

# ***BIODIESEL TRANSESTERIFICATION OF SPENT COFFEE GROUNDS USING DBU AS A CATALYST***

## **Using DBU to Make Biodiesel from Coffee Grounds**

### **Abstract**

Coffee is one of the most universally consumed beverages. The world produces 60 million tons of spent coffee grounds (SCGs) as waste per year. Biodiesel made from SCGs is an eco-friendly alternative fuel but proves difficult to produce. 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) was used as both a solvent and a catalyst with methanol to determine the efficiency of biodiesel production from SCGs. The process was completed by extracting coffee oil from acid-washed spent coffee grounds (AWSCGs) using hexane. The two extraction methods that were explored were Soxhlet and agitation. DBU is an interesting choice as a solvent and a catalyst because of its ability to change polarities when exposed to carbon dioxide or nitrogen. However, it was shown through experimentation that the product had large crystals and copious amounts of glycerol while producing negligible amounts of biodiesel. Both AWSCGs and regular SCGs were tested along with variations of DBU and methanol volumes, but the results show an insufficient yield in each case. From this, it can be concluded that using DBU as a solvent and catalyst with methanol and coffee oil for production of biodiesel is ineffective. Future experiments to convert SCGs into biodiesel should look for other catalysts and ways to optimize reaction conditions for biodiesel production.

### **Keywords**

biodiesel, biofuel, coffee grounds, renewables, DBU, sustainability

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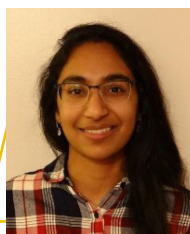
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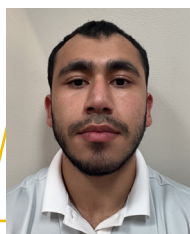
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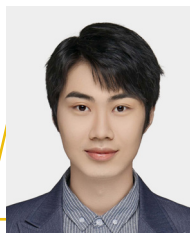
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## Mentor



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## INTRODUCTION

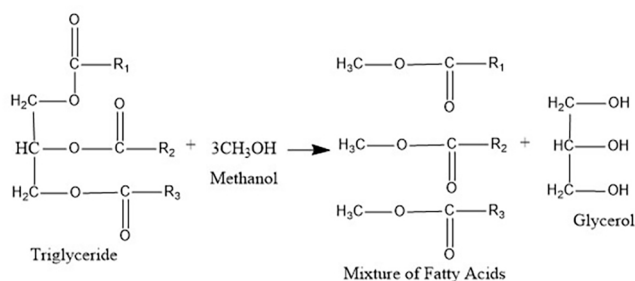
Spent coffee grounds (SCGs) are being explored as a source of biodiesel because they are cheap, ecologically friendly, and have relatively high oil content—10–20% (Leal Vieira Cubas et al., 2020). The world creates “60 million tons” of SCGs per year, so their use in biodiesel production presents an opportunity to recycle into fuel what has historically been waste (Forcina et al., 2023). A major challenge to the commercial favorability of biodiesel production is the need to redirect agricultural resources to be sources of biomass, resulting in a general loss in revenue and food resources to be used as biomass for biodiesel. SCGs could help solve this issue, as they can be used as a low-cost alternative to other biomass sources, therefore improving the economic viability of the process.

In the search for an environmentally safe and economically viable catalyst for the production of biodiesel, 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) has been suggested for its potential to be recycled as well as its dual function as a catalyst and solvent in the transesterification reaction. The polarity of DBU is reversible when exposed to CO<sub>2</sub>, allowing for easy extraction of the nonpolar biodiesel product from the solvent (Nyugen et al., 2020).

This project set out to find the optimal procedure and conditions for the transesterification of SCGs and SCG oil into biodiesel. Previous studies have found that the direct transesterification of SCGs is viable and efficient (Nyugen et al., 2020), but these results have been impossible to replicate. Therefore, the extraction of coffee oil, as well as its use as the biomass source for transesterification, was investigated in this report.

The experiments done for this project used a transesterification reaction initially with SCGs and ethanol as the reactants, later using extracted coffee oil and methanol, and (DBU) as the catalyst and solvent as displayed in Figure 1 (ETIP Bioenergy, 2022).

The purpose of this project was to explore the effectiveness of the transesterification reaction and DBU as a solvent and catalyst for use with SCGs. This project expands on previous research (ETIP Bioenergy, 2022) and looks to optimize the reactions by using different amounts of catalysts and methanol. With the optimized



**FIGURE 1.** Transesterification process for biodiesel production (ETIP Bioenergy, 2022).

reaction, the project plans to look at economic feasibility to analyze whether it is worth pursuing this reaction without a way to clean the DBU once the reaction is completed. As with previous experiments, acid-washed spent coffee grounds (AWSCGs) were used to produce the coffee oil, and gas chromatography–mass spectrometry (GC-MS) was used to determine the fatty acid methyl ester concentrations with a focus on the following compounds: palmitic, stearic, oleic, linoleic, arachidic acid, and long-chain carbon molecules.

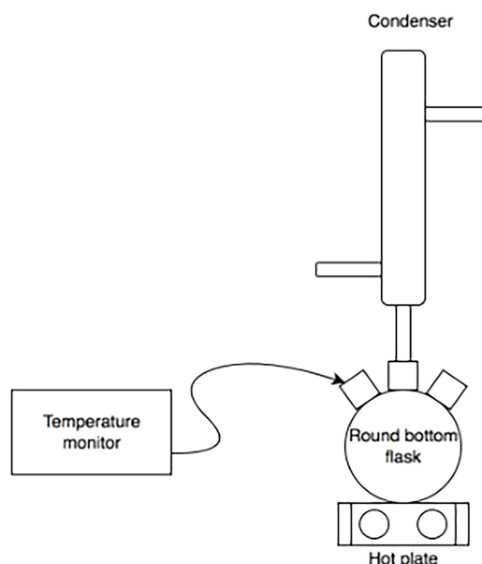
## EQUIPMENT AND MATERIALS

### Material

The SCGs used in this experiment were collected from Vienna Espresso in West Lafayette, Indiana. Hydrochloric acid solution was used for acid wash. Hexane was utilized for coffee oil extraction. The direct transesterification reaction was carried out using methanol as the alcohol reactant and 99% DBU as the catalyst and solvent. Carbon dioxide gas was used to polarize DBU.

### Equipment and Overall Procedure

To dry the SCGs, an oven was used at 155°F. An agitator was operated at 70°C for two hours to produce the AWSCGs. To extract coffee oil, a shaker oven was used under 300 rpm and 35°C for two hours. A rotary evaporator was used to evaporate the hexane from the coffee oil. To perform the transesterification reaction, a 300-mL 3-neck round bottom flask, a condenser, a temperature controller, and a hot plate were required, configured as illustrated in Figure 2. Lastly, a centrifuge machine was used to separate the DBU from the



**FIGURE 2.** Schematic of experimental setup for direct transesterification of SCGs or coffee oil.

biodiesel. To analyze the component of the final product, a GC analysis was performed.

## PROCEDURE

The team collected spent coffee grounds (SCGs) from the designated coffee shop in the campus area. The SCGs were dried in an oven at 155°F for 24 hours to ensure sufficient drying. The team conducted an acid wash on the dried SCGs to eliminate the free fatty acids that favor the saponification reaction. This method took inspiration from the work done by Liu et al. (2017). However, hydrochloric acid was used instead of sulfuric acid because it was safer for the team to handle. The procedure included mixing the dried SCGs and 0.257 M HCl solution at a ratio of 0.243 g dried SCGs per mL HCl solution. With that ratio, each batch reactor contained 80 g of dried SCGs, 169 mL of HCl, and 160 mL of reverse osmosis water. The acid wash requires a 500-mL tube reactor with an impeller, a temperature probe, and a condenser. The batch reactor operated for 2 hours at 70°C. The product was then filtered to gather the wet acid-washed spent coffee grounds (AWSCGs). The AWSCGs were dried in the oven for 24 hours at 155°F and stored in the fridge.

For the coffee oil extraction from the dry AWSCGs, the team considered Soxhlet extraction and agitator

extractions. Soxhlet extraction was used at the beginning of the experiments, and then the agitator method was devised to have a shorter extraction time and a similar yield.

The Soxhlet setup uses 60 grams of dry AWSCGs and 350 mL of hexane to extract the coffee oil. The Soxhlet extraction can be run for as long as possible, but the yield is maximized at 4 hours. The 60 grams of AWSCGs are placed in a thimble inside the Soxhlet extractor. The Soxhlet extractor is placed on top of a 500 mL 3-neck round bottom flask with the 350 mL of hexane and the temperature probe. The other two necks are plugged and the condenser is placed on top of the Soxhlet extractor. The temperature is set to 70°C and the hexane is allowed to boil into the extractor. The hexane then extracts the coffee oil and mixes into the 500 mL round bottom flask, and the solution is separated using a rotary evaporator.

To extract coffee oil from the dry AWSCGs with the agitator, 20 grams of AWSCGs and 84 mL of hexane were placed in a 250 mL flask and agitated for 2 hours at 35°C (Gagliardi et al., 2021). The hexane-coffee oil mixture was then separated using a rotary evaporator for approximately two hours using a 38°C water bath, and the remaining coffee oil was collected and stored in a smaller flask.

The transesterification reaction equipment includes a 300-mL 3-neck round bottom flask, a temperature controller, a heating mantle, a hot plate, and a stir bar. The amount of DBU and methanol used was varied during the experiments to see which ratio would produce the highest percent yield. The reaction vessel was heated to 70°C and allowed to react for 30 minutes. The solution was then allowed to cool to room temperature (around 25°C) before it was separated using a rotary evaporator. Using the rotary evaporator, the methanol was removed from the solution at a temperature of 40°C. The sample was heated to about 30°C under a vacuum so the methanol would evaporate while leaving the reaction product as a liquid. Carbon dioxide was bubbled through this product to polarize the DBU for at least 30 minutes or until the sample was noticeably more viscous. The polarized sample was centrifuged for 30 minutes at 5,000 rpm to separate the biodiesel, DBU, and a solid material. Finally, the biodiesel layer was extracted via a pipette from the final sample and weighed for yield. To further

analyze the resulting biodiesel, the team ran a GC-MS analysis to determine the component of the fatty acids in the biodiesel, as well as their percentages. The team also performed three other transesterification reactions using different reactants. Coffee oil was switched to either AWSCG or SCG, and methanol was changed to ethanol.

At the beginning of the semester, the SDS and lab safety protocols, such as the Chemical Hygiene Plan (CHP), were reviewed by the researchers. This was an important step because of the toxicity of DBU, the primary solvent for the reaction. Gloves were always replaced if DBU got onto them. DBU and solutions containing DBU were stored in sealed containers under the fume hood at all times. Chemicals were disposed of in the proper channels according to lab protocols.

## RESULTS

Four experiments were run testing the use of regular SCGs directly using the tube reaction vessel with an impeller. These experiments didn't produce any biodiesel. Then two experiments using AWSCGs directly were conducted in the 300-mL 3-neck round bottom flask, which also didn't produce biodiesel. The data from these trials can be seen in Table 1.

Four more experiments were performed using oil obtained from the AWSCGs. These experiments were run using the 300-mL 3-neck round bottom flask, and the results shown in Table 2 were obtained.

Unfortunately, there was insufficient data for JMP to perform a confidence interval analysis on the results obtained. However, from these results, the following model was able to be obtained from JMP to quantify the factors affecting percent yield of biodiesel:

$$\begin{aligned} \% \text{ Yield} = & 36.795 - 4.385 * \left( \frac{\text{DBU Volume} - 14}{2} \right) \\ & - 7.425 * \left( \frac{\text{MeOH Volume} - 10}{2} \right) \\ & + 0.885 * \left( \frac{\text{DBU Volume} - 14}{2} \right) * \left( \frac{\text{MeOH Volume} - 10}{2} \right) \end{aligned} \quad (1)$$

The samples referenced in Table 2 were not able to be analyzed using GC-MS. To find the ratio of DBU to methanol that produced the most yield, 16 experiments were run using acid-washed oil as the feedstock. Only five

**TABLE 1.** Data of all experiments conducted to compare SCGs, AWSCGs, and AW oil.

Experiment #	Temperature (C)	Mass SCG	mL Methanol	mL DBU	rxn time (hr)	CO2 time (hr)
1 (SCGs)	70	12	72	144	0.5	0.5
2 (SCGs)	70	12	72	144	0.5	0.75
3 (SCGs)	70	10	80	140	0.5	1
4 (SCGs)	70	10	100	160	0.5	1
5 (AWSCGs)	70	5	30	60	0.5	0.5
6 (AWSCGs)	70	5	30	60	0.5	0.75
7 (AW Oil)	70	4.22	34	51	0.5	0.5
8 (AW Oil)	70	4.63	56	56	0.5	0.5
9 (AW Oil)	70	4.76	38	76	0.5	0.5
10 (AW Oil)	70	5.22	63	84	0.5	0.5

experiments created an extractable amount of biodiesel. The full list of experiments can be seen in Table 3.

Table 4 shows the calculated yields for the experiments that produced biodiesel, and Table 5 shows the GC-MS results for these samples.

**TABLE 2.** Percent yield of biodiesel.

Volume of Methanol (mL/g of coffee oil)	Volume of DBU (mL/g of coffee oil)	Percent Yield (% by mass)
8	12	49.49
8	16	38.95
12	12	32.87
12	16	25.87

There was insufficient data for JMP to perform a full confidence interval analysis on the results obtained in Table 4. However, it was found that the amount of DBU present was significant with 90% confidence in determining biodiesel yield. From these results, the following model was able to be obtained from JMP to quantify the percent yield of biodiesel by mass:

$$\% \text{ Yield} = -63.9 - 0.355 * (\text{Methanol (mL)}) + 1.345 * (\text{DBU (mL)}) \quad (2)$$

Unfortunately the internal standard peak was not given, so a percent yield could not be found using the GC-MS results in Table 5.

As stated above, GC-MS was used to gather the results. The GC-MS provided the areas of each of the different molecules in what was gathered from the biodiesel layer

**TABLE 3.** Data of all experiments conducted to find optimal ratio.

Experiment #	Oil (g)	DBU (mL)	MeOH (mL)	Temperature (C)	Rxn Time (hr)	CO2 Time (hr)	Centrifuge Time (hr)	Product weight (g)	Yield (mass %)
1	5	60	30	70	0.5	0.5	0.5	0	0
2	5	60	40	70	0.5	0.5	0.5	0.2	3.994
3	5	60	50	70	0.5	0.5	0.5	0	0
4	5	60	60	70	0.5	0.5	0.5	0	0
5	5	70	30	70	0.5	0.5	0.5	0.785	15.692
6	5	70	40	70	0.5	0.5	0.5	1.056	21.114
7	5	70	50	70	0.5	0.5	0.5	0.328	8.585
8	5	70	60	70	0.5	0.5	0.5	N/A	N/A
9	5	80	30	70	0.5	0.5	0.5	0	0

**TABLE 4.** Mass percent yield of biodiesel.

Sample Number	Volume of Methanol (mL)	Volume of DBU (mL)	Mass Percent Yield (%)
1	40	60	3.994
2	30	70	15.692
3	40	70	21.114
4	50	70	8.585
5	40	80	30.9

from the centrifuge. These areas were then added up and each area was divided by that total area to find the amount of each acid in the final sample, as seen in Table 7. This helps find the true yield as the yield calculated with the mass includes non-biodiesel molecules, such as cosanes. The ester content was calculated within the biodiesel sample—and the conversion efficiency of each experiment—by using the GC-MS results and the internal standard to find the ester content, which is shown in Table 7. From there, the ester content was multiplied by the mass percent yield to get a conversion efficiency for each experiment. These calculations were

**TABLE 5.** Biodiesel composition characterization by GC-MS.

Sample Number	Palmitic (% of total)	Stearic (% of total)	Oleic (% of total)	Linoleic (% of total)	Arachidic (% of total)
1	32.74	39.93	12.28	5.63	2.17
2	28.53	24.76	5.55	4.94	2.04
3	39.26	34.53	8.62	5.97	2.34
4	35.16	28.70	7.86	6.28	2.69
5	19.25	16.10	4.13	3.55	1.61

**TABLE 6.** Data of all experiments conducted to find optimal ratio.

Experiment #	Oil (g)	DBU (mL)	MeOH (mL)	Temperature (C)	Rxn Time (hr)	CO2 Time (hr)	Centrifuge Time (hr)	Product weight (g)	Yield (mass %)
1	5	60	30	70	0.5	0.5	0.5	0	0
2	5	60	40	70	0.5	0.5	0.5	0.2	3.994
3	5	60	50	70	0.5	0.5	0.5	0	0
4	5	60	60	70	0.5	0.5	0.5	0	0
5	5	70	30	70	0.5	0.5	0.5	0.785	15.692
6	5	70	40	70	0.5	0.5	0.5	1.056	21.114
7	5	70	50	70	0.5	0.5	0.5	0.328	8.585
8	5	70	60	70	0.5	0.5	0.5	N/A	N/A
9	5	80	30	70	0.5	0.5	0.5	0	0

**TABLE 7.** Ester content and conversion efficiency.

Sample Number	Volume of Methanol (mL)	Volume of DBU (mL)	Ester content (%)	Conversion Efficiency (%)
1	40	60	26.084	1.042
2	30	70	11.434	1.794
3	40	70	6.242	1.317
4	50	70	41.715	3.581
5	40	80	10.725	3.314

**TABLE 8.** Biodiesel composition characterization by GC-MS.

Sample Number	Palmitic (% of total)	Stearic (% of total)	Oleic (% of total)	Linoleic (% of total)	Arachidic (% of total)	Yield (%)
1	42.590	18.140	2.229	18.084	11.794	1.68
2	37.144	14.768	0.940	16.086	21.353	2.82
3	34.819	12.789	0.880	15.418	20.019	2.12
4	38.070	18.251	0.820	17.008	18.035	5.26
5	30.347	20.106	1.347	19.616	15.947	7.18

**TABLE 9.** Composition of high molecular weight FAMES in different samples.

Sample Number	325.3 (% of total)	368.4 (% of total)	339.4 (% of total)	382.4 (% of total)	396.5 (% of total)	441.4 (% of total)
1	0.249	0.641	0.764	2.167	0.391	2.949
2	0.643	1.597	1.781	4.764	0.789	0.132
3	0.574	1.417	1.536	4.147	0.668	7.730
4	0.436	1.080	1.244	3.345	0.540	1.174
5	1.905	1.791	2.321	5.702	0.778	0.139

modeled from a method found on ResearchGate (Suraj, sk, 2018). The samples shown in Table 4 and Table 5 are different from the samples in Tables 2 and 3.

Again, there was insufficient data for JMP to perform a confidence interval analysis on the results obtained. However, from these results, the following model was able to be obtained from JMP to quantify the percent yield of biodiesel based on GC-MS:

$$\% \text{ Yield} = -20.318 + 0.122 * (\text{Methanol (mL)}) + 0.275 * (\text{DBU (mL)}) \quad (3)$$

However, this model does not have significant factors with 90% confidence. As seen in Table 5, the sample that gave the highest yield was sample 5. This experiment used a ratio of 80 mL DBU and 40 mL methanol with 5 g of coffee oil made from acid-washed coffee grounds.

From Table 9, it can be seen that other fatty acid methyl-esters (FAMES) were present in the samples—the specific FAMES have not been identified, but the numbers denote their peak numbers from the GC-MS analysis. It can be seen that although the percentage yields are significantly smaller compared to the composition of known fatty acids shown in Table 4, numerous FAMES are produced.

Figure 3 shows one reaction product that was contaminated by these FAMES. These FAMES made the process of recovering biodiesel from the reaction product difficult, as the crystallization covered some of the biodiesel present. Further results are shown in Tables 10 and 11.

**FIGURE 3.** Example of one of the crystallized samples.



**TABLE 10.** Data of all experiments conducted to compare SCGs, AWSCGs, and AW oil.

Experiment #	Temperature (C)	Mass SCG	mL Methanol	mL DBU	rxn time (hr)	CO2 time (hr)
1 (SCGs)	70	12	72	144	0.5	0.5
2 (SCGs)	70	12	72	144	0.5	0.75
3 (SCGs)	70	10	80	140	0.5	1
4 (SCGs)	70	10	100	160	0.5	1
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6 (AWSCGs)	70	5	30	60	0.5	0.75
7 (AW Oil)	70	4.22	34	51	0.5	0.5
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10 (AW Oil)	70	5.22	63	84	0.5	0.5

**TABLE 11.** Data of all experiments conducted to find optimal ratio.

Experiment #	Oil (g)	DBU (mL)	MeOH (mL)	Temperature (C)	Rxn Time (hr)	CO2 Time (hr)	Centrifuge Time (hr)	Product weight (g)	Yield (mass %)
1	5	60	30	70	0.5	0.5	0.5	0	0
2	5	60	40	70	0.5	0.5	0.5	0.1997	3.994
3	5	60	50	70	0.5	0.5	0.5	0	0
4	5	60	60	70	0.5	0.5	0.5	0	0
5	5	70	30	70	0.5	0.5	0.5	0.7846	15.692
6	5	70	40	70	0.5	0.5	0.5	1.0557	21.114
7	5	70	50	70	0.5	0.5	0.5	0.3275	8.585
8	5	70	60	70	0.5	0.5	0.5	N/A	N/A
9	5	80	30	70	0.5	0.5	0.5	0	0
10	5	80	40	70	0.5	0.5	0.5	1.545	30.9
11	5	80	50	70	0.5	0.5	0.5		0
12	5	80	60	70	0.5	0.5	0.5		0
13	5	90	30	70	0.5	0.5	0.5		0
14	5	90	40	70	0.5	0.5	0.5		0
15	5	90	50	70	0.5	0.5	0.5		0
16	5	90	60	70	0.5	0.5	0.5		0

## DISCUSSION

The extraction of coffee oil from SCGs was done with a shaker oven in lieu of a Soxhlet extractor due to time reduction while producing the same results. Soxhlet extraction is the traditional, commonly used method for oil extraction because of its advantages in requiring little training and producing more product mass than other methods (Danlami et al., 2014). However, the setup of

the Soxhlet extraction process poses certain limitations. Such limitations include the potential for solvent vapors to escape into the air, thermal decomposition, and safety concerns specific to coffee oil extraction. It is required that the solvent medium must be heated to boiling point for evaporation. Because hexane acts as the solvent, temperatures must reach above 70°C; this raises concerns for potential thermal degradation of the oil due to uneven heating. Altering the composition of the coffee

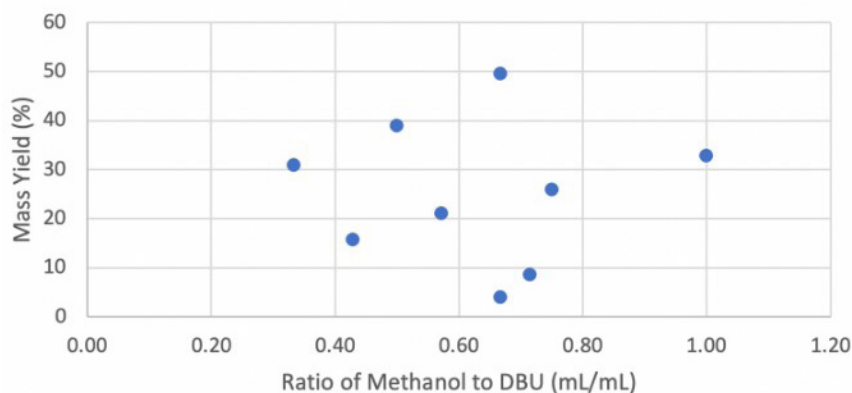
oil drastically impacts the makeup of the biodiesel because of the difference in FAME content and the minute amounts produced. Additionally, the temperature requirements and evaporation of the solvent along with the experimental run time leads to a potential for hexane to escape into the atmosphere. This is a safety concern because hexane vapors are toxic and dangerous if not handled properly. It was deemed that using a shaker oven to extract coffee oil would be a suitable alternative because of the lower extraction time and safer conditions. The shaker oven has a simple setup and can have multiple samples extracted simultaneously in the same machine, making it apt for coffee oil extraction from SCGs. It also mitigates high heating temperatures and safety concerns from boiling the solvent, and it requires lower amounts of hexane to operate.

From Table 3 in the results, a relationship can be derived from the amount of methanol and DBU used and the product biodiesel yield. Increasing both the amount of methanol and DBU increases the yield of biodiesel; however, only increasing one or the other (methanol or DBU) causes the opposite effects. For example, in Table 3, it can be seen that biodiesel yields drop significantly when methanol is increased from 40 mL to 50 mL while keeping the amount of DBU at 80 mL. It can be concluded that additional reactants aid in producing more product, but an excess of one or the other hinders transesterification performance. At 40 mL methanol and 80 mL DBU is also where maximum yield was observed, so further exploration of reactant volume within this range would be beneficial in determining optimum yields.

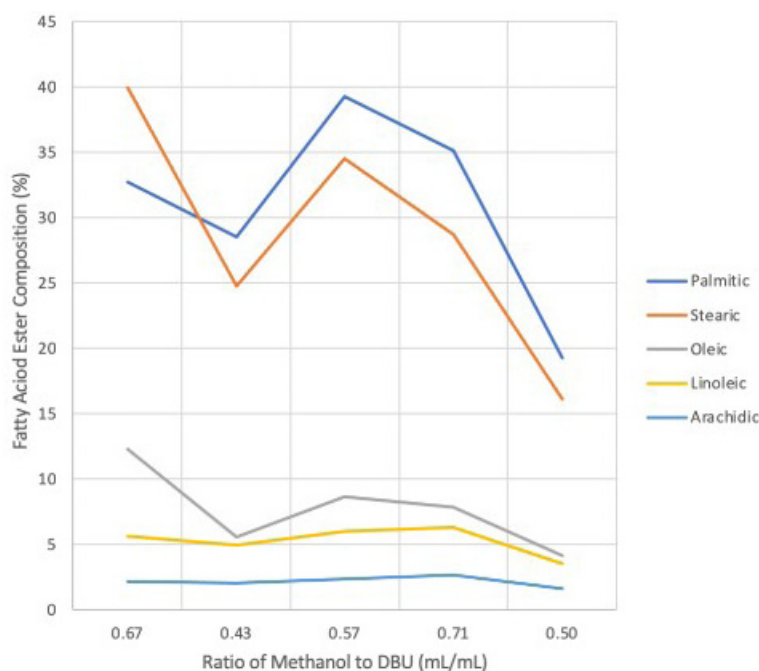
During experimental testing, there were multiple instances for potential human error. Measuring reagents, experimental run times, coffee oil separation via rotary evaporator, biodiesel extraction from the transesterification reaction, and preparing the biodiesel sample for GC-MS analysis were all done manually, so variance from human error is to be expected. The small amounts of biodiesel yielded after each experimental run also allowed for less accuracy when extracting for GC-MS analysis, so the purity of the biodiesel could potentially be hindered by unwanted compounds, such as glycerol. Time constraints were also present between each step of the biodiesel production process due to scheduling conflicts and equipment breakdown. For example, the evaporation of methanol from the transesterification reaction was dependent on when the rotary evaporator was available. This led to ranges between 1 and 14 days when the solution could sit and compound degradation could have taken place.

Statistical analysis using JMP of biodiesel production was conducted on the yield calculations to determine what factors affected yield. It was seen that mass percent calculations showed higher yield percent compared to results obtained from GC-MS analysis because mass percent accounted for all compounds that were present within the sample. Figure 4 graphically depicts the yield of the 10 samples of biodiesel that were analyzed in relation to the ratio of methanol to DBU that was used.

As can be seen, there was little correlation between the ratio of methanol to DBU and the yield of biodiesel as determined by manual weighing. However, not all



**FIGURE 4.** Mass yield percent of biodiesel versus the ratio of methanol to DBU used in the reaction.



**FIGURE 5.** Composition percent of the five typical fatty acid esters in five samples of biodiesel made from original SCGs using varying ratios of methanol to DBU.

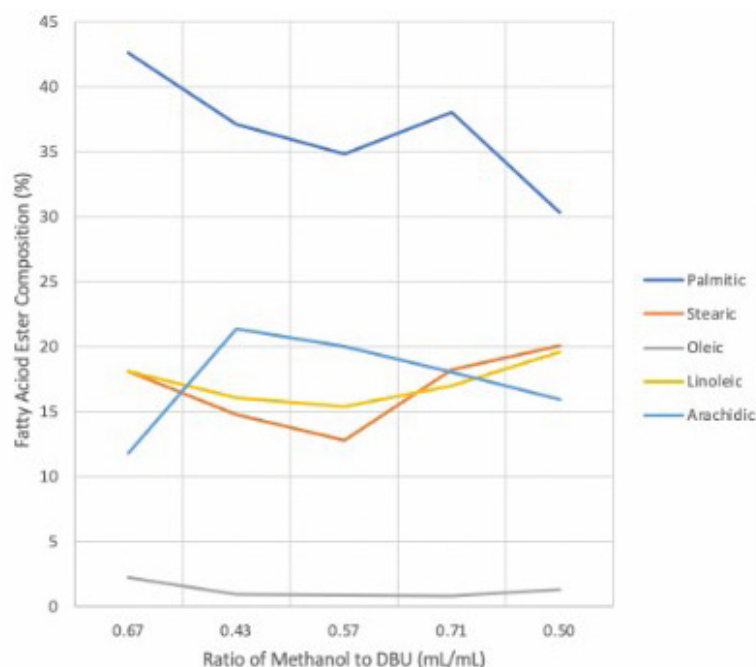
compounds in the sample are necessarily part of what is defined as biodiesel. The GC-MS analysis allowed for characterization, so yield percent was lower but results are more accurate and representative of what the yield percent actually is. As well, GC-MS analysis allowed for determination of the composition of fatty acid methyl esters in the biodiesel, shown in Figures 5 and 6.

As shown in Figures 5 and 6, the composition of the fatty acid methyl esters typical of biodiesel had no significant correlation with the ratio of methanol to DBU used in the reaction. Rather, it seems to depend more on the oil sample used for biomass, as the fatty acid composition can range heavily depending on which coffee grounds are used for extraction. From yield results, optimization of biodiesel production will have to be continued to determine the optimum temperature, methanol amount, and DBU amount needed. The yield of biodiesel is also near negligible, so research on potential unwanted side reactions must also be conducted.

In the scope of environmental and economic considerations, further research must also be conducted to determine how to successfully recycle the DBU catalyst for future trials. Current attempts to remedy this

problem by depolarizing the DBU with nitrogen were met with some difficulty because the samples crystallized over time, as shown in Figure 3. It is hypothesized that the crystallization of the DBU-rich solution is because of an abundance of the undesired reaction taking place, and in turn the products formed from these reactions have a significant impact on the crystallization of DBU solution. These undesired products are also hypothesized to be larger and heavier, making their melting points around the range of 50–60°C. This range was hypothesized because of the temperature range at which experimental reactions are run, as well as what other observed FAME melting points are. For example, lignoceric acid methyl ester is found in each sample and has a melting point around 60°C and a molecular weight of 382.4 (National Center for Biotechnology Information, 2023).

To improve results, continued analysis of why crystallization occurs should be continued using GC-MS to determine the compounds causing such phenomenon. Future experiments to determine the validity of recycling DBU and continuing with DBU as a catalyst will be conducted, but current results display inefficient support for DBU being the optimal catalyst for the transesterification.



**FIGURE 6.** Composition percent of the five typical fatty acid esters in five samples of biodiesel made from different SCGs and using varying ratios of methanol to DBU.

## CONCLUSION AND RECOMMENDATIONS

In conclusion, this reaction needs more refining before it becomes viable. One way to make this happen faster is to research how to recycle the DBU. Even with the most optimized experiment, the yield of biodiesel was too low to justify the cost of DBU for the reaction. When a method to convert the contaminated DBU into a reusable form is found, the reaction would be more economically viable. A potential issue is the DBU sample composition that could be a consequence of the acid-washing process that was introduced previously. While the process does reduce the amount of soap produced, it can be noted that the samples of the contaminated DBU crystallized after time. With some early tests, it was found that long carbon chain fatty acids (LCCFAs) formed and their high melting point may be the cause of the crystallization. This could cause problems when finding a way to clean the DBU in the future.

While DBU looks to be a dead end, these experiments have proven that it is possible to produce biodiesel from coffee oil. Future experiments should look to use other catalysts that are currently being used in biodiesel

production. Researchers should focus on finding the optimum reaction conditions for coffee oil and maybe using just SCGs. Future experiments should also explore whether the acid-washing step is worth the extra resources and time needed.

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