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The Effect of Multi-Anion Hydrotalcite Catalyst and Amount of CO_3^{2-} Anion Towards Selective Etherification of Glycerol to Diglycerol

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ABSTRACT

The rapid expansion of biodiesel production has resulted in a substantial surplus of glycerol. To maintain balance in the global glycerol market, it is essential to advance research focused on converting glycerol into high-value products. This study demonstrates the conversion of glycerol into diglycerol using a basic-catalyzed etherification reaction. Multi-anion hydrotalcite was studied by combining the added anion sources (NO_3^- and SO_4^{2-}) with the main anion source (CO_3^{2-}). The tri-anion ($CO_3NO_3SO_4$) hydrotalcite sample demonstrated a 92.73% selectivity in the etherification process aimed at producing diglycerol over a 16-hour period. Enhancing the concentration of anions within the hydrotalcite layers can boost basicity, thereby enhancing catalytic activity. Of the samples tested, the one with a 50% concentration of CO_3^{2-} anion emerged as the most effective for etherification, surpassing the performance of the 30% and 70% variants. The optimal proportion of the primary anion source (CO_3^{2-}) plays a crucial role in maintaining the stability of hydrotalcite throughout the reaction. Thus, the appropriate amount of main anion source (CO_3^{2-}) could influence the stability of hydrotalcite during reaction. This study suggested that the modification of hydrotalcite might improve the catalytic activity of hydrotalcite during reaction. For future use, this study is highly potential in overcoming the glycerol surplus problem and broadens the utilization of glycerol in many industries.

Keywords: Multi-anion; hydrotalcite; selective etherification; glycerol; diglycerol

INTRODUCTION

The uncontrolled production of glycerol has led to an excess supply, prompting numerous efforts, particularly among researchers, to develop innovative solutions to address this surplus problem. Various techniques have been explored to enhance glycerol from a lower to a higher grade by modifying its physico-chemical properties (Dhabhai et al. 2023). Extensive research has been conducted on converting glycerol into various derivatives through methods like etherification, oligomerization, and esterification, among other processes (Zakaria et al. 2021).

This study focuses on diglycerol as the key product derived from the etherification process. Among the various isomers produced during the reaction, diglycerol (DG) is anticipated to be the most prevalent. Diglycerol finds applications in three primary industries: food, cosmetics, and polymers. Its incorporation can enhance the quality of products in these sectors. In the food industry, diglycerol's conversion into esters aids in creating superior emulsified food items. As a food additive, diglycerol is highly effective due to its typical production from oleochemical sources (Perez-Santana et al. 2023).

Diglycerol is extensively utilized in the cosmetics industry, where it serves as an advanced ingredient for developing superior personal care and gel products. Its distinctive high refractive index has played a role in reducing the manufacturing costs of pharmaceutical products and possibly in the production of clean products (Tomiie et al. 2016). In the polymer sector, diglycerol is employed in creating a variety of polymer items.

Additionally, it has facilitated the innovation of a new biodegradable plastic, offering an eco-friendly alternative (Zhang et al. 2020).

Layered material catalysts are known for their ability to efficiently transform glycerol into valuable products. Among these, Hydrotalcite (HT) stands out as a promising catalyst. Previous studies have documented the use of HT catalysts, which are primarily produced through methods such as co-precipitation and urea hydrolysis (Ibrahimova, 2022). These catalysts offer the advantage of not experiencing leaching issues and achieving a conversion rate exceeding 50 % (Jaiswal et al. 2024). In the current research, HT is employed to improve the etherification process. The preparation involves an initial combustion step, followed by the creation of metal oxides and recrystallization. This approach is advantageous because it does not require solvents and reduces the synthesis time. Furthermore, it has the potential to address the leaching of alkaline metals, as the catalyst's basicity is regulated by anions in the interlayer, leading to a high-purity final product.

Studies have highlighted the integration of diverse anions within the layers of hydrotalcite (HT). These anions are pivotal in influencing the basicity of HT. A notable feature of HT is its ability to incorporate various counterion species into its framework. Anions suitable for intercalation in HT can be categorized into four principal groups. (Prentice at al. 2020) 1) Inorganic oxyanions and Halides, CO_3^{2-}, NO_3^-, SO_4^{2-}, $Si_2O_5^{2-}$, BO_3^{3-}, AsO_4^{3-}, SeO_4^{2-} , F^-, Cl^-, Br^-, I^- etc. 2) Iso-and hetero-polyoxometallate anions, VO_4^{3-} , CrO_4^{2-}, OsO_4^{2-}, $V_{10}O_{28}^{6-}$, $Mo_7O_{24}^{6-}$, $PW_{12}O_{40}^{3-}$ etc. 3) Transition metal anionic complexes such as, $Fe(CN)_6^{2-}$/ $Fe(CN)_6^{3-}$ etc. 4) Volatile organic anions, CH_3COO^-, $C_6H_5COO^-$, $C_{12}H_{25}COO^-$, $C_2O_4^{2-}$ etc.

Previous research predominantly employed simple anions due to their ease of use, which simplifies the intercalation process and avoids expensive procedures. In this study, carbonate is chosen as the primary anion, a preference supported by earlier findings (Conterosito et al. 2015). The reactivity of HT is affected by the basicity of anions, with carbonate-intercalated materials demonstrating greater activity than those intercalated with halide-type anions (Ibrahimova, 2022; Rana & Shaw, 2024).

This research explored the impact of using multiple anions. The process involved intercalating more than one type of anion, a method not previously documented. In this context, one anion is designated as the primary anion, while the other is considered an additional anion. The aim of incorporating multiple anions was to examine their influence on the structural properties and, subsequently, the catalytic performance of HT in the etherification of glycerol. Several reasons inspired this investigation. Firstly, introducing larger anions expands the spacing between the brucite-like layers, thereby increasing the gallery height of HT, with the reaction predominantly occurring in the interlayer. Secondly, the increased gallery height might also enhance the porous network of HT, potentially facilitating selective reactions.

This paper aims to investigate the effect of multi-anion in HT towards catalytic activity of etherification reaction of glycerol to diglycerol. The anions utilized in the study were CO_3^{2-}, NO_3^-, and SO_4^{2-}. Given that CO_3^{2-} has been previously employed in the synthesis of HT (Meyer et al. 2010; Prentice et al. 2020), it is designated as the primary anion. A di-anion refers to a pair of anions, specifically CO_3^{2-} + NO_3^- or CO_3^{2-} + SO_4^{2-}. In contrast, a tri-anion encompasses the combination of all three anions (CO_3^{2-}+ SO_4^{2-} + NO_3^-). NO_3^- and SO_4^{2-} are classified as additional anions. Furthermore, the influence of the multi-anion on the characteristics of HT was also examined.

METHODOLOGY

HT CATALYST PREPARATION

Hydrotalcite was produced through a combustion technique, utilizing glucose as a fuel source. This method, which involved creating a MgAlO oxide mixture and a recrystallization step, was adapted from Anuar et al. (2016), with the Mg/Al ratio consistently set at 3. The calculation of magnesium's mass relative to aluminum was based on the total weight of the sample, using their molecular weights and maintaining a 3:1 molar ratio. Magnesium and aluminum were each dissolved in 80 ml of deionized water, placed in separate containers to enhance suspension, and heated to 80°C. The samples were then combined in a single container and kept at 80°C. Subsequently, 20 % by weight of Na_2CO_3 and 10 % by weight of glucose, based on the total sample weight, were incorporated. The mixture was stirred thoroughly and maintained at 80°C until complete evaporation of the water occurred. The resulting solid underwent calcination in a furnace at 650°C for 5 minutes, leading to the formation of an oxide mixture.

The oxide compound (MgAlO) was immersed in a 0.05 M anion solution for a duration of five minutes at ambient temperature. This solution contained CO_3^{2-}, SO_4^{2-} , and NO_3^- ions, sourced from 0.05 M Na_2CO_3, 0.05 M Na_2SO_4, and 0.05 M KNO_3, respectively. To investigate the effect of multiple anions, mixtures of anion solutions (including both di-anion and tri-anion solutions) were formulated with CO_3^{2-} constituting 50% of the total. The impact of varying the CO_3^{2-} anion concentration was explored by altering the ratio of the primary anion (CO_3^{2-}) to the supplementary anions (NO_3^- and SO_4^{2-}) to 30 %, 50

%, and 70 %. The specific proportions of anions and the quantities of CO_3^{2-} used in the preparation of tri-anion samples, based on percentages (30 %, 50 %, and 70 %), for each HT sample preparation are outlined in Table 1.

TABLE 1. The summary of the quantities of di-anion and tri-anion sources utilized in the recrystallization process

Samples	CO_3^{2-} anion amount (%)	Second anion amount (%)			Second anion amount in 60 ml of anion solutions (ml)		
		CO_3^{2-}	NO_3^-	SO_4^{2-}	CO_3^{2-}	NO_3^-	SO_4^{2-}
$CO_3 + NO_3$	50	50	50	-	30	30	-
$CO_3 + SO_4$	50	50	-	50	30	-	30
$CO_3 + NO_3 + SO_4$ 50 %	50	50	25	25	30	15	15
$CO_3 + NO_3 + SO_4$ 30%	30	30	35	35	18	21	21
$CO_3 + NO_3 + SO_4$ 70 %	70	70	15	15	42	9	9

CATALYTIC ETHERIFICATION REACTION

The glycerol etherification process served as the basis for demonstrating the catalytic activity. To assess the catalyst's performance, the etherification process was modified by adjusting various parameters. The reaction took place in a setup that included a stirring heating mantle, a three-necked round-bottom flask, a condenser, and a thermocouple.

The experimental procedure was executed as follows: To begin, 2 wt. % of the catalyst was incorporated into 50 g of glycerol. The etherification reaction was performed at a temperature of 240°C for a duration of 16 hours, maintained under a nitrogen atmosphere with continuous stirring. The water by-product formed during the reaction was extracted using a cooled water solenoid. Sampling involved withdrawing 1.5 mL of the reaction mixture every 2 hours, followed by centrifugation at 1000 rpm for 5 minutes to isolate the solid components. The centrifuged sample was subsequently collected for chromatographic analysis.

Samples of the etherification product, specifically diglycerol, were examined using a gas chromatograph (GC) with a flame ionization detector (FID) from Agilent Technologies, managed by Agilent GC Chemstation software. This GC was fitted with a silica capillary column, also from Agilent Technologies, measuring 0.32 mm in internal diameter, 30 meters in length, and 0.25 μm in film thickness, with a temperature range of 60°C to 325°C. The stationary phases included silicon-based polymers (polysiloxanes), polyethylene glycols, and solid adsorbents. The injector was set to a temperature of 100°C, while the detector was kept at 250°C. The column temperature was consistently maintained at 100°C. Prior to injection into the GC, the samples underwent a silylation process.

HT CATALYST CHARACTERIZATION

The catalysts underwent characterization through methods such as X-ray diffraction, surface analysis, Hammett basicity, and chemical composition analysis (EDX).

RESULTS AND DISCUSSION

IMPACT OF DI-ANION AND TRI-ANION SOURCES ON THE CATALYTIC PERFORMANCE OF HT

The multi-anion HT was a revised version of the HT sample, which was synthesized using glucose as a fuel and subsequently calcined at 650 °C. In comparison to the single anion source, the HT sample with multiple anion sources exhibited a lower glycerol conversion rate (Figure 1(a)). After a 16-hour reaction period, the tri-anion sample (HT- CONOSO) demonstrated the highest glycerol conversion, approximately 40.77%. The figure shows that the conversion rates for both the di-anion and tri-anion samples were quite similar, ranging from 36.27% to 40.77%. Although there was a reduction in conversion, the results were still in line with those of a previous study (García-Sancho et al. 2011). Therefore, by adjusting the anion system of HT, it is possible to enhance conversion within a relatively shorter reaction time.

The catalytic activity of the catalyst remained largely unaffected by the presence of anion species within the interlayer. Determining the exact composition of the anion population in the interlayer proved challenging. The interaction between HT and the anion solution occurred naturally, without any deliberate impregnation of anions into the HT structure. Furthermore, the introduction of various anions into the interlayer could potentially increase the gallery height, which might lead to a reduction in the anion population (Cavani et al. 1991; Tichit et al. 1998). This could result in a slightly lower glycerol conversion compared to the sample with a single anion source.

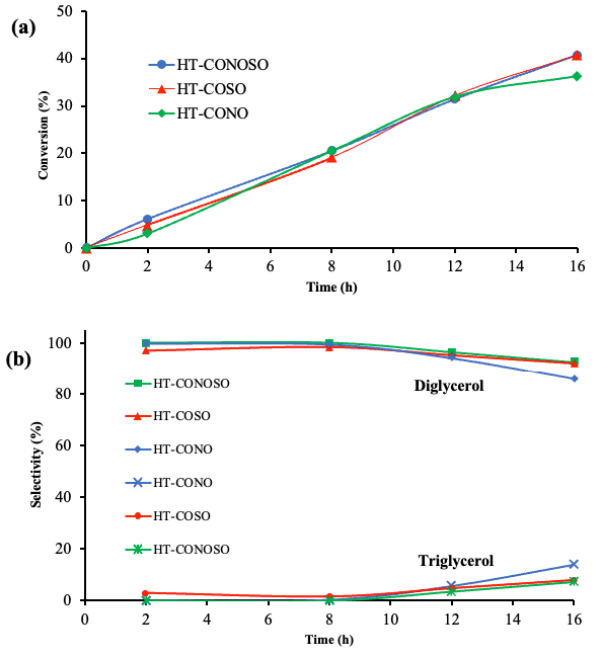


FIGURE 1. (a) The catalytic transformation of glycerol into polyglycerols and (b) the time-dependent selectivity trends for diglycerol and triglycerol during the etherification process, utilizing hydrotalcite catalysts synthesized from di-anion and tri-anion sources.

The alterations made to the anion sources of HT resulted in a more precise reaction favoring diglycerol, as depicted in Figure 1(b). Over the course of the 16-hour reaction, diglycerol selectivity ranged from 86.04% to 94.32%, following the order HT-CONOSO > HT-COSO > HT-CONO. The tri-anion variant (HT-CONOSO) consistently exhibited superior selectivity for diglycerol. For example, within the initial 8 hours, a conversion rate of 20.46% was achieved with complete selectivity for diglycerol. After the 8-hour point, triglycerol began to form, likely due to the tri-anion HT's surface area being optimally configured for the selective etherification of glycerol into diglycerol. Additionally, the active area of the tri-anion sample was adequate for producing diglycerol, and the pore size also played a role in its formation. These findings indicate that HT with a tri-anion source is the most stable, as it facilitated a selective reaction and could potentially increase the yield of the desired product.

The findings also indicate that the formation of triglycerides was significantly inhibited. This inhibition might be attributed to the suitable pore size of HT, which restricts the reaction, ensuring that only the desired process takes place. The pore size was considered ideal for diglycerol because it allowed only diglycerol to fit, preventing further reactions with larger molecules like triglycerol. Additionally, the issue of internal mass transfer might have resulted in the production of triglycerol solely on the catalyst's external surface. Consequently, this reaction is constrained.

IMPACT OF AMOUNT OF CO_3^{2-} ANION ON CATALYTIC PERFORMANCE OF HT

Given that the tri-anion source HT (HT-CONOSO) achieved the highest conversion rate, it was chosen for further analysis in a follow-up study. The concentration of the primary anion (CO_3^{2-} anion) was subsequently adjusted. This adjustment led to a minor reduction in glycerol conversion when compared to the sample with 50% CO_3^{2-} anion. As illustrated in Figure 2(a), the conversion rates of glycerol dropped to 29.86% and 30.44% for samples with 30% and 70% CO_3^{2-} anion, respectively. The findings indicate that an ideal concentration of 50% CO_3^{2-} anion is necessary for diglycerol, as this level sufficiently exposes the active sites to triglyceride, thereby facilitating the conversion to the desired product.

For HT-CONOSO 70 %, the conversion rate exhibited a steady rise over time, particularly noticeable after the initial 2 hours. During the first 2 hours, there was a marked increase, reaching a conversion of 22.78 %. At the beginning, the unreacted glycerol molecules were effectively drawn into the vacant pores. As the production of diglycerol within these pores increased, they eventually became saturated. This saturation led to a mass transfer challenge, shifting the reaction to occur on the catalyst's outer surface. As a result, the reaction slowed down, leading to a lower conversion rate by the conclusion of the process. The HT-CONOSO 30 % sample displayed a similar pattern.

Figure 2(b) demonstrates that modifying the CO_3^{2-} anion concentration to 30% and 70% led to a notable shift in the selectivity pattern of diglycerol. The selectivity percentages for diglycerol in the two samples were 93.27% and 84.99%, respectively. Nevertheless, during the initial 2 hours of the reaction, there was a noticeable variation in the selectivity trends for diglycerol. In this early stage, the reaction favored the production of triglycerol over diglycerol. This could explain the sharp rise in glycerol conversion observed at the start of the reaction, as more glycerol molecules were transformed into triglycerol. Once the pores were saturated with triglycerol, mass transfer issues became more pronounced, potentially limiting the reaction to the external surface.

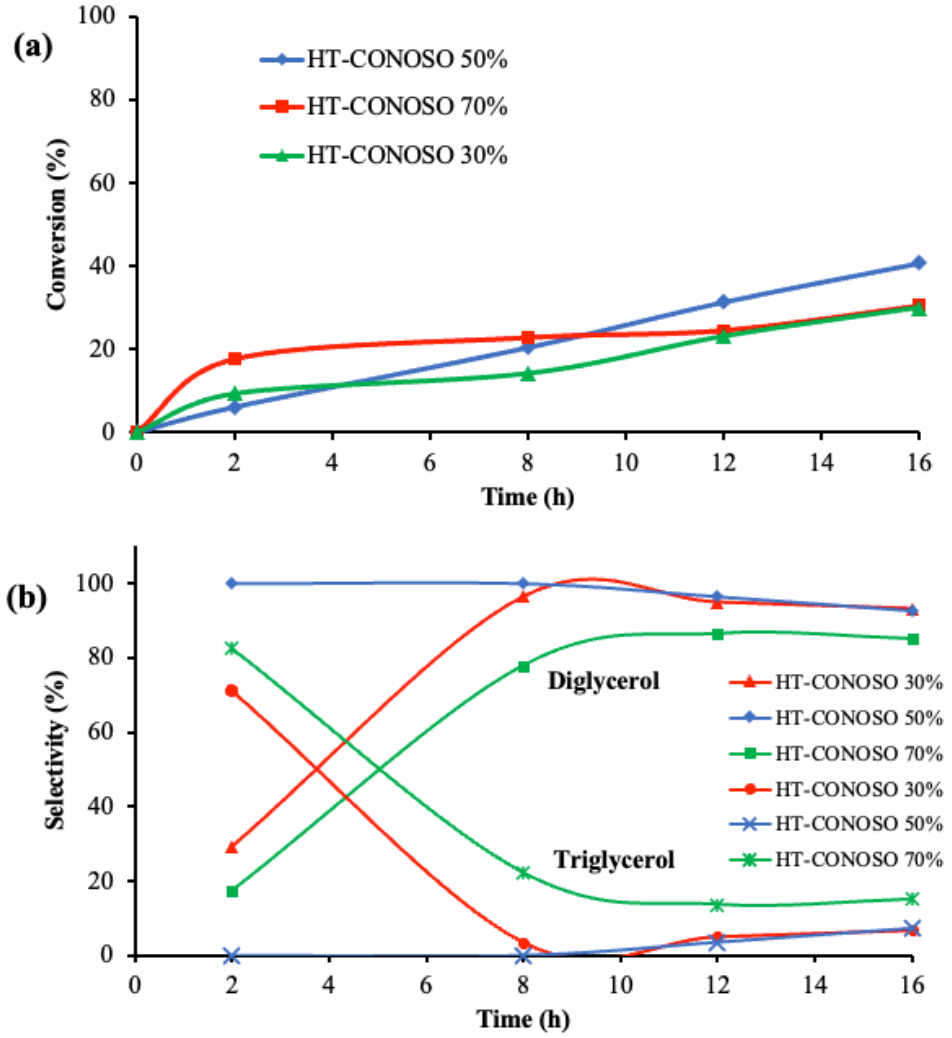


FIGURE 2. (a) Conversion of glycerol into polyglycerols through catalysis and (b) the selectivity trends for diglycerol and triglycerol over time during the etherification process using hydrotalcite catalysts, which were synthesized with varying amounts of tri-anions.

CHARACTERIZATION OF HT CATALYST

As shown in Table 2, all multi-anion HT samples exhibited reductions in the total surface area when compared with HT-CO. The surface area was found to range from 57 m^2/g to 72 m^2/g. Both di-anion samples showed nearly identical surface properties. A slightly smaller reduction was noted for the tri-anion HT. This pattern was also observed when the concentration of CO_3^{2-} anion was altered. For HT-CONOSO 50%, there was a notable decrease in surface area. The presence of multiple anions led to an excess, which could result in anion adsorption within the microchannel, potentially causing blockages. Additionally, changing the amount of CO_3^{2-} anion might lead to an irregular arrangement of anion species in the interstitial position (interlayer).

Additionally, this phenomenon impacts the pore volume. In the case of HT with a single anion source, the pore volume was approximately 0.171 cc/g. However, when additional anions were introduced into the HT interlayer, the pore volume significantly dropped to between 0.03 cc/g and 0.04 cc/g. This reduction could explain the observed decrease in glycerol conversion. On the other hand, the pore diameter of samples containing multiple anions increased compared to the single anion HT, which had a pore diameter of 5.17 nm. The pore diameter for all samples was fairly consistent, ranging from 14.00 nm to 14.16 nm.

The incorporation of two anion sources (di-anion) clearly enhanced the basic sites of HT. Both di-anion HTs exhibited identical basic strength within the range of $15 < H_- < 18.4$. In contrast, the tri-anion HT demonstrated a further increase in basic strength, reaching the range of $18.4 < H_- < 26.5$, despite a variation in the CO_3^{2-} anion quantity. It was noted that HT with multiple anion sources successfully boosted the basic strength compared to a

previously reported study (Xie et al. 2006). However, this could lead to an excessive concentration of anions, where they are unevenly distributed within the pore, causing a sudden reduction in pore volume. This also suggests the increase in triglyceride conversion trend for all anion samples.

The EDX analysis revealed that the elements nitrogen (N) and sulfur (S) were not significantly present in the di- and tri-anion HT samples, with only trace amounts observed. Comparing the HT-CONOSO samples at 50% and 30%, there was a negligible reduction in these elements. However, introducing 70% CO_3^{2-} anion led to a marked decrease, with N at 0.47% and S at 0.34%. These findings imply that the CO_3^{2-} anion predominantly intercalates into the interlayer. In summary, despite some fluctuations in the levels of N and S, these elements are still present, confirming the successful creation of a tri-anion HT system. Nonetheless, further enhancements in anion intercalation are warranted.

TABLE 2. Surface characteristics, basic strength and chemical composition of multi-anion HT

Sample	Total surface area (m^2/g)	Pore diameter (nm)	Pore volume (cc/g)	Basic strength	Chemical composition (%)	
					N	S
HT-CO	127	5.17	0.17	9.3 < H_ < 15.0	0	0
HT-CONO	66	14.16	0.04	15.0 < H_ < 18.4	2.64	0
HT-COSO	57	14.02	0.04	15.0 < H_ < 18.4	0	2.55
HT-CONOSO 30%	31	7.50	0.04	18.4 < H_ < 26.5	1.32	1.23
HT-CONOSO 50%	72	14.00	0.03	18.4 < H_ < 26.5	2.14	1.30
HT-CONOSO 70%	24	6.30	0.04	18.4 < H_ < 26.5	0.47	0.34

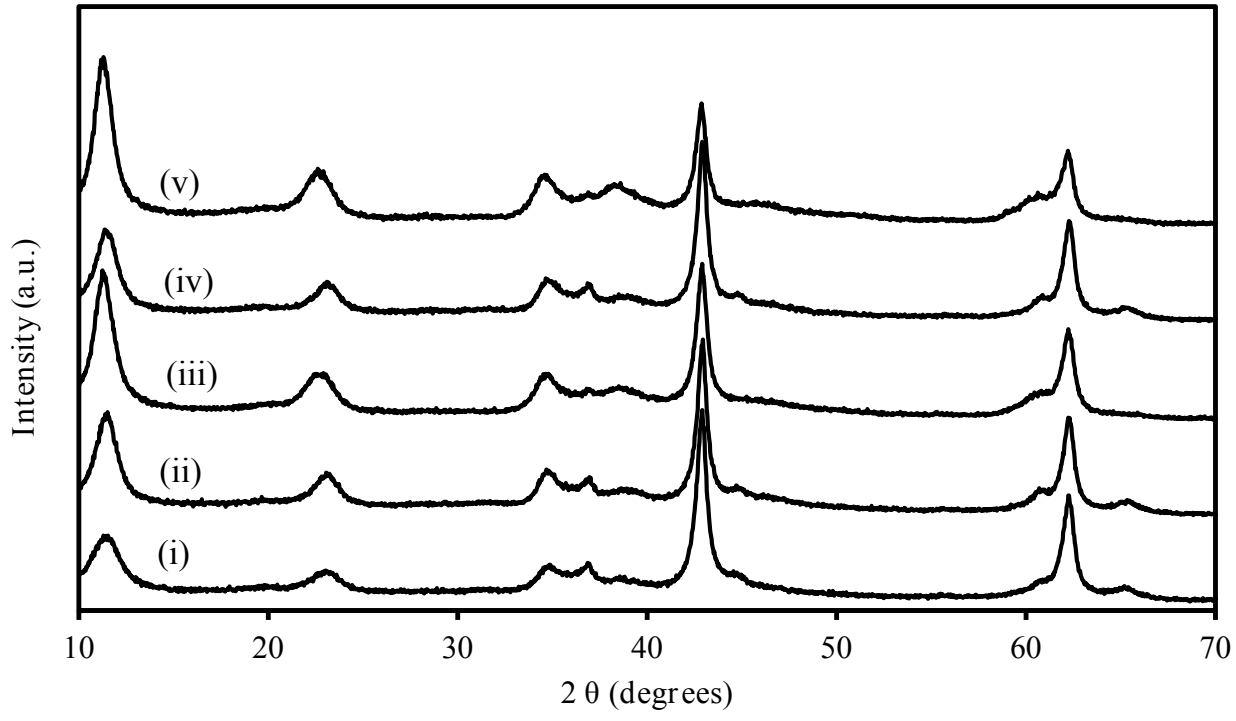


FIGURE 3. XRD patterns of (i) HT-COSO (ii) HT- CONO (iii) HT-CONOSO 30 % (iv) HT-CONOSO 50 % (v) HT-CONOSO 70 % catalysts.

As depicted in Figure 3, the samples display identical peaks that correspond to the HT and MgO periclase phases, with differences only in the intensity of these peaks. The HT phase is characterized by peaks at 11.9°, 23.2°, 34.7°, 38.7°, and 60.8°, whereas the periclase phase shows peaks at 36.9°, 43.0°, and 62.1°. For the di-anion sample and HT-CONOSO 50 %, spinel peaks are detected at 44.5° and 65.3°. The surplus anion added during the recrystallization stage interacts with the MgO phases, which are then redistributed into other phases after calcination. This interaction could lead to a more pronounced presence of MgO.

Altering the CO_3^{2-} anion in the tri-anion HT led to a marked enhancement in the intensity of the HT peaks. Furthermore, the XRD pattern for the HT sample containing 30% and 70% CO_3^{2-} anions showed no spinel peaks at 2θ = 44.5° and 65.3°. This observation suggests that the modification promoted a more uniform synthesis of the HT phase. Additionally, the formation of MgO was diminished, as evidenced by the decreased intensity of the periclase peak. The Al_2O_3 phase peaks for HT-CONOSO at 30% and 70% were more pronounced than those at 50%, indicating that the Al^{3+} cations were less evenly distributed within the MgO structure (Prentice et al. 2020).

CONCLUSION

Efforts to enhance HT were made by incorporating di-anion and tri-anion HT catalysts into the etherification process. This led to the successful synthesis of multi-anion HT. Although the introduction of different anion sources slightly reduced glycerol conversion, it effectively increased diglycerol selectivity to 92.73%. The large pore diameter and small pore volume hindered the further reaction of glycerol to form triglycerol. Additionally, the basic strength improved, with values between 15.0 < H_ < 26.5. EDX analysis confirmed the presence of multi-anions in the HT, detecting small amounts of N and S in each di-anion and tri-anion HT sample.

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DECLARATION OF COMPETING INTEREST

None.

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