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Glycerol Acetylation with Acetic Acid to Produce Triacetin using Modified Canature 001x8FG as Heterogeneous Catalyst

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ABSTRACT

The fast increase in biodiesel production has resulted in an excess of glycerol, a key byproduct that requires effective valorization solutions. This research looks at acetylation of glycerol with acetic acid as a sustainable way to manufacture triacetin, a high-value fuel additive. Canature 001x8FG, a heterogeneous ion-exchange resin catalyst, was tested in its unmodified, modified (sulfonated), and uncatalyzed forms. Batch studies were carried out utilizing a reflux condenser and mechanical stirring, and the product was analyzed using gas chromatography. The modified Canature resin was made by treating it with 0.1 M sulfuric acid. The sulfonated resin displayed better catalytic efficiency, attaining up to 100% glycerol conversion under ideal conditions defined by Response Surface Methodology (RSM): 95 °C, 11:1 acetic acid-to-glycerol molar ratio, and 1.5-hour reaction duration. Triacetin was the most common product under these circumstances. At 95 °C with a 6:1 ratio and 1.5 hours, the modified resin converted about 70% of glycerol, whereas the original resin only converted 13%. The uncatalyzed process demonstrated insignificant conversion. FTIR-ATR analysis indicated effective sulfonation, with the improved catalyst showing stronger -SO_3H (S=O and S-O) stretching bands at 1171, 1125, and 1035 cm^{-1}. Increased Brønsted-acid site density leads to higher catalytic efficiency and selectivity for triacetin. These results emphasize the importance of chemical modification in increasing resin catalyst activity and show the potential of sulfonated Canature 001X8FG resin as a viable heterogeneous catalyst for glycerol valorization in biodiesel-related processes.

Keywords: Glycerol acetylation; triacetin; ion-exchange resin; sulfonation; heterogeneous catalysis

INTRODUCTION

Glycerol is a major byproduct of biodiesel manufacturing, with approximately 100 kg generated per ton of biodiesel through transesterification of vegetable oils or animal fats (Ayoub & Abdullah 2012; Hidayati et al. 2026). Although glycerol is widely used in food, pharmaceutical, cosmetic, and chemical industries, its oversupply due to biodiesel expansion has diminished its economic value, prompting research into valorization strategies (Nda-Umar et al. 2020). This need for innovative conversion pathways also resonates with broader sustainability perspectives, such as those highlighted by Muska et al. who emphasize the European Green Deal's objective of advancing environmentally responsible chemical processes, underscoring the importance of developing greener valorization routes for surplus glycerol (Muska et al. 2025). Among these, acetylation with acetic acid offers a promising route to produce monoacetin, diacetin, and triacetin, with triacetin being particularly valuable for its diverse industrial applications (Banu et al. 2019; Fronza et al. 2025). Achieving high selectivity toward triacetin while minimizing intermediate acetins requires careful optimization of reaction parameters such as temperature, molar ratio, and catalyst loading.

Acidic ion-exchange resins, such as Amberlyst-15 and Dowex Monosphere 650C, have demonstrated strong catalytic performance in glycerol acetylation, offering advantages over homogeneous acids including ease of separation, reduced corrosion, and reusability (Reinoso & Boldrini 2019; Zhou et al. 2012). However, studies remain limited in exploring alternative resins such as Canature

001x8FG, which possesses comparable physicochemical properties and potential for high triacetin yield. Furthermore, systematic optimization of reaction conditions using Canature 001x8FG has not been comprehensively reported, and the influence of resin modification on catalytic activity and selectivity remains insufficiently understood. Addressing these gaps is essential to establish scalable, cost-effective, and environmentally sustainable processes for glycerol valorization.

This study therefore investigates glycerol acetylation using Canature 001x8FG resin and acetic acid, focusing on the effects of temperature, molar ratio, and reaction time. Performance comparisons between unmodified and acid-treated resins are conducted to elucidate the role of sulfonation in enhancing catalytic efficiency. The objective is to develop a low-cost and practical pathway for triacetin synthesis that contributes to the sustainable utilization of surplus glycerol from biodiesel production (Nda-Umar et al. 2020; Banu et al. 2019).

METHODOLOGY

MATERIALS AND CHEMICALS

Acetic acid (99.8% purity, R & M Chemical, Malaysia) and glycerol (99.7% purity, DChemie Malaysia) were used as reactants. Methanol (GC grade, Merck, Malaysia) was employed to rinse and clean the GC syringe to ensure accurate gas chromatography analysis, while acetone (analytical grade, Merck, Malaysia) was used to clean the FTIR sensor to maintain instrument sensitivity and reliability. Deionized water was utilized for dilution during preparation of calibration standards. Commercial Canature 001x8FG ion exchange resins were obtained from ITech Water Filter Specialists (Malaysia). For thermal control, commercial cooking oil (palmbased, Saji brand, Malaysia) was used as the heating medium to ensure uniform heat distribution.

EQUIPMENT

To achieve precise reaction and analysis in the glycerol acetylation experiment, many apparatuses were used. The reactor was a round-bottom flask with a heating mantle, hot plate, and magnetic stirrer to provide regulated heating and consistent mixing. A reflux condenser was used to condense volatile vapors such as acetic acid. Gas chromatography and FTIR (Perkin Elmer and Thermo Nicolet) were used to quantify products and analyze functional groups, respectively. Moisture was removed from the ion exchange resin using a drying oven, and exact sample quantities were extracted using a GC syringe. An analytical balance and thermometer were used to determine catalyst mass and reaction temperature.

PREPARATION OF STANDARD

A 10% glycerol solution was made by weighing 1.2643 g (equal to 1 ml) of pure glycerol in a 50 ml beaker to minimize sensitivity error. Separately, 9 g of deionized water was weighed and added to the glycerol, then swirled until well combined. The same procedure was followed for 30% and 60% glycerol concentrations. Using a GC syringe, 1 µL of each standard solution was injected into a gas chromatography (GC) system (AutoSystem XL, Perkin Elmer, USA) equipped with a flame ionization detector and a fused silica column. The GC program included a temperature ramp from 100 to 220 °C. The injection and detector temperatures were 250 and 275 degrees Celsius, respectively, using nitrogen as the carrier gas. The same technique was followed by acetic acid (10%, 30%, and 60%) and pure water.

CATALYST PREPARATION

An analytical balance was used to weigh exactly 100 grams of Canature 001x8FG ion exchange resins with a moisture level of less than 2%. The resin was was soaked with 0.1 M sulfuric acid in a 250 mL beaker and stirring with a glass rod for 30 minutes to introduce sulfonic acid groups (–SO_3H) onto the resin surface, thereby increasing its acidity and active site density, which enhances its catalytic performance in glycerol acetylation. The beaker was sealed with parafilm and allowed to stand overnight. After that, the resin was filtered using a filter funnel and paper. To eliminate remaining moisture, the soaked resin was transferred to a watch glass dish and dried in an oven at 110°C for 10 minutes. The dried catalyst was kept in a beaker covered with parafilm.

GLYCEROL ACETYLATION USING MODIFIED CANATURE 001X8FG

Figure 1 illustrates the reaction setup, consisting of a roundbottom flask equipped with a heating mantle, reflux condenser, magnetic stirrer, thermometer, and a 1000 mL beaker containing cooking oil as the heating medium. Cooling water was circulated through the condenser, and the heating device was activated to maintain the desired temperature. For the first experimental run, glycerol and acetic acid were introduced at a 3:1 molar ratio (AA/G), with 7.06 g of ionexchange resin serving as the catalyst. The mixture contained 44 mL of acetic acid and 56 mL of

glycerol, heated to 80 °C and stirred at 290 rpm. Once the target temperature was reached, the remaining acetic acid was added, and the reaction time was recorded. The process was maintained for 1.5 h, after which the mixture was cooled, filtered, and collected. Subsequent experiments were conducted under varying conditions of temperature, molar ratio, and reaction time, as summarized in Table 2.

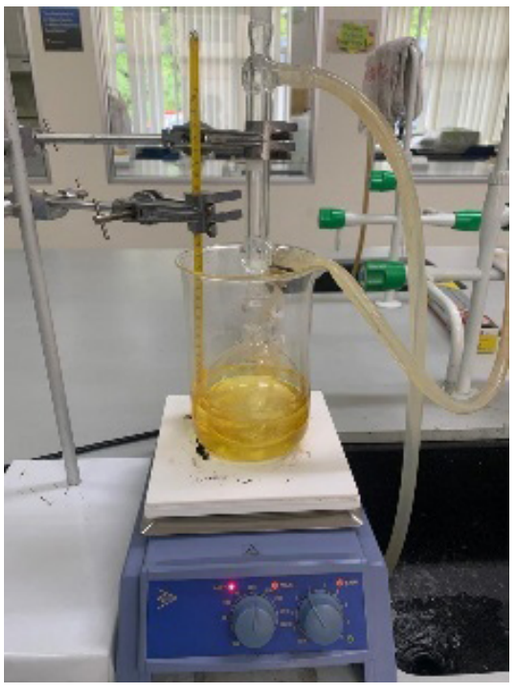

FIGURE 1. Glycerol acetylation experimental setup

OPTIMIZATION OF RESPONSE SURFACE METHODOLOGY (RSM)

Response Surface Methodology (RSM) was used to analyze and optimize the glycerol acetylation process by varying three critical variables: reaction temperature (°C), acetic acid to glycerol molar ratio, and reaction duration (hours). The ranges and levels of these independent variables are summarized in Table 1, which was used to determine the experimental design in Minitab software. A central composite design (CCD) with three factors and two levels was generated, resulting in a total of 20 experimental runs as recommended by the program (Table 2).

TABLE 1. The numeric and categoric factors

Code	Variables	Unit	Low	High
A	Temperature	**°C**	80	110
B	Molar ratio (AA: G)	mol/mol	3	9
C	Reaction time	hour	1	2

GAS CHROMATOGRAPHY (GC)

The sample was placed in a 25 mL beaker. Methanol (GC grade) was used to rinse the GC syringe three to four times to remove impurities, as methanol is a volatile solvent that evaporates quickly and leaves minimal residue, ensuring the syringe is free from contaminants that could interfere with chromatographic analysis. After rinsing, the syringe tip was wiped with laboratorygrade tissue. To eliminate any residual methanol, 5 µL of sample was drawn and discharged into an empty 10 mL beaker three times to saturate the syringe. 1 µL of the prepared sample was then injected into the GC column using a microsyringe. Analyses were performed on an AutoSystem XL (Perkin Elmer, USA) equipped with a flame ionization detector (FID) and a fused silica capillary column (30 m length, 0.32 mm internal diameter, 0.25 µm film thickness). The column temperature program was set as follows: 100 °C for 1 min, ramped to 130 °C at 15 °C/min and held for 1 min, then increased to 220 °C at 40 °C/min and maintained for 9 min. The injector and detector temperatures were 250 °C and 275 °C, respectively, with nitrogen as the carrier gas. Each run was completed within 15 min. Glycerol conversion (%) was calculated using the following formula:

$$(\%)\ \text{glycerol conversion} = \quad \times 100\% \qquad (1)$$

CHARACTERIZATION OF MODIFIED AND UNMOFIDIED CANATURE 001X8FG CATALYST

The functional groups of both modified and unmodified Canature 001X8FG ionexchange resin catalysts were characterized using a Thermo Nicolet iS10 Fourier Transform Infrared (FTIR) spectrometer equipped with an Attenuated Total Reflectance (ATR) accessory. The ATR unit contained a diamond crystal sensor and a pressure arm with a locking knob to secure the sample and ensure optimal contact for signal detection. A small amount of resin was placed on the ATR crystal, the pressure arm lowered, and the knob adjusted to achieve stable positioning. Spectra were recorded using OMNIC software over the range of 4000–400 cm^{-1} and saved for subsequent analysis. The same procedure was repeated for the unmodified resin under identical conditions. Comparative evaluation of the spectra allowed identification of differences in functional group composition between the modified and unmodified catalysts.

RESULTS AND DISCUSSION

EXPERIMENTAL RESULTS

Table 2 shows the experimental results and design matrix for the glycerol conversion (Response).

TABLE 2. Experimental results

No. of runs	Temperature (°C)	Mole ratio (AA/G)	Time (h)	Glycerol conversion (%)
1	95	1	1.5	28
2	95	11	1.5	100
3	95	6	2.3	36
4	70	6	1.5	31
5	119	6	1.5	33
6	95	6	1.5	15
7	95	6	0.7	21
8	95	6	1.5	14
9	110	9	2.0	4
10	110	3	1.0	35
11	95	6	1.5	70
12	95	6	1.5	31
13	95	6	1.5	7
14	80	9	2.0	60
15	110	9	1.0	0
16	80	3	1.0	15
17	80	3	2.0	17
18	95	6	1.5	22
19	80	9	1.0	14
20	110	3	2.0	22

A mathematical equation was devised to describe how temperature (A), the mole ratio (Acetic Acid: Glycerol) (B), and reaction time (C) affect glycerol conversion. The regression coefficients were obtained using Minitab software.

Glycerol conversion = -370 + 5.83 Temperature + 8.1 Molar ratio + 124 Time taken - 0.00162 Temperature*Temperature + 1.097 Molar ratio*Molar ratio - 20.7 Time taken*Time taken - 0.278 Temperature*Molar ratio - 0.88 Temperature*Time taken + 5.43 Molar ratio*Time taken (2)

Among the three factors, reaction time had the biggest positive influence on glycerol conversion, with a value of +124, followed by molar ratio at +8.1. Temperature has a lesser impact and, owing to its negative quadratic effect, returns decline as one move higher. The optimum values of the variables were obtained using Eqn. (2)

SCREENING STUDY VIA STATISTICAL ANALYSIS BY USING DESIGN OF EXPERIMENT

TABLE 3. ANOVA table

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	10	6378.9	637.9	0.94	0.544
Blocks	1	583.1	583.1	0.86	0.379
Linear	3	1586.4	528.8	0.78	0.536
A-Temperature	1	175.1	175.1	0.26	0.624
B-Molar ratio	1	1064.4	1064.4	1.56	0.243
C-Time taken	1	346.9	346.9	0.51	0.494
Square	3	2039.8	679.9	1	0.437
A*A	1	176.5	176.5	0.26	0.623
B*B	1	1373.2	1373.2	2.02	0.189
C*C	1	333.1	333.1	0.49	0.502
2-Way Interaction	3	2135.8	711.9	1.05	0.419
A*B	1	1255.6	1255.6	1.84	0.208
A*B	1	348.6	348.6	0.51	0.492
B*C	1	531.6	531.6	0.78	0.4
Error	9	6129.9	681.1		
Lack-of-Fit	5	3951.1	790.2	1.45	0.37
Pure Error	4	2178.8	544.7		
Total	19	12508.8			

The ANOVA table shows how temperature, molar ratio, and reaction duration affect the conversion of glycerol during esterification (Nda-Umar et al. 2021). The complete model, including linear, quadratic, and interaction components, has an F-value of 0.94 and a p-value of 0.544, suggesting it is not statistically significant at the 95% level. This shows that the combined impacts of the variables do not account for a large percentage of the conversion, meaning that most of the variability might be attributable to random error or other unaccounted-for causes. Individually, none of the linear factors exhibit significant impacts (p-values > 0.05). Although statistically insignificant (p = 0.224), the molar ratio seems to be more relevant, as proven by an experimental run attaining 100% conversion at an 11:1 ratio and 95°C for 1.5 hours.

The quadratic and interaction components did not approach statistical significance, indicating a lack of substantial nonlinear or synergistic effects throughout the sample. The molar ratio squared term exhibited the lowest p-value (0.144), indicating that conversion may grow to an optimal point before plateauing or falling owing to side reactions or dilution. Similarly, temperature and time squared factors (p = 0.623 and 0.504) had minor effects, while interaction effects like molar ratio × time (p = 0.400) were insignificant. The statistical model accurately explains the data, as shown by the lack-of-fit test (F = 1.45, p = 0.370). The lack of statistical significance may be attributed to experimental discrepancies rather than systemic methodological errors. Variability in catalyst activity, measurement uncertainty or inconsistent mixing efficiency

could have introduced noise into the dataset, masking true effects. These uncontrolled factors, combined with limitations such as a relatively small sample size and restricted experimental runs, reduce the statistical power of the analysis and increase susceptibility to random variation. Collectively, these findings highlight the constraints of the experimental design and underscore the need for more rigorous control of operating conditions, expanded sample sets, and replication to ensure reliable detection of significant trends.

THE INDIVIDUAL EFFECTS OF TEMPERATURE, MOLAR RATIO, AND REACTION TIME ON GLYCEROL CONVERSION

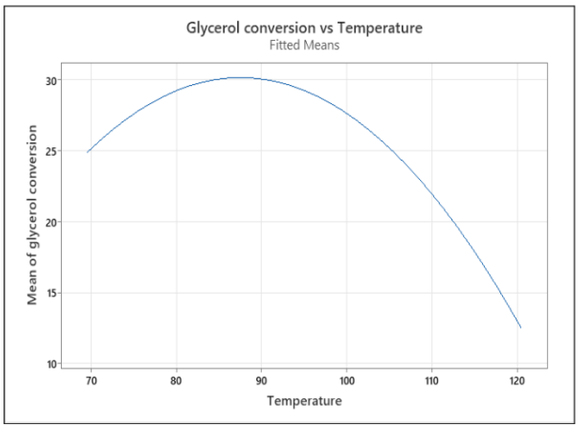

FIGURE 2. Glycerol conversion vs Temperature

Increasing the temperature from 70 °C to 90 °C enhances glycerol conversion via improved reaction kinetics and better diffusion of reactants to catalyst active sites (Banu et al. 2019) (Figure 2). At 90 °C, the Canature 001X8FG ion exchange resin performs well, resulting in the maximum conversion since the resin remains active, acetic acid is liquid, and reaction kinetics are maximized. Sample 4 at 70 °C converts just 31%, whereas sample 11 at 95 °C converts 70%, consistent with Dalibera et al.'s (2021) results of 93% conversion at 90 °C and 96% at 100 °C. At temperatures exceeding 90 °C, the resin's acid sites deactivate, and acetic acid is lost by evaporation, resulting in decreased conversion. This is noticeable when comparing sample 9 (4% conversion at 110 °C) and sample 14 (60% conversion at 80 °C). Mufrodi et al. (2020) also found a reduction from 98.23% to 96.42% when the temperature climbed from 80 °C to 100 °C. The catalyst performs best at a reaction temperature of about 90°C.

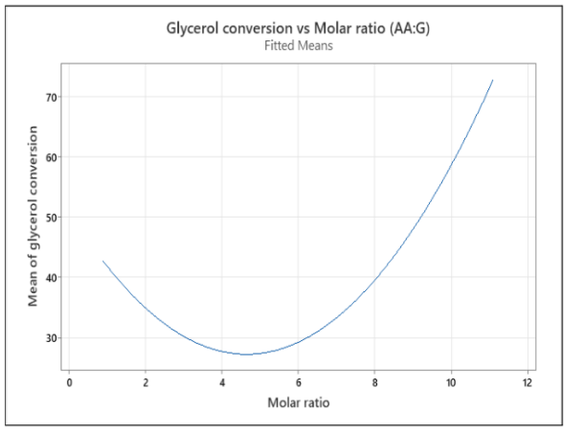

FIGURE 3. Glycerol conversion vs Molar ratio

Figure 3 demonstrates a U-shaped pattern, with glycerol conversion lowest at mid-range molar ratios and rising at both low and high ratios, as verified by Table 2. At a low molar ratio of 1 (Run 1), conversion is 28%, greater than many mid-range values, such as 7-36% for molar ratios of 6 (e.g., Runs 3-8, 11-13, 18), independent of temperature or reaction duration. For example, Run-13 (molar ratio 6) achieves just 7% conversion and Run-6 only 15% despite extended reaction durations, indicating equilibrium restrictions and inadequate resin swelling at these ratios (Pal et al. 2012). Conversion peaks at the extremes, with Run 2 (molar ratio 11) at 100%, and Runs 14 (molar ratio 9) and 20 (molar ratio 3) hitting 60% and 22%, respectively. Reinoso and Boldrini (2019) reinforce this tendency, finding that raising the acetic acid: glycerol ratio from 6:1 to 9:1 increased conversion from 98% to 99.6%. The results confirm the U-shaped pattern and emphasize the importance of molar ratio in maximizing conversion while using Canature 001X8FG resin.

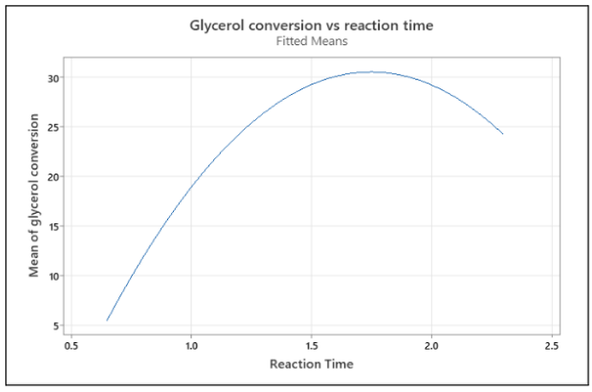

FIGURE 4. Glycerol conversion vs Reaction time

Figure 4 depicts a bell-shaped pattern, with glycerol conversion increasing with reaction time until an ideal point, then decreasing. Runs 11 (1.5h, 70%) and 2 (1.5h, 100%) have the greatest conversion rates, whereas longer durations, such as Run 3 (2.3h, 36%), result in lesser conversion owing to catalyst deactivation or side reactions (Pal et al. 2012). Similarly, shorter timeframes result in lesser conversion, with Run 7 obtaining just 21% in 0.7 h. Sandid et al. (2024) found that increasing response durations from 4 h to 7 h decreased conversion from 99.6% to 99%, supporting this tendency. In summary, the findings demonstrate an optimal reaction time of 1.5-1.8 hours when utilizing Canature 001X8FG resin, emphasizing its relevance in increasing glycerol conversion.

VARIABLE INTERATIONS

Figure 5 demonstrates how temperature and molar ratio affect glycerol conversion. The graph shows three lines for molar ratios of 3:1, 6:1, and 9:1, demonstrating how temperature affects the molar ratio. At a 9:1 molar ratio (green dashed line), conversion peaks at 70-80 °C with values around 65%, but rapidly decreases beyond this range, reaching as low as 10% at 120 °C. Excess acetic acid increases conversion at low temperatures, but may promote catalyst degradation or side reactions at higher temperatures (Pal et al. 2012). This tendency was verified by Runs 9 (4%) and 15 (0%) at 110 °C.

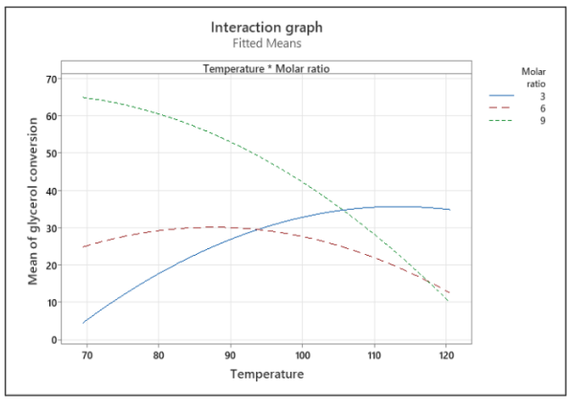

FIGURE 5. Glycerol conversion vs Temperature*Molar ratio

The 6:1 molar ratio (red dotted line) produces more balanced results throughout temperatures, culminating at 90-100 °C with a conversion rate of 30–35%. At 95 °C, Run 11 achieved a 70% conversion rate, whereas Run 6 only reached 15%, indicating the importance of response time. The 3:1 molar ratio (blue line) indicates a continuous increase in conversion with temperature, reaching about 35-40% at temperatures around 110 °C. Runs 10 and 20 showed 35% and 22% conversion, respectively, consistent with Mufrodi et al.'s (2020) findings that lower molar ratios need higher temperatures for greater conversion.

In general, smaller molar ratios benefit at higher temperatures, whereas larger molar ratios function best at moderate temperatures. This suggests a trade-off between thermal activation and reactant availability. The 9:1 ratio is best for temperatures below 95 °C, while the 3:1 ratio thrives for temperatures between 100-110 °C. The 6:1 ratio offers a balanced approach throughout a larger range. Figures 7 and 8 show that the optimal molar ratio for glycerol acetylation with Canature 001X8FG ion exchange resin is 9-11:1 at temperatures below 95 °C.

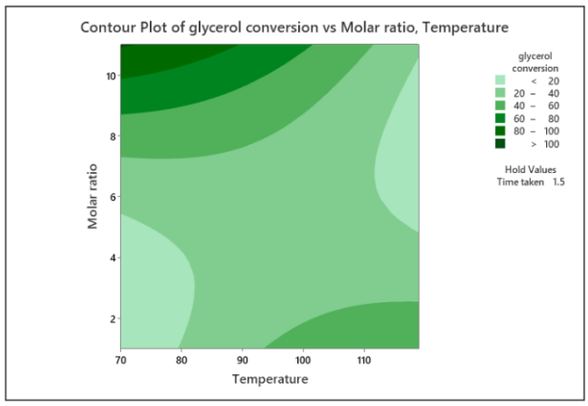

FIGURE 6. Contour plot glycerol conversion vs Temperature*Molar ratio

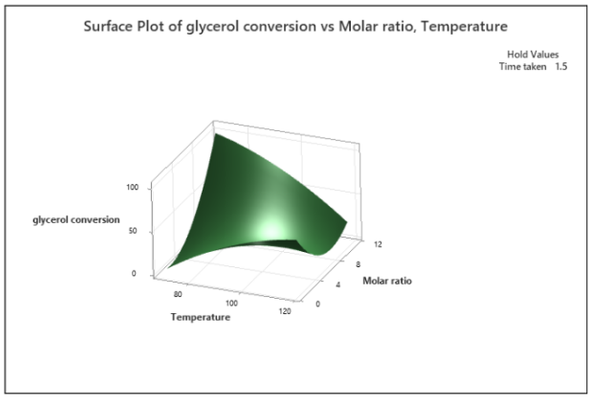

FIGURE 7. Surface plot Glycerol conversion vs Temperature*Molar ratio

Figure 8 illustrates how temperature and reaction time affect glycerol conversion during the acetylation process. The interaction graph shows a nonlinear pattern in which conversion grows with temperature until a certain point, beyond which it decreases. Shorter reaction periods of one hour result in poor conversion across temperatures, suggesting that the catalyst and substrate do not have

enough time to react properly. Run 10 (110°C, 1 hour) had a 35% conversion rate, whereas Run 16 (80°C, 1 hour) had just 15%. Sandid et al. (2024) found that lengthier response periods of 5-7 h resulted in conversion rates of 97-99%, highlighting the significance of extended contact times.

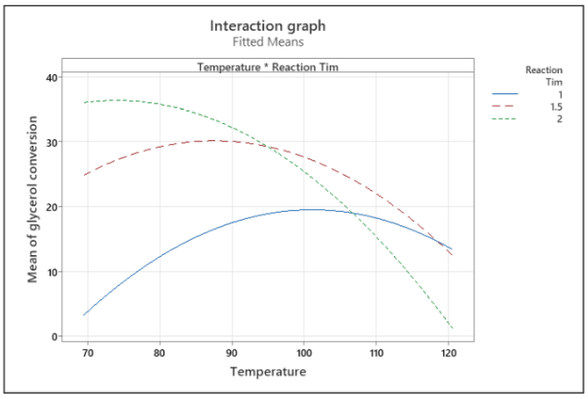

FIGURE 8. Glycerol conversion vs Temperature*Reaction time

With a response time of 1.5 hours, conversion improves and peaks at 90-100°C, indicating an appropriate equilibrium. Run 1 (95°C, 1.5h) produced 28% conversion, whereas Run 5 (119°C, 1.5h) yielded 33%. Run 11 (95 °C, 1.5 h) had the maximum conversion at 70%, showing that this catalyst is best suited for this temperature and period. Under similar circumstances, Runs 6 and 8 yielded much lower conversion rates (15% and 14%), due to experimental variability, catalyst saturation, or different feed compositions (Pal et al. 2012).

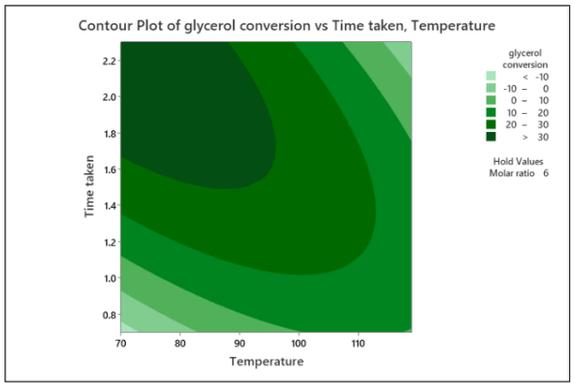

FIGURE 9. Contour plot Glycerol conversion vs Temperature*Reaction time

Conversion rates are maximum at moderate temperatures (80-95 °C) and about 2 hours. Run 14 (80 °C, 2 h) produced 60%, but higher temperatures (Runs 9 and 20, 110 °C) produced just 4% and 22%, respectively. This trend indicates that greater temperatures might cause side reactions, catalyst deactivation, or thermodynamic constraints. Sandid et al. (2024) found that utilizing Canature 001X8FG ion exchange resin for glycerol acetylation yields the best results with longer reaction periods at moderate temperatures (100% at 80 °C, 8 h). Figures 10 and 11 show that the largest conversion occurs at temperatures below 95 °C and response times over 1.5 h.

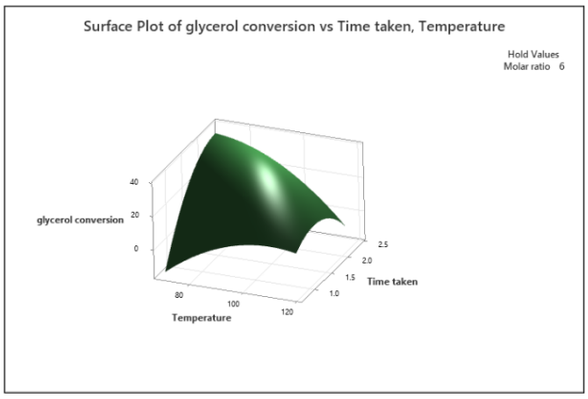

FIGURE 10. Surface plot Glycerol conversion vs Temperature*Reaction time

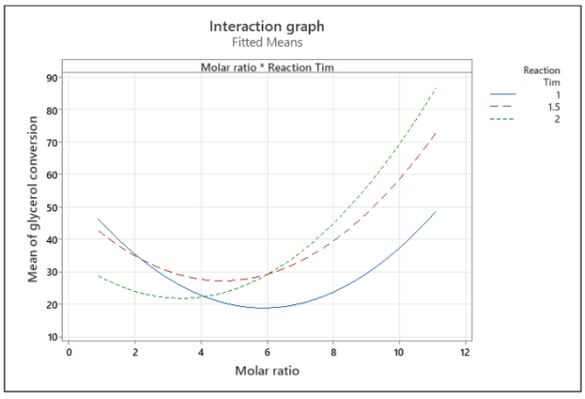

FIGURE 11. Glycerol conversion vs Molar ratio*Reaction time

Figure 11 illustrates how the molar ratio of acetic acid to glycerol and reaction duration impact conversion using the Canature 001X8FG catalyst. The pattern is parabolic, with conversion declining drastically at mid-range ratios (3-6) and climbing when ratios are extremely low or very high, particularly over longer time periods. If given adequate time, conversion rates at low ratios (1-3) range from moderate to high. For example, Run 2 (11:1, 1.5 h)

produced 100% conversion, but Run 1 (1:1, 1.5 h) yielded just 28%. This highlights the role of acetic acid in driving the process (Reinoso and Boldrini, 2019).

Mid-range ratios (3-6) resulted in poor conversion rates independent of response time, suggesting an inhibitory zone. Run 6 (1.5 h, 6:1) gave 15%, whereas Run 3 (2.3 h, 6:1) produced 36%. greater ratios (>6) and longer periods resulted in greater conversion rates, such as Run 14 (2h, 9:1), which reached 60%. Inconsistent findings (Run 9) indicate additional issues, such as beginning moles or temperature effects (Pal et al. 2012).

Overall, longer periods and higher molar ratios performed better, corroborating the results of Sandid et al. (2024), who found that longer times and ratios of 9:1 or 6:1 resulted in 97–99% conversion. Figures 13 and 14 show that the maximum conversion occurs when response durations exceed 1.5 hours and the molar ratio is more than 10, highlighting the necessity of maximizing both factors.

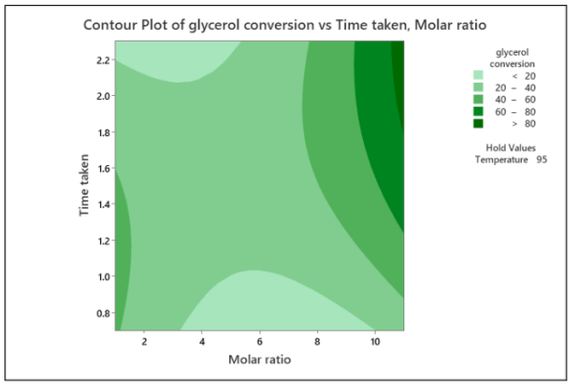

FIGURE 12. Contour plot Glycerol conversion vs Molar ratio*Reaction time

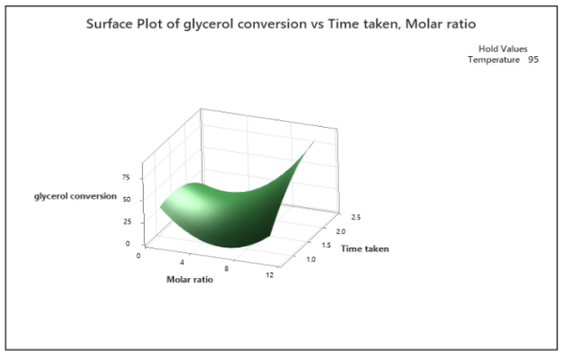

FIGURE 13. Surface plot Glycerol conversion vs Molar ratio*Reaction time

PRODUCT ANALYSIS

According to Figure 14, the FTIR spectra clearly demonstrate the effective synthesis of triacetin. The blue line (original sample) and black line (final sample) exhibit high ester carbonyl (C=O) absorption at 1722 cm^{-1} and 1721 cm^{-1}, respectively, showing the presence of ester groups typical of triacetin. The peaks slightly over 3000 cm^{-1} (3043 cm^{-1} in the original sample and 3033 cm^{-1} in the final sample) indicate C-H stretching vibrations of aliphatic chains. The final sample shows absorptions in the 1014-1048 cm^{-1} and 880-932 cm^{-1} ranges, indicating C-O stretching and CH_2 bending vibrations, confirming the ester linkages and alkane-like structure of triacetin.

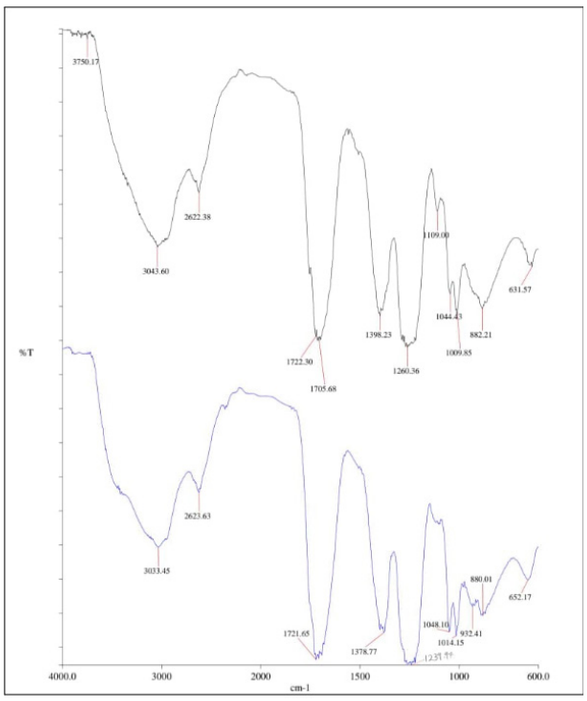

FIGURE 14. FTIR spectrum for sample 2 before(top) and after(bottom) reaction

The final sample shows no appreciable absorption at 3500 cm^{-1}, suggesting the absence of O-H stretching from unreacted glycerol or moisture. There were no peaks at 3390 cm^{-1} or 1625 cm^{-1}, indicating no residual ethanol or acetic acid (Wepoh, 2015). These data show that the end product is very pure triacetin, with distinct ester properties and little residues of starting ingredients or byproducts. This demonstrates that the reaction converted 100% of glycerol, which is consistent with the findings and interpretations of Lacerda et al. (2015) and Wepoh (2015).

COMPARATIVE ANALYSIS OF MODIFIED, UNMODIFIED, AND ABSENCE OF CANATURE 001X8FG CATALYST IN GLYCEROL ACETYLATION

The findings in Table 4 show a clear contrast between Run 21 (using the unmodified Canature 001X8FG ion exchange resin) and Run 22 (no catalyst). The unaltered resin converted 13% of the glycerol in Run 21, but just 0% in Run 22. The unaltered resin has catalytic activity owing to its sulfonic acid (-SO_3H) groups, which may protonate acetic acid and react with glycerol's hydroxyl groups. This supports its effectiveness. Even without acid pretreatment, the resin may generate active sites for esterification, as opposed to the non-catalyst situation, where conversion was entirely absent.

TABLE 4. Experimental runs and glycerol conversion (unmodified catalyst and absence of catalyst)

No. of runs	Temperature (°C)	Mole ratio (AA/G)	Time (h)	Glycerol conversion (%)
6	95	6	1.5	15
8	95	6	1.5	14
11	95	6	1.5	70
12	95	6	1.5	31
18	95	6	1.5	22
21	95	6	1.5	13
22	95	6	1.5	0

However, the modified resin outperforms the original resin. In Table 4, the modified resin produced conversions ranging from 14% to 70% throughout Runs 6-18. Acid treatment improves surface acidity and the accessibility of active sites, making the esterification process far more efficient. Heating and drying the resin may also increase its surface area and porosity, enabling reactants and active sites to interact more effectively.

These results are consistent with those of Sopolapikul et al. (2023), who showed that advanced treatments of ion exchange resins, such as functionalization with TEOS, SG, and magnetic nanoparticles, greatly increased both conversion and triacetin selectivity. Their modified resin had near-total conversion and greater triacetin selectivity than the original, demonstrating the advantages of surface augmentation and acid site formation.

CHARACTERIZATION OF MODIFIED AND UNMODIFIED CANATURE 001X8FG ION EXCHANGE RESIN

According to Figure 16, the unmodified resin exhibits typical peaks of a sulfonated polystyrene-divinylbenzene matrix. The wide O-H stretch at 3388.62 cm^{-1} indicates adsorbed water or hydroxyl groups, while the significant C-H stretch at 2928.14 cm^{-1} validates alkyl chains. The aromatic backbone is visible via the C=C stretch at 1494.73 cm^{-1} and peaks at 832.37 and 671.85 cm^{-1}, showing aromatic C-H and C-S vibrations. The S=O and S-O lengths between 1172-1035 cm^{-1} indicate the existence of sulfonate groups, which are essential for the resin's catalytic properties.

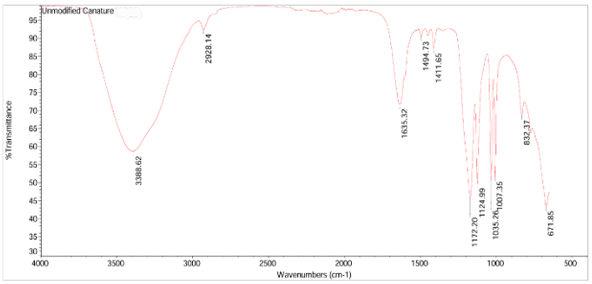

FIGURE 16. FTIR-ATR Spectrum of unmodified Canature 001X8FG ion exchange resin

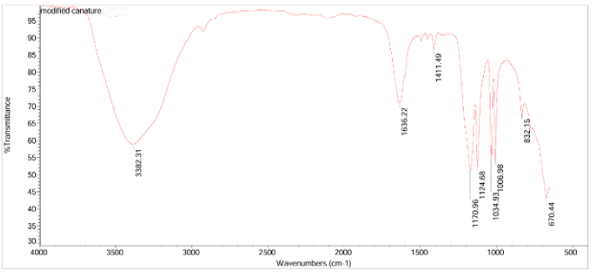

FIGURE 17. FTIR-ATR Spectrum of modified Canature 001X8FG ion exchange resin

Figure 17 demonstrates that acid treatment (0.1 M H_2SO_4, overnight soak) and drying at 110 °C increased the Canature 001X8FG resin's sulfonate peaks (1170.96, 1124.68, and 1034.93 cm^{-1}). This suggests better protonation and surface concentration of -SO_3H groups. The minor shift and increased intensity of the O-H stretching band at 3382.31 cm^{-1} indicates partial dehydration while retaining some bound moisture. Importantly, the aromatic C-H and C-S peaks remained intact, indicating that acid treatment increased the resin's catalytic activity without damaging its backbone. Compared to the Indion 225H resin (Sopolapikul et al. 2023), which exhibited more well-defined sulfonate peaks and a unique ester-related band (1770-1780 cm^{-1}), the treated Canature resin lacked structural complexity, indicating a lower variety and accessibility of functional groups despite improved acidity.

Acid treatment of Canature 001X8FG resin results in increased sulfonate group intensity, making it more effective for Brønsted acid catalysis. However, the lack of

phenyl/ester bands and well-defined sulfonate peaks in Indion 225H indicates that the latter possesses a more durable and flexible structure for esterification. The better triacetin selectivity (31.63%) of Indion 225H (Sopolapikul et al. 2023) verifies its superior design, however acid-treated Canature resin still shows potential for improved catalytic performance in glycerol acetylation.

CONCLUSION

The findings reveal that the acetic acid: glycerol (AA:G) ratio, reaction temperature, and duration all have a significant effect on conversion. The molar ratio showed a U-shaped pattern, with modest conversion (15%) at mid-range ratios (about 6:1) and significant jumps at both extremes (28-100%). At 90 °C, conversion was optimal, while temperatures beyond 100 °C resulted in deactivation and acid loss. The optimal period for conversion was 1.5-1.8 hours, with yields ranging from 70-100%. Shorter or longer intervals yielded less favorable outcomes.

Conversion rates were 13% with unaltered resin (Run 21) and practically 0% with no catalyst (Run 22). In comparison, the sulfuric acid-treated resin obtained 14-70% conversion across screening cycles and over 100% under ideal circumstances. Increased -SO_3H site availability and porosity enhance acid protonation and esterification efficiency relative to raw resin.

FTIR-ATR identified both resins as sulfonated polystyrene-divinylbenzene. The unaltered resin had a wide O-H stretch at 3388 cm^{-1}, C-H bands at 2928 cm^{-1}, aromatic C=C peaks at 1494 cm^{-1}, and S=O and S-O peaks at 1172-1035 cm^{-1}. After acid treatment, -SO_3H peaks rose dramatically at 1170.96, 1124.68, and 1034.93 cm^{-1}, suggesting a greater acid site density. The polymer backbone remained intact. In summary, acid treatment significantly increased active site availability, making the modified resin a substantially more efficient catalyst.

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DECLARATION OF COMPETING INTEREST

None.

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