitude used for probe sonicator; **S:** Solvent; **L/S:** solvent to SCG waste ratio; **T:** temperature; **RT:** Room temperature; **ED:** Extraction duration; **MP:** Microwave power level; **K:** Kelvin; **MPa:** Megapascal; **Pre:** Predicted value; **Exp:** Experimental value; **SOX:** Soxhlet method; **MAC:** Maceration; **BBD:** Box–Behnken design; **CCRD:** Central composite rotatable design.