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ANALYSING THE EFFECT OF HEAT TRANSFER ON THE BIODIESEL PRODUCTION FROM SPENT COFFEE GROUNDS

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Abstract. *Coffee is one of the world's most popular beverages, and its industry generates vast amounts of trash that can be repurposed, amounting to around 5 million tonnes. The most tempting is Spent Coffee Grounds (SCG), which is created after the brewing process and is often thrown as garbage or sent to landfills without proper valorization, but it should be strictly avoided owing to its possible toxicity and organic nature. This trash contains 15 wt% oil, which was extracted and transesterified into biodiesel utilizing ethanol as an extraction solvent and methanol as a transesterification reactant. Because of its high acid value, coffee oil was extracted in a prototype Soxhlet extractor, followed by acid-catalyzed pretreatment. Basic-catalyzed transesterification was used to create biodiesel. This is a common biodiesel production process that involves a reaction between vegetable oil and primary alcohol in the presence of a catalyst to produce esters and glycerol. The oil extraction process was assessed in terms of solid-liquid extraction yield utilizing three distinct solvents with varied SCG particle sizes. Soxhlet extraction yielded a maximum SCG oil yield of 13.87 0.05% when diethyl ether was used, 14.23 0.67% when ethanol was used, and 12.92 0.24% when methanol was used. When the extraction procedure was scaled up, it produced 11.70 0.82% utilizing ethanol as a solvent. The maximum biodiesel yield was produced using a Methanol-to-oil molar ratio of 60:1, a reaction temperature of 60 °C, and 1.6% w/w KOH catalyst. Biodiesel derived from spent coffee grounds appears to be a fascinating and viable solution due to its enhanced heating value as well as low viscosity, density, and corrosion qualities. Furthermore, biodiesel is a non-toxic, biodegradable, and environmentally friendly fuel that contains no aromatics and nearly little sulfur, lessening its environmental impact.*

Keywords: *Biodiesel, Spent coffee grounds, Heat transfer, Extraction, Transesterification.*

1. INTRODUCTION

The production of renewable sources fuels is highly advantageous in terms of economic and environmental aspects. The increasing energy demand and environmental concerns encourage an increase in the consumption of cleaner and more sustainable energies, including biofuels such as biodiesel. Biodiesel has received particular attention because it can be prepared from a variety of vegetable oils and animal fats and as an efficient alternative to fossil fuels due to its renewability and very low greenhouse gas emissions (Caetano *et al*., 2012, Yaashikaa *et al*., 2022). Unlike fossil diesel fuel, biodiesel is a non-toxic, biodegradable, and environmentally friendly fuel that contains no aromatics and almost no sulfur, reducing its negative impacts on the environment (Cante *et al*., 2021; IEA, 2023).

International Energy Agency (IEA) Renewables (2023) indicated that during 2022 to 2027 the demand for vegetable oil, waste, and residue oils and fats increased by 56%, to 79 million tonnes in the production of biofuels such as biodiesel, renewable diesel, and biojet fuel. This high demand is related to GHG and feedstock policy objectives in the United States and Europe. In fact, waste and residues are expected to be used for 13% of biofuel production in 2027, up from 9% in 2021.

Factors such as environmental concerns, peak oil, energy security, fuel diversity, and sustainability drive the European biofuel market. According to the European Commission (2022) by 2030, the share of renewable energy in the EU's transport sector is expected to increase to at least 14%, including a minimum share of 3.5% for advanced biofuels, such as biodiesel. Coffee is one of the most consumed beverages in the world and is the second most globally traded commodity. Coffee is made by grinding and hot water extraction of roasted coffee beans. The International Coffee Organization (2023) has reported a projected to grow of 3.3% in world coffee consumption reaching approximately 10 billion tonnes in 2021/22.

The preparation of coffee beverages produces a solid residue known as spent coffee grounds (SCG), which accounts for the majority of byproducts (55 to 67%), reported by Ha *et al*. (2019). SCG is generally discarded as waste directly sent to landfills without proper valorization (Scalia *et al*., 2021; Sermyagina *et al*., 2021). The disposal of SCG causes rather serious environmental problems. The SCG has high organic content and the presence of compounds such as caffeine and polyphenols have negative effects. Furthermore, coffee has a significant impact on climate change, one kilogram of coffee corresponds to 16.50 kg carbon dioxide equivalent (CO2eq) which is a measurement of greenhouse gas (GHG) (Mitraka *et al*., 2021).

SCG has a high potential value to be involved in every process and obtain a wide range of products, the reuse of SCG is becoming an important social and environmental issue (Tuntiwiwattanapun *et al*., 2017, Kookos, 2018). This waste is an appropriate resource for garden fertilizer, feedstock for bioethanol, biodiesel, biogas, briquettes, pellets, and bio-oil production (Carlucci *et al*., 2017). In particular, the use of SCGs has lately received interest as a viable source for biodiesel synthesis due to their high oil content in the order of 10–20wt% and their high calorific value of about 24.9 MJ/kg (dry weight) (Mueanmas *et al*., 2019, Sugebo, 2021).

Transesterification reaction has become a popular process to produce biodiesel between vegetable oil (triglycerides) and a primary alcohol (ethanol or methanol) and has been traditionally catalyzed by alkali in a homogeneous reaction, to obtain esters and glycerol (Brahma *et al*., 2022; Thangaraj *et al*., 2019). Factors such as extraction and reaction temperature, the catalyst, type of alcohol and oil, the molar ratio of alcohol to oil, reaction time and stirring during the reaction influence the efficiency of biodiesel production (Ahmed *et al*., 2022; Supang *et al*., 2022). Significant quantities of free fatty acid (FFA) and water, make some materials unsuitable for existing homogeneous alkaline-catalyzed process. Neutralization of FFAs can be carried out by the addition of excess alkali, but it will cause saponification during the transesterification reaction and will reduce the yield and the quality of biodiesel (Atelge, 2022).

Methanol has higher purity is high and lower cost when compared to other alcohols and therefore its more common use. However, methanol is synthesized from fossil fuels, leading to the search for other greener alcohols, such as ethanol. Ethanol is synthesized from a renewable source and causes a less inhibitory effect on lipase compared to methanol (Norjannah *et al.*, 2016).

2. MATERIALS AND METHODS

2.1 Samples

The Spent coffee grounds samples were obtained in a local coffee (*Pão Prosa* bakery) near to the Pontifical Catholic University of Paraná (PUC-PR) in the city of Curitiba, Brazil. The waste material was dried in an air circulation dryer at 60°C for 30h until constant moisture. Moisture content on a dry basis was determined before and after the drying process by a halogen moisture analyzer (Mettler-Toledo, model HE53/01). The average particle size of the dried material was estimated using Tyler series sieves in a vertical vibratory sieve shaker, in which each sieve size was mesh 32 (500 µm), mesh 35 (425 µm), mesh 60 (250 µm), mesh 65 (212 µm), mesh 80 (180 µm), and mesh 100 (150 µm). A small portion of each sieved fraction was separated, packed in plastic bags, and stored until their use for further laboratory-scale solvent extraction. The rest was blended and packed in plastic bags and stored until their use for pilot-scale solvent extraction.

2.2 Soxhlet Extraction of Oil

The extracted oil was determined by gravimetric analysis using three different solvents: diethyl ether (98% v/v), methanol (99.8% v/v), and anhydrous ethanol (99.5% v/v). Additionally, based on the quantity of material held in each sieve and the overall average particle size, three different sample sizes were assessed: SCG with an average size of 604, 463, and 338 µm which corresponds to dried material retained at mesh 32, 35, and 60, respectively. The samples were packed in a cellulose cartridge containing approximately 5g of dry spent coffee grounds. Each extraction was performed for 6 h in a Soxhlet apparatus with approximately 200 ml of solvent. After the extraction process, the solvent was evaporated. The oil sample was further dried at 100°C for 2 h, to remove any residual solvent. The experiments were performed in triplicate. The oil yield extracted from the spent coffee grounds samples was calculated based on the dry weight of the sample according to

$$Oil\ yield\ (wt\ \%) = \frac{w_{oil}\ (g)}{w_{SCG}\ (g)} \times 100 \quad (1)$$

where w_{oil} and w_{SCG} oil is the mass of oil after the extraction process and the mass of the dried SCG, respectively.

To achieve move SCG oil a scaled-up extraction process is required, therefore, a pilot-scale Soxhlet extractor was used. Approximately 1.1 to 1.3 kg of dried SCG wrapped in filter cloth was placed in the extractor along with 7l of anhydrous ethanol (99.5% v/v) for the extraction process. Based on the laboratory scale oil yield results, exclusively anhydrous ethanol was used as a solvent. The resulting liquid was then subjected to rotary vacuum evaporation to remove the ethanol. The oil sample was dried at 80 °C for 2 h, to remove residual ethanol. The oil yield was determined according to Eq. (1).

2.3 Esterification and Transesterification

2.3.1 Free Fatty Acid Content

The acid value and FFA (free fatty acid) content of the oil, pre-treated oil, and biodiesel samples were determined through the gravimetric method of titration with phenolphthalein as an indicator. To enhance the method's accuracy, which relies on identifying the exact point of color change and, consequently, ensuring the reliability of the obtained results, pH measurement was employed to identify the equivalence point. The acid values were determined by

$$Acid\ value\ \left(\frac{mg_{KOH}}{g}\right) = 56.1\,\frac{C_{KOH}V_{KOH}}{w_{sample}} \quad (2)$$

where C_{KOH} is the titrant concentration, V_{KOH} is volume of titrant consumed to neutralization and w_{sample} is the weight of the sample. From the acid value, the FFA content is calculated by

$$FFA\ (wt\ \%) = Acid\ value \times \frac{M_{oleic\ acid}}{56.1} \times \frac{100}{1000} \quad (3)$$

where FFA is expressed in terms of Oleic acid, where $M_{oleic\ acid}$ is its molecular weight (282.5 g/mol).

2.3.2 Acid-Catalyzed Pretreatment

The extracted SCG oil (approximately 40g for each sample) had been previously homogenized and preheated. The reactant methanol (99.8% v/v) was added also in the presence of sulfuric acid. The alcohol-FFA molar ratio varied from 8.5:1 to 14.3:1 and catalyst-to-FFA weight percentages varied from 5-11.7%. The mixture was stirred for at least 2 h after the start of the reaction, a duration that was found to be sufficient in previous studies (Mueanmas *et al*., 2019). The samples were neutralized with potassium hydroxide (85% w/w) and prepared for base-catalyzed transesterification.

2.3.3 Base-Catalyzed Transesterification

In this step of the process, the samples of pre-treated oil were mixed with methanol in excess to complete the required alcohol-to-oil molar ratio in the presence of potassium hydroxide (85% w/w). Potassium hydroxide was selected based on the results reported in previous studies (Al-Hamamre *et al*., 2012; Kondamudi *et al*., 2008) that examined the base-catalyzed transesterification of SCG oil, in which FAME conversion yields of 99 to 100% w/w were achieved with a KOH catalyst. The experiments were conducted at a temperature of 55 and 60 °C for 4 h while the reaction mixture was constantly stirred. The alcohol-to-oil molar ratio and catalyst-to- oil weight percentages were 60:1 and catalyst-to-oil weight percentages varied from 1 to 2%.

The mixture was subjected to rotary vacuum evaporation to remove the methanol. The final reaction products were allowed to settle in a separation funnel overnight the upper biodiesel phase was separated from the lower glycerol phase. The resulting ester phase was successively and carefully washed with warm (50 °C) distilled water to remove the basic catalyst, the salt potassium sulfate (K_2SO_4) product from the neutralization, and any soap formed during the transesterification. The washing process also served to remove any residual glycerol, methanol, and other spent coffee grounds components. After that, the FAMEs (Fatty acid methyl esters) were subjected to thermal heating at 100 °C to remove residual water. Finally, a filtration process with a cellulose filter (J Prolab, 14 μm) was performed to remove solid traces. The % w/w FAME reaction yield relative to SCG oil was calculated by,

$$FAME\ yield\ (\%) = \frac{w_{FAME,exp}\ (g)}{w_{FAME,teo}\ (g)} \times 100 \quad (4)$$

where $w_{FAME,exp}$ and $w_{FAME,teo}$ oil is the experimental mass of biodiesel and theoretical mass of biodiesel, respectively. The theoretical mass of biodiesel is defined as the ideal quantity of biodiesel that can be obtained, *i.e.*, all w_{oil} is ether esterified or transesterified and the conversion in both reactions is 100%.

$$w_{FAME,teo}\ (g) = M_{Biodiesel} w_{oil} \left[\frac{FFA}{M_{FFA}} + \frac{3(1-FFA)}{M_{Lipids}}\right] \quad (5)$$

where $M_{Biodiesel}$, M_{FFA} and M_{Lipids} are the average molar weight of biodiesel (287,01 g/mol), FFA (272,98 g/mol) and lipids (856,99 g/mol), respectively. These values were based on quantification of the fatty acids distribution in SCG oil according to Williamson *et al*. (2022).

3. RESULTS AND DISCUSSION

3.1 Sample Preparation

The initial moisture of the collected samples was found to be 55.71 ± 0.65%. After undergoing the drying process, the final moisture content decreased to 2.73 ± 0.56%. The particle size distribution of each material fraction retained in the sieves is shown in Figure 1. The weight fractions retained in each sieve were as follows: 604 µm (36.95 ± 1.40%), 463 µm (19.49 ± 4.34%), 338 µm (38.05 ± 2.70%), 231 µm (3.41 ± 1.43%), 196 µm (0.98 ± 0.74%), 165 µm (0.95 ± 0.72%), and 75 µm (0.18 ± 0.16%). On average, the particle size is 453.07 ± 2.46 µm, the vast majority of the material (approximately 95%) is larger than mesh size 60 (250 µm). This decision to select sample sizes for the laboratory-scale extractions is justified by the distribution of particle sizes obtained.

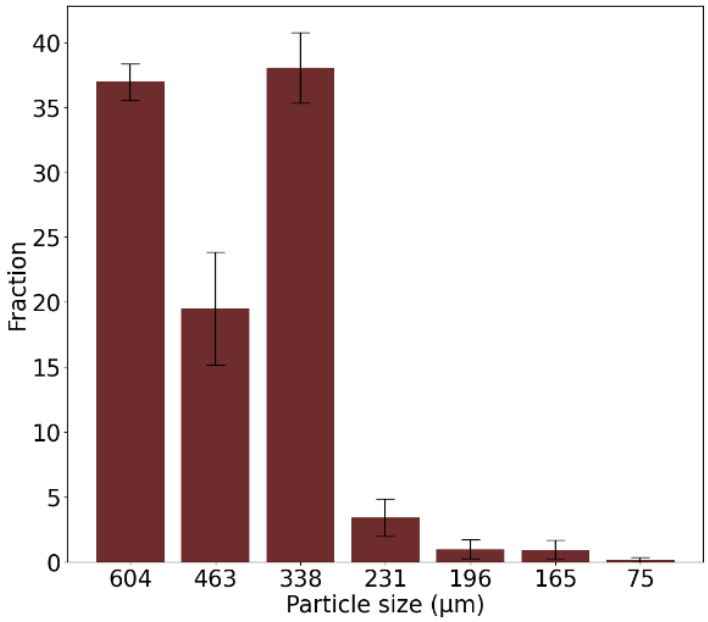

Figure 1: Spent coffee grounds particle size distribution.

3.2 Soxhlet Extraction

The impact of SCGs (spent coffee grounds) particle size on oil extraction yield is depicted in Figure 2. The results clearly demonstrate that the particle size has a significant influence on the amount of oil extracted. Smaller particles offer a larger surface area, allowing the solvent to effectively permeate and penetrate the pores of SCGs, leading to higher oil removal. Specifically, among the solvents tested, ethanol exhibited the highest oil yield for the smallest particle size,

while diethyl ether showed the highest yield for the two larger particle sizes, although only slightly higher than ethanol. Oppositely, methanol resulted in the lowest oil yield across all particle sizes.

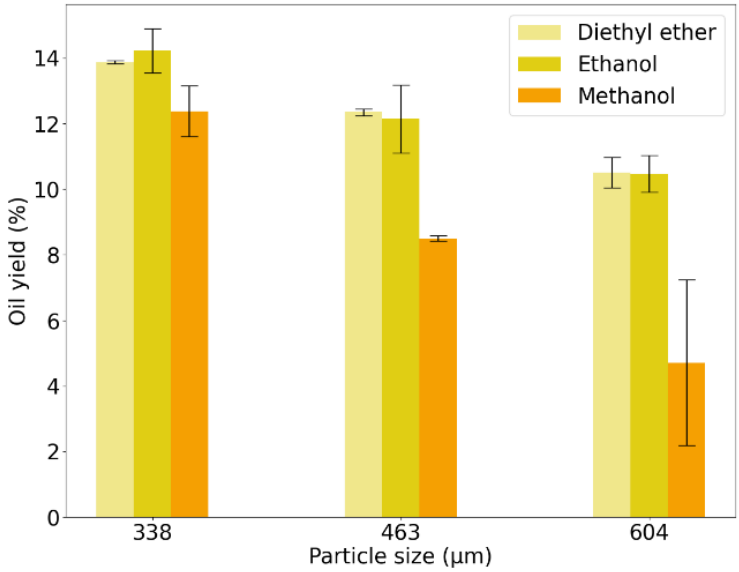

Figure 2: Soxhlet extraction with various solvents and particle sizes.

The yield of lipid extraction was influenced by the properties of the solvents used. The relevant details of the three solvents, including their chemical formula, molecular weight, boiling point, dielectric constant, and dipole moment, are presented in Table 1. The comparable yields obtained from diethyl ether and ethanol extractions can potentially be attributed to the extraction temperature. Since the boiling point of ethanol is more than double that of diethyl ether, higher temperatures are advantageous for the extraction process. However, it should be noted that due to the alcohol's polarity, certain substances, such as melanoidins responsible for the characteristic "brown" color of roasted coffee, are also extracted. This was observed by the color of the extracted solutions: light yellow (diethyl ether), gold yellow (ethanol), and orange (methanol).

Table 1: Chemical formulas and properties of the solvents used in the Soxhlet extractions.

Solvent	Diethyl ether	Ethanol	Methanol
Chemical formula	$(C_2H_5)_2O$	C_2H_5OH	CH_3OH
Molecular weight (g/mol)	74.123	46.069	32.042
Boiling point (°C)	34.44	78.65	64.54
Dielectric constant	4.33	24.26	32.7
Dipole moment (D)	1.15	1.69	1.69
Polarity	Non-polar	Polar	Polar

The ethanol extraction process, when scaled up, resulted in an oil yield of 11.70 ± 0.82%. This value was comparable with the results obtained on the laboratory scale, considering that the entire particle size distribution was included. Due to the larger quantity of oil obtained, its true appearance was that of a viscous, dark brown liquid. Analysis of the acidity through titration revealed an acid value of 61.17 ± 2.12 mg_{KOH}/g_{oil}, corresponding to a free fatty acid (FFA) content of 30.80 ± 1.07% w/w. The relatively high acid value obtained, also reported by Efthymiopoulos *et al*. (2019) and can likely be attributed to the extraction temperature, which was close to the boiling point of solvent, ethanol. The high FFA content indicates a potential issue for direct basic transesterification, as the FFA can react with the basic catalyst to form soap, thereby reducing the biodiesel yield.

3.3 Esterification and Transesterification

The acid-catalyzed pretreatment esterifies the FFA and lowers its content. During the reaction, especially in cases where the reaction temperature is higher than 50 °C or the reaction time longer than 3 hours the dark brown color of the

SCG oil changed to dark green, and the formation of a blue-green-colored solid precipitate. Similar findings were reported by Jenkins et al. (2014), who observed the formation of blue-green unsaponifiable precipitates during an esterification reaction involving an acid catalyst. These precipitates are derived from a range of compounds, including sterols, terpenes (cafestol and kahweol, specifically found in coffee), and organic acids. Therefore, the successful biodiesel production from the SCG oil requires taking to account the presence of the unsaponifiable matter. However, it does not explain the lower yield obtained shown in Table 2 as these components are found in very small quantities in spent coffee grounds.

The transesterification is what converts triglycerides (lipids) into ester (biodiesel) with the presence of a basic catalyst. The highest yield of biodiesel obtained was 44.24 % in experiment 2, in which the Methanol-to-oil molar ratio was 60:1, reaction temperature 60°C and 1.6% w/w of catalyst. It was also observed the solidification of the resulting transesterification reaction during experiment 3. Clear evidence of the formation of too many soap, despite practically the same reaction conditions (Table 2) of the highest yielded experiment. The indication of the soap formation could be seen as the dark brown color changed to a dark red color during the transesterification reaction.

Table 2: Experimental results of biodiesel production from spent coffee grounds under different conditions.

Experiment	Acid pre-treatment (esterification)				Transesterification[(1)]			Biodiesel Yield (%)
	Methanol-to-FFA molar ratio	H_2SO_4 (%)	T(°C)	Time (h)	Methanol-to-Oil molar ratio	KOH (%)	T(°C)	
1	11.7	5.0	60	4	60	2.0	60	34.05
2	8.5	5.0	50	2	60	1.6	55	44.24
3	14.3	11.7	55	3	60	1.6	56	- [(2)]
4	11.5	6.8	50	2.5	70	1.1	55	31.94
5	9.3	7.8	45	2	60	1.3	55	23.45

[(1)]Reaction time of 4h; [(2)]Final product solidified.

One of the possible reasons why the yields were low is that, after the completion of the basic transesterification followed by the decantation process, the upper and lower phases are troublesome to identify due to the dark brown appearance of the SCG oil. Moreover, the washing process had to be performed very carefully given that the resulting reaction mixture when mixed with warm water was readily emulsified. Nonetheless, the biodiesel that was produced had a traditional yellow color and exhibited a viscosity that was higher than that of water, yet lower than the viscosity of the extracted oil obtained from the spent coffee grounds.

4. CONCLUSIONS

The research looked at how different solvents and particle sizes of discarded coffee grounds (SCG) affected the extraction process. The information gathered in this study will be critical in determining the viability of using SCG as a valuable source of chemicals with high potential for biodiesel synthesis. The results showed that utilizing ethanol as the solvent and an average particle size of 338 m resulted in the maximum oil output, reaching 14.23 ± 0.67%. When the process was scaled up, the yield dropped to 11.70 ± 0.82%. The greatest biodiesel yield obtained was 44.24%. Because this is early work, the biodiesel yields are still low. The SCG processing to the final biodiesel output can be a difficult process. Although this research is still in its early stages, the findings suggest that oil obtained from spent coffee grounds (SCG) has potential as a non-edible feedstock for biodiesel synthesis. Future research will focus on increasing biodiesel yield by experimenting with different operating conditions, such as acid pretreatment and basic transesterification, while taking temperature, alcohol-to-oil ratio, and catalyst weight percentage into account. Another important goal of this research is to assess the quality of the biodiesel produced, allowing for later tests involving its combustion in a diesel engine.

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