SUSTAINABLE BIODIESEL PRODUCTION THROUGH THE INTERESTERIFICATION OF WASTE COOKING OIL USING CATALYST DERIVED FROM EGGSHELLS AND MINE TAILINGS

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Abstract:

The heavy reliance on fossil fuels to meet rising global energy demands is exacerbating global warming. To mitigate these effects, investing in research of sustainable alternatives like biodiesel is essential. This experiment explored the utilization and potential of mine tailing (mine waste) impregnated eggshells as a catalyst in the interesterification of waste cooking oil. The catalyst was synthesized through impregnation and calcination methods. The mine tailing impregnated eggshell catalyst (MTIEC) was characterized using a Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) to identify the morphology and element composition, respectively. The experimental design framework was architected utilizing the Taguchi Design Model to optimize biodiesel synthesis trials by systematically adjusting the methyl acetate to oil ratio (MA: Oil), catalyst loading (wt%), reaction time (min), and calcination temperature combinations. A maximum biodiesel yield of 145% was achieved at a 20:1 MA: Oil ratio, 1% catalyst loading, 30 min reaction time, and a calcination temperature of 800˚C, maintaining a constant reaction temperature of 40˚ C. The findings exemplify the potential of using a waste-derived catalyst in the production of biodiesel, while also highlighting the importance of calcination temperatures to optimize catalyst effectiveness.

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1. Introduction

With rising global populations and the expansion of industries worldwide, the demand for energy has reached unprecedented levels (Li et al., 2024). During contemporary times, the burning of fossil fuels has been the main supplier for this demand. However, the depletion of fossil reserves and the negative health impacts that are affiliated with it drive research for more sustainable alternatives (Saka et al., 2025). One of these alternatives, which has caught the attention of many due to its environmental performance, safety, and functional properties, is biodiesel. Being a renewable energy source, biodiesel has the potential to reduce recyclable waste making it an enticing alternative to replace fossil fuels (Ismaeel et al., 2024).

Although biodiesel seems to be an exemplar for renewable energy, its functionality is unavailing if the reactants used to make biodiesel are not renewable themselves; one of the most important of these reactants is the choice of feedstock. The selection of feedstock plays a crucial role in determining the cost, sustainability, and feasibility of the biodiesel production. When considering these factors one option that stands out is waste cooking oil (WCO) due to its low cost, abundance, and potential for recycling (Lopresto, 2025). One major barrier in the creation of biodiesel has been the large cost of starting materials. Fortunately, due to cheapness and availability, low-grade feedstock like WCOs have become a highly attractive option (Aderibigbe et al., 2021). Abundance also plays a role. Every year over 190 million metric tons of used vegetable oils are produced (*Vegetable Oils Consumption Worldwide 2024/25| Statista*, 2025). This plethora of materials exemplifies how waste mitigation using WCOs can ultimately support the concept of circular economy (Mannu et al., 2020).

While feedstock choice is important, it is not the only aspect to consider. The efficiency, cost, and sustainability of biodiesel production is also heavily reliant on the choice of catalyst (Yeneneh & Sufe, 2025). Although usage of homogeneous catalysts is typically considered the traditional route, they are fraught with issues such as difficulty of separation, challenges in reusability, and need for extensive purification steps (Saka et al., 2025). On the other hand, enzyme-based catalysts provide an environmentally friendly solution, but they are not economically viable due to their high price and challenges in handling (Ashok et al., 2024). In

comparison, both catalysts fall short of the capabilities demonstrated by heterogeneous catalysts. Heterogenous catalysts have the advantage of reusability, efficient conversion, and easy separation/purification of the product (Aderibigbe et al., 2021). The evident advantages that heterogenous catalysts contribute have solidified its position as a strong contender for future catalyst usage.

Two specific sources for heterogenous catalysts that display high potential are mine tailings and eggshells. Mine tailings are waste produced by mining operations. They contain metals and metal oxides that can be used as catalysts for various chemical reactions (Kim et al., 2025). Eggshells contain Calcium Oxide (CaO) which is highly appealing for its ease of synthesis from inexpensive materials, high basic strength, and low environmental impact (Ahmad Khan et al., 2025). Both processes of converting waste-to-resources support the goal of environmental conservation while simultaneously lowering costs.

To produce biodiesel, the catalysts specified above are used for two different reactions, transesterification and interesterification. Transesterification is the reaction between triglyceride and a short chain alcohol in the presence of a catalyst that produces biodiesel and glycerol (Aderibigbe et al., 2021). Transesterification has been noted as the customary method for creating biodiesel; however, its production of glycerol has prompted researchers to test for alternatives like interesterification (Youssef et al., 2024). Interesterification is a reaction where methyl acetate is used as replacement for the alcohol, resulting in the production of triacetin instead of glycerol. Triacetin's solubility in biodiesel enhances its value relative to glycerol (Ansori et al., 2025).

Several researchers have worked on the production of biodiesel through WCO. For instance, Qadeer et al. (2025) reported the optimal conditions (1:24 oil to methyl acetate molar ratio, 24 min, 0.9% wt% CH_3ONa catalyst) to produce biodiesel with the interesterification of WCO through microwave irradiation. The experiment highlighted the viability of using interesterification by optimizing the process to create a 92.5% conversion rate. Another experiment, conducted by Ahmad Khan et al. (2025), investigates the feasibility of TiO_2 in fusion with egg-shell derived CaO catalyst to produce biodiesel. This research explores the application

of utilizing a sustainable catalyst approach with waste materials to produce a high biodiesel yield of 93.73%. Although the research that is planned to be conducted has similar goals of emphasizing environmental sustainability of the production of biodiesel through options like WCO and eggshells, a key aspect that discerns this experiment is the use of mine tailings. Despite promising results from using mine tailings as a catalyst, there is still a need for further exploration into the optimization of mine tailings as a catalyst (Kim et al., 2025).

Based on the aforementioned, this research aims to enhance the development of biodiesel from waste cooking oil through interesterification in the presence of Mine Tailings-Impregnated Eggshell Catalyst (MTIEC). One facet of this research involves evaluating the properties and effectiveness of the catalyst using energy-dispersive X-ray spectroscopy (EDS). Another aspect of this research is to thoroughly investigate the physiochemical properties of the produced biodiesel using gas chromatography–mass spectrometry (GC-MS).

2. Materials and Methods

2.1 Materials

The feedstock used in this experiment was waste cooking oil (WCO). WCO was obtained from a personally spent cooking oil. The catalyst that was utilized in this experiment was Mine Tailings-Impregnated Eggshell Catalyst (MTIEC). The mine tailings were received from the Lowell Institute for Mineral Resources while the eggshells originated from store bought eggs then personal usage. All other chemicals used in this experiment including Methyl Acetate (99% w/w), Potassium Hydroxide (90% w/w), Hydrochloric Acid (37% w/w), Sulfuric Acid (98% w/w), Ethanol (95% w/w), Phenolphthalein Indicator, and Petroleum-Ether (99% w/w) were analytic grade purchased from Sigma Aldrich.

2.2 Methods

2.2.1 Pretreatment

The pre-treatment of WCO is a critical step to remove contaminants that could hinder results (Youssef et al., 2024). Therefore, the WCO was filtered with a sieve to remove large particles.

In addition to preparing WCO, the catalyst must also be prepared. For the eggshells, they were initially rinsed with distilled water to remove any impurities. Then, they were allowed to dry in a drying rack oven for 24 hours. Next, they were ground into a fine powder to increase surface area for a faster reaction. Finally, it was stored in a desiccator until further use. On the other hand, the mine tailings were received as fine grain and then stored in a desiccator prior to use.

Once both the mine tailings and the eggshells were properly prepared, the impregnation process was perused. First, mine tailings were dissolved in 1M sulfuric acid (H_2SO_4) until color stayed consistent and no bubbles were forming. Then ground eggshells were gradually added to the solution and agitated thoroughly until bubbling stopped. The mix ratio of mine tailings and eggshell was 1:1. After agitation, the mixture was filtered to remove liquid, then it was allowed to dry in the drying rack oven for 24 hours. This completes the impregnation process. After impregnation, the calcination process followed. The solid catalyst was calcinated at 400˚C, 600˚C, and 800˚C to create three different catalysts. These catalysts were then labeled $MTIEC_4$, $MTIEC_6$, $MTIEC_8$.

2.2.2 Operational Procedures

30 grams of WCO were first measured and put into a flask. Next, the amount of catalyst was weighed out based on wt.% (e.g., 0.1% × 30 g). After that, based on stoichiometry the amount of methyl acetate needed was measured out. Thereafter, a magnetic stirrer that has a controllable temperature was used to agitate the mixture. To prevent material loss, the top was sealed with a rubber stopper that was fitted with a hole for a temperature sensor. A foil paper was also used to cover the top of the system. The reaction is allowed to run for the specified time.

After the reaction time elapsed, the product was poured into a filter paper to separate the catalyst. The remaining biodiesel was then poured into a flask and was allowed to settle for 24 hours. Finally, the amount of product was then recorded then stored for characterization. Figure 1 demonstrates the experimental setup.

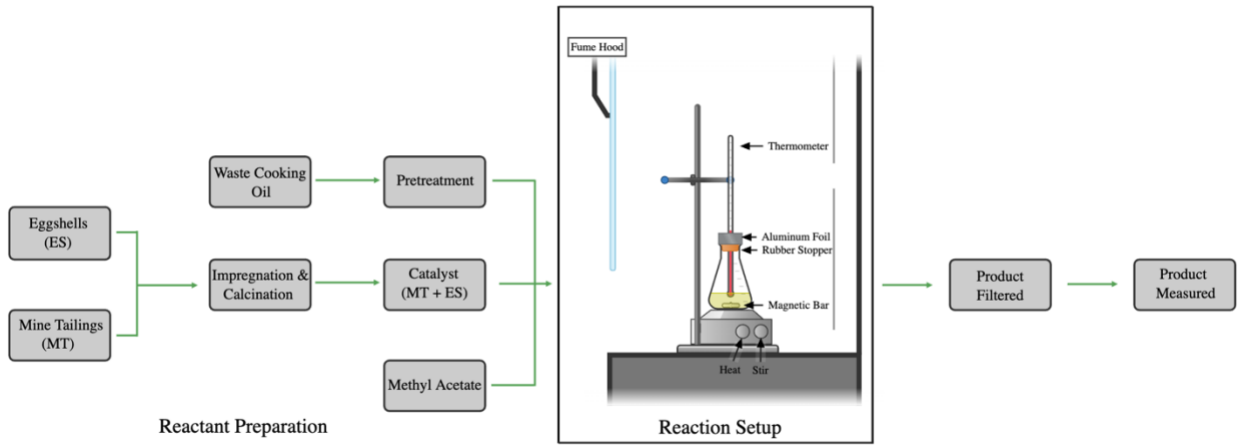

Figure 1. Experimental Setup

2.3 Experimental Design

The experiment has three independent variables: methyl acetate to oil ratio, catalyst loading/concentration, and reaction time. Conditions used were chosen based off analysis of four different interesterification experiments and their optimal conditions (Ansori et al., 2025; Makarevičienė et al., 2024; Qadeer et al., 2025; Youssef et al., 2024). For methyl acetate to oil molar ratio the range was from 10:1–20:1. Catalyst loading/reaction (wt%) was on a range from 0.5–1.0. Reaction time was measured at 30 min to 60 min. The dependent variable in the experiment is biodiesel yield. Biodiesel yield was calculated according to Equation 1.0. Additionally, a control variable that is particularly important in this experiment is the temperature was held stable at 40˚C throughout the entire reaction. Based on this range, design expert software was used to develop the design of the experiment [Taguchi Design Model] as shown in Table 1a, 1b, and 1c. Each table uses the same design of the experiment, but different catalyst design.

$$\text{Biodiesel Yield } (\%) = \frac{\text{mass of biodiesel used}}{\text{mass of oil used}} \times 100 \qquad (1.0)$$

Table 1a: design of experiment for $MTIEC_4$

Run	Factor 1 A:MA/OIL ratio	Factor 2 B:Cat Loading wt.%	Factor 3 C:Time min	Response 1 Biodiesel Yield
1	10	0.5	30	
2	20	0.5	60	
3	10	1	60	
4	20	1	30	

Table 1b: design of experiment for $MTIEC_6$

Run	Factor 1 A:MA/OIL ratio	Factor 2 B:Cat Loading wt.%	Factor 3 C:Time min	Response 1 Biodiesel Yield
1	10	0.5	30	
2	20	0.5	60	
3	10	1	60	
4	20	1	30	

Table 1c: design of experiment for $MTIEC_8$

Run	Factor 1 A:MA/OIL ratio	Factor 2 B:Cat Loading wt.%	Factor 3 C:Time min	Response 1 Biodiesel Yield
1	10	0.5	30	
2	20	0.5	60	
3	10	1	60	
4	20	1	30	

2.4 Characterizations

2.4.1 Catalyst Probe

Understanding the catalyst composition is one of the most important aspects of this experiment. Therefore, the technique employed to acquire the morphological structure and all

the elemental components of the catalyst was Scanning electron, microscopy and energy-dispersive X-ray spectroscopy [SEM-EDS].

2.4.2 WCO and Biodiesel

One important aspect of the experiment is finding the molecular weight of the WCO to add the appropriate amount of methyl acetate. To do this, the acid value was determined experimentally through a standard titration; initially, the saponification value was also determined experimentally through a standard reflux saponification method. However, after multiple trials, sufficient saponification was not achieved. Consequently, the saponification value was taken as an average for vegetable oil found in Ivanova's study (Ivanova et al., 2022). These two values were then used to calculate the molecular weight of the oil with the formula below.

$$M = 56.1 * 1000 * \frac{3}{(SV - AV)} \quad (2.0)$$

To determine both WCO properties and biodiesel properties, gas chromatography–mass spectrometry (GC-MS) was employed.

3. Results

3.1 $MTIEC_4$

3.1.1 $MTIEC_4$ Biodiesel Yield

Figure 2 shows that increasing the MA:Oil ratio from 10:1 to 20:1 at 1% $MTIEC_4$ catalyst loading results in a peak biodiesel yield of 141%. Additionally, when catalyst loading increased from 0.5% to 1% at a MA:Oil ratio of 10:1 the biodiesel yield drops slightly.

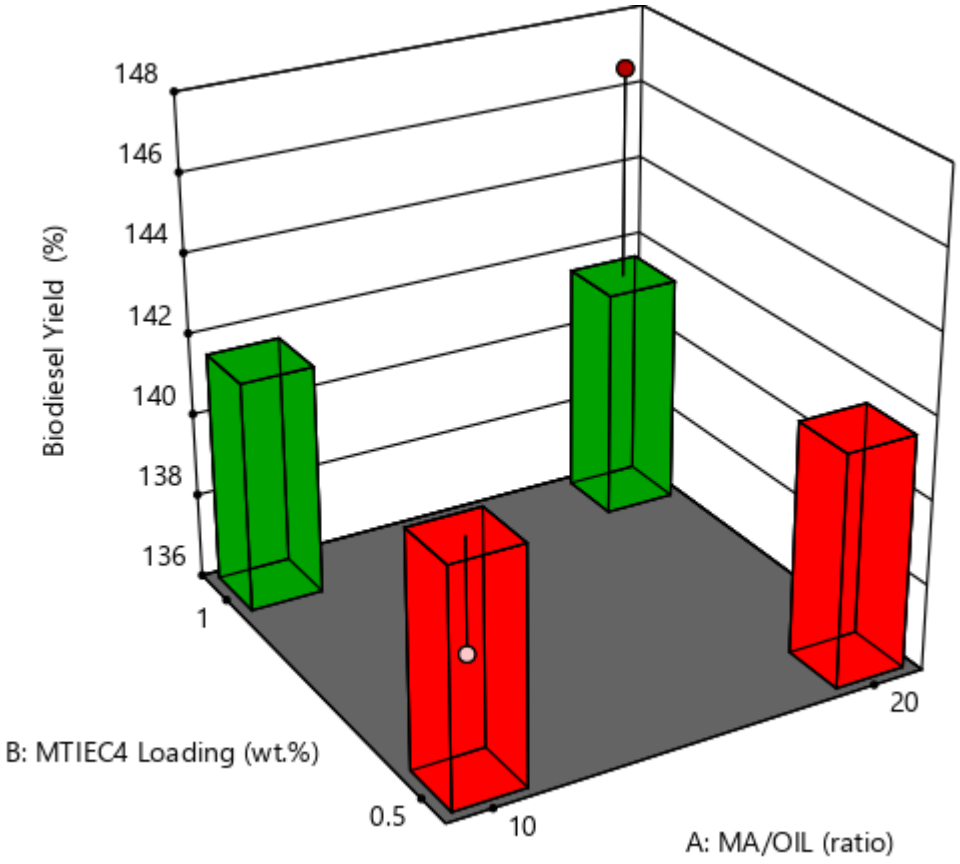

Figure 2. 3D plot for the effect of MA: Oil molar ratio and catalyst concentration ($MTIEC_4$) on biodiesel yield

3.1.2 $MTIEC_4$ Morphology and Elemental Composition

The morphological examination of $MTIEC_4$ catalyst through SEM is seen in Figure 3 (A, B, C). The imaging in Figure 3 (C), scaled at 100 µm, reveals the catalyst has an irregular shape with heterogeneous particles. As you zoom in from Figure 3 (C) to Figure 3 (A), scaled at 5 µm, it is still heterogenous, but the smooth surface is highlighted. This indicates that there are not many pours.

The elemental composition of $MTIEC_4$ is seen in Figure 3 (D). EDS imaging of $MTIEC_4$, used to obtain Figure 3 (D), was able to confirm the presence of important elements like Ca, Si, and other metal ions that aid in the catalytic reaction. Important features of the graph include 45,000 counts of Si and 10,000 counts of Ca showing a high Si:Ca ratio.

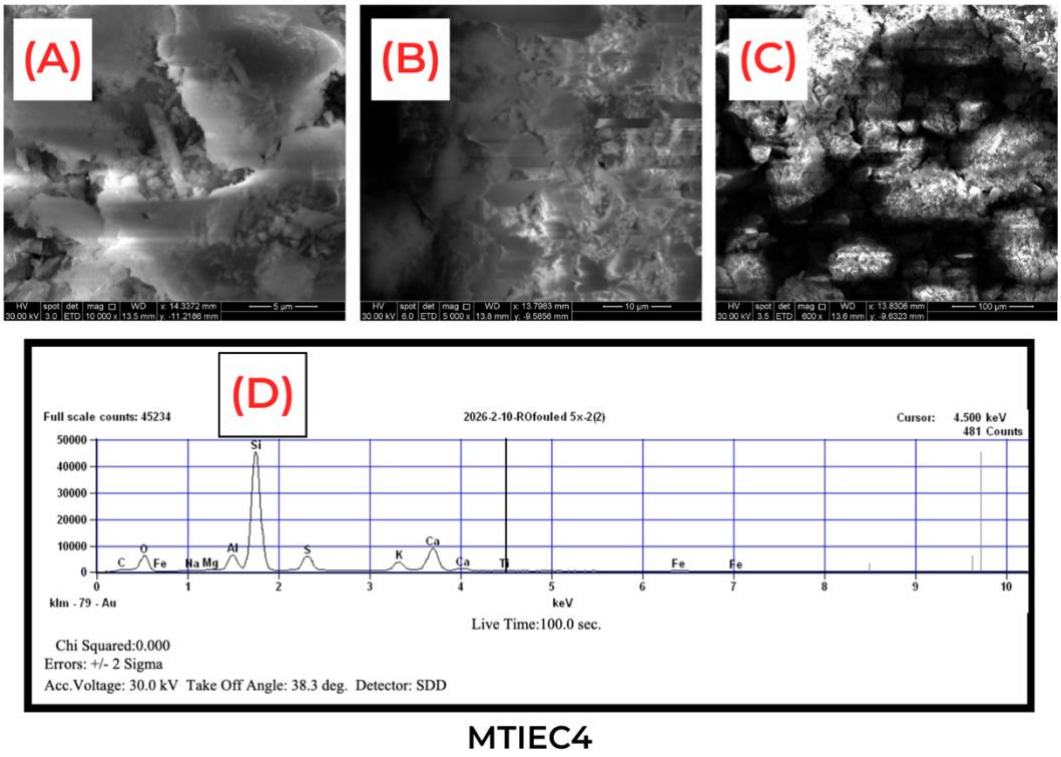

Figure 3. SEM imaging of $MTIEC_4$ (A, B, C), EDS imaging of $MTIEC_4$ (D)

3.2 $MTIEC_6$

3.2.1 $MTIEC_6$ Biodiesel Yield

Figure 4 shows that increasing the MA:Oil ratio from 10:1 to 20:1 at 1% $MTIEC_6$ catalyst loading results in a peak biodiesel yield of 137%. Additionally, when catalyst loading increased from 0.5% to 1% at a MA:Oil ratio of 10:1 the biodiesel yield drops slightly.

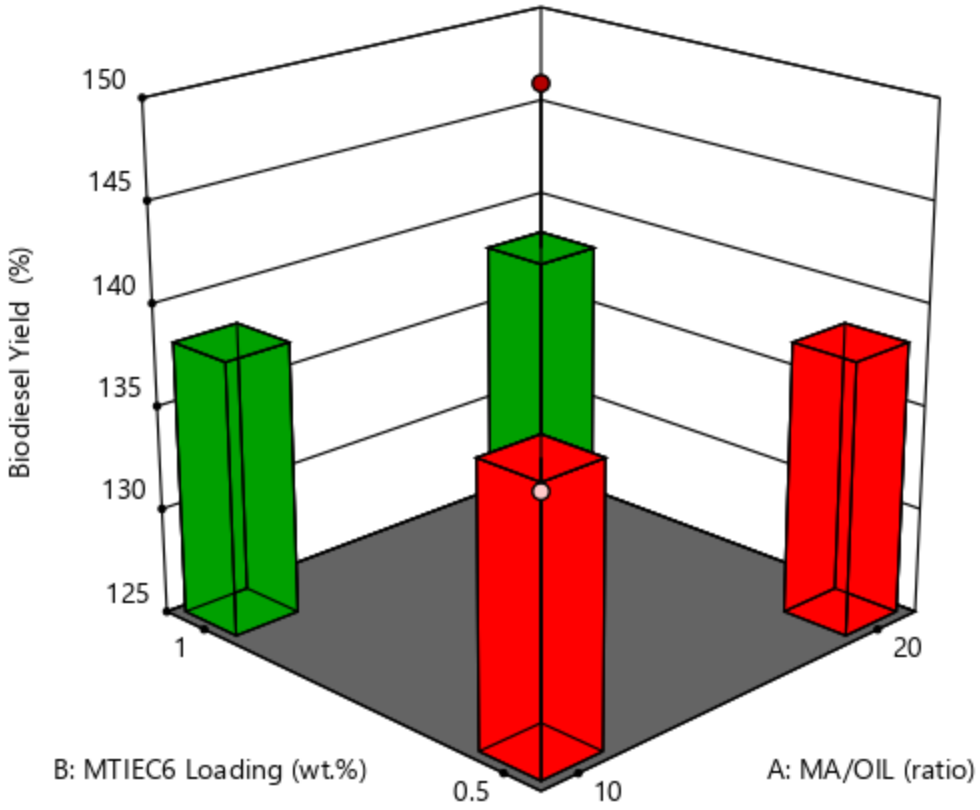

Figure 4. 3D plot for the effect of MA: Oil molar ratio and catalyst concentration ($MTIEC_6$) on biodiesel yield

3.2.2 $MTIEC_6$ Morphology and Elemental Composition

The morphological examination of $MTIEC_6$ catalyst through SEM is seen in Figure 5 (A, B, C). The imaging in Figure 5 (C), scaled at 100 µm, reveals the catalyst has an irregular shape with heterogeneous particles. As you zoom in from Figure 5 (C) to Figure 5 (A), scaled at 5 µm, it is still heterogenous, but the smooth surface is highlighted. This indicates that there are not many pours.

The elemental composition of $MTIEC_6$ is seen in Figure 5 (D). EDS imaging of $MTIEC_6$, used to obtain Figure 5 (D), was able to confirm the presence of important elements like Ca, Si, and other metal ions that aid in the catalytic reaction. Important features of the graph include 6,000 counts of Si and 8,100 counts of Ca showing a low Si:Ca ratio.

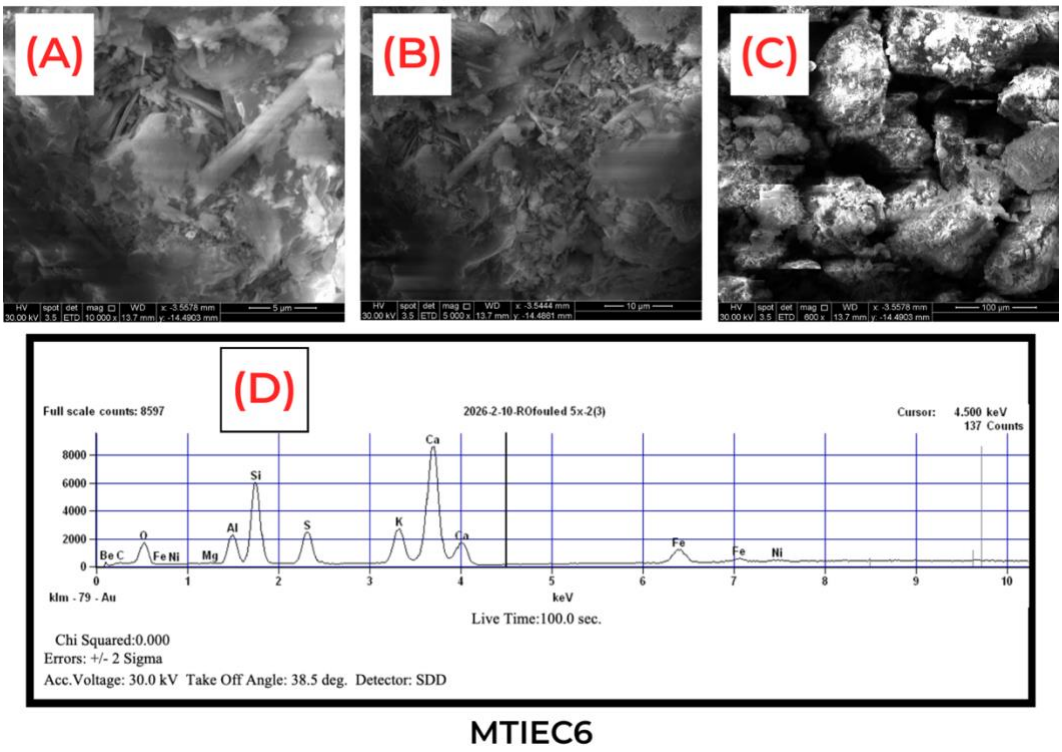

Figure 5. SEM imaging of $MTIEC_6$ (A, B, C), EDS imaging of $MTIEC_6$ (D)

3.3 $MTIEC_8$

3.3.1 $MTIEC_8$ Biodiesel Yield

Figure 6 shows that increasing the MA:Oil ratio from 10:1 to 20:1 at 1% $MTIEC_6$ catalyst loading results in a peak biodiesel yield of 145%. Additionally, when catalyst loading increased from 0.5% to 1% at a MA:Oil ratio of 10:1 the biodiesel yield drops slightly.

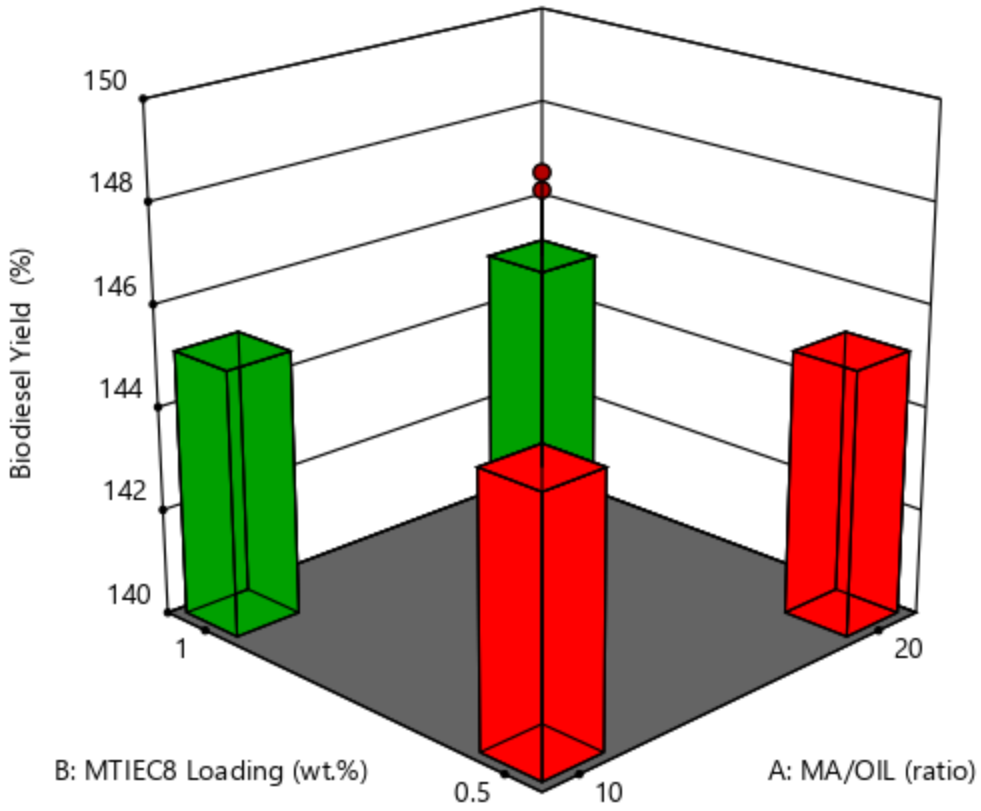

Figure 6. 3D plot for the effect of MA: Oil molar ratio and catalyst concentration ($MTIEC_8$) on biodiesel yield

3.3.2 $MTIEC_8$ Morphology and Elemental Composition

The morphological examination of $MTIEC_8$ catalyst through SEM is seen in Figure 7 (A, B, C). The imaging in Figure 7 (C), scaled at 100 µm, reveals the catalyst has an irregular shape with heterogeneous particles. As you zoom in from Figure 7 (C) to Figure 7 (A), scaled at 5 µm, it is still heterogenous, but the smooth surface is highlighted. This indicates that there are not many pours.

The elemental composition of $MTIEC_8$ is seen in Figure 7 (D). EDS imaging of $MTIEC_8$, used to obtain Figure 7 (D), was able to confirm the presence of important elements like Ca, Si, and other metal ions that aid in the catalytic reaction. Important features of the graph include 10,000 counts of Si and 35,000 counts of Ca showing a low Si:Ca ratio. Moreover, traces of transition metals are present along with 25,000 counts of S.

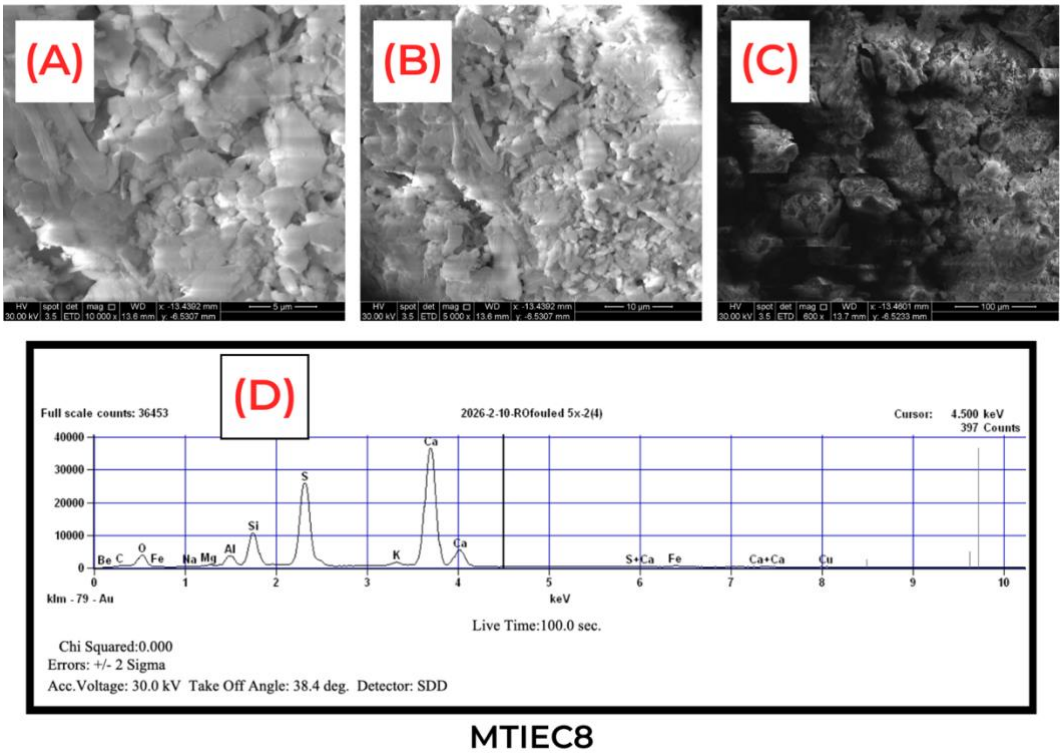

Figure 7. SEM imaging of $MTIEC_8$ (A, B, C), EDS imaging of $MTIEC_8$ (D)

4. Discussion

4.1 Biodiesel Yield

4.1.1 Comparison of 3 Catalyst and Implications

The results from figure 2, 4, and 6 all show the trend that an increase in MA: Oil ratio has a very high influence on biodiesel yield. This trend is consistent with Le' Chatelier's principal. Additionally, all catalyst trials exhibited a similar trend of a slight decrease of biodiesel yield when catalyst load increased from 0.5% to 1% of a MA: Oil of 10:1. This exemplifies a potential issue when increasing the catalyst load to a certain point. Finally, when comparing biodiesel yield, $MTIEC_8$ consistently showed the highest results for all trials while $MTIEC_6$ consistently exhibited the lowest results for all trials.

4.1.2 Justification

Similar biodiesel results were also obtained by Omar Youssef. In this study, Youssef utilizes a sulfuric acid catalyst for the interesterification of WCO. Similar to this experiment, Youssef found that the highest biodiesel yield of 142% was obtained at their highest MA:Oil ratio of 12:1. Furthermore, the article found that when they were at a lower MA:Oil ratio, increasing the catalyst concentration had detrimental effects on yield, while at a higher MA:Oil ratio, a increasing the catalyst concentration had positive effects on the yield (Youssef et al., 2024). While the difference between catalyst loading in this experiment drops only slightly when at a lower MA:Oil ratio (10:1), it still portrays a similar trend.

4.2 Catalyst Characterization

4.2.1 Comparison of 3 Catalyst and Implications

The results from figure 3 (A, B, C), figure 5 (A, B, C), and figure 7 (A, B, C) all exhibit similar morphological structure. After analyzing the structure, the images are not differentiable enough to allows for any reasonable conclusions. Additionally, images from the SEM have a clear charging effect making it less reliable to analyze. On the other hand, the results from figure 3 (D), figure 5 (D), and figure 7 (D), provides us with better insight regarding the elemental composition of the catalysts. A noticeable trend amongst all three catalyst is as the calcination temperature increased the Si: Ca ratio decreased likely indicating more successful calcination. A more successful calcination helps support the results that $MTIEC_8$ contained the highest biodiesel. An aberration that is noticed in the EDS results is seen in figure 5 (D) where there is a drop-in full-scale count by an order of magnitude when compared to figure 3 (D) and figure 7 (D). A feasible explanation for this abnormality is an issue with the $MTIEC_6$ sample itself. This would explain the lowest biodiesel yield obtained when utilizing $MTIEC_6$. Finally, all three samples contain small traces of metal ions likely due to mine tailing impregnation. However, the metal traces amount is small enough that the success of the mine tailings on enhancing the catalyst is still equivocal.

4.2.2 Justification

Similar catalyst EDS imaging results were also found by Rashid Imran Ahmad Khan and Linjun Li. In Khan's experiment they tested the efficacy of an eggshell-derived $CaO@TiO_2$

nanocatalyst on biodiesel production through the transesterification of waste soybean oil. In this experiment the eggshells were first calcinated at 900˚C for 5 h. Then TiO_2 was impregnated onto the CaO where it was soon after subjected to controlled calcination at 600˚C for 4 h. After imaging with EDS, Khan found high spikes in Ca, Ti, and O, three elements that are expected due to the impregnation of TiO_2 and calcination of eggshells (Ahmad Khan et al., 2025). A similar trend in elements was seen across the results from this experiment as well. One significant difference is seen in the amount of metal ions present. In Khan's experiment, the metal ion (Ti) had a significant presence in the EDS imaging. Linjun Li's experiment also used eggshells-based catalyst and found similar results in their EDS imaging (Li et al., 2024).

4.3 Limitations and Future Research

Due to a lack of time, only 6 of the 12 trials of interesterification were completed while other parts of the data were extrapolated using software. Additionally, due to a lack of equipment some of the processes were unsuccessful. One notable case was the inability to get the waste cooking oil to saponify in order to obtain a saponification value. Another example showcasing lack of equipment was the unsuccessful attempt to characterize the WOC and biodiesel with the GC-MS; after attempting to image, esterified fatty acids were unable to be identified likely due to improper column in the GC-MS.

In the future, completing the 6 other trials of interesterification would allow for more conclusive results with less speculation. Additionally, with more time, more characterization techniques could allow for more insight and explanations for observations during the experiment. Finally, conducting research with better equipment would ensure a smaller impact of external factors that could potentially alter the experiment.

5. Conclusion

This research underlines the viability of a sustainable method to produce biodiesel through the interesterification of waste cooking oil and employing mine tailing impregnated eggshells as a catalyst. Overall, the highest biodiesel yield obtained was 145%, further supporting this method as feasible. The experimental design to determine maximum biodiesel yield was designed by the Taguchi Design Model, where combinations were discovered to test

the methyl acetate to oil ratio, catalyst loading, time, and catalyst type. In the end, $MTIEC_8$ was the most successful in biodiesel yield with EDS characterizations to support the results. While this research proves the concept is sensible, there are still many uncertainties. Consequently, additional research can be done to improve the certainty of the results. Although this study is not as conclusive as others online, many of the results found were similar to or related to those of previous publications. This connection ultimately allows for further knowledge and advancement in the research fields attempting to find alternative, renewable, and sustainable energy production.

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