International Research Journal on Advanced Engineering Hub (IRJAEH)
e ISSN: 2584-2137
Vol. 03 Issue: 08 August 2025
Page No: 3385-3392
https://irjaeh.com
https://doi.org/10.47392/IRJAEH.2025.0498

A Sustainable Catalysis Process for Converting Acrylic Acid into Methyl Acrylate by Modifying Bentonite with KOH

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Abstract
This study investigated potassium hydroxide (KOH) and bentonite as catalysts for producing methyl acrylate via esterification. Bentonite was impregnated with KOH at 1:20 and 1:4 ratios to examine the effect of varying KOH concentrations. X-ray diffraction (XRD) analysis characterized the catalysts and natural bentonite, revealing that impregnation partially converted KOH to potassium oxide (K2O) during calcination, likely enhancing catalytic activity.A catalytic esterification reaction between acrylic acid and methanol was performed to evaluate the KOH-bentonite's performance. Key parameters, including catalyst ratio, reaction time, reactant molar ratio, catalyst concentration, and reaction temperature, were systematically optimized. The KOH-bentonite catalyst with a 1:4 KOH-to-bentonite ratio demonstrated the highest catalytic activity, achieving a 94% conversion of acrylic acid to methyl acrylate. This optimal conversion was attained under specific conditions: a 3-hour reaction time, 10% catalyst concentration, a 1:4 molar ratio of acrylic acid to methanol, and a temperature of 160°C. XRD also confirmed the bentonite's crystal structure remained largely intact after impregnation.These findings confirm KOH-bentonite as an effective solid base catalyst for methyl acrylate production. Future research could focus on improving catalyst reusability, stability, and process scalability to advance a more sustainable and efficient esterification process.
Keywords: *Impregnation; KOH-bentonite, Methyl acrylate, Potassium oxide (K2O), XRD analysis.*

1. Introduction
Green catalysts are the subject of research due to the need for inexpensive and ecologically friendly chemical processes. The production of esters, such methyl acrylate, which is utilized in paints, adhesives, and textiles, is frequently accomplished in industries through esterification processes. However, conventional catalysts utilized in these reactions are frequently costly, non-reusable, and corrosive.In this work, we used potassium hydroxide (KOH) and bentonite clay to create a catalyst. Bentonite is a naturally occurring clay that is ideal for catalyst development because of its high cation exchange capacity and good surface area. Strong bases like KOH can improve esterification reactions when they are supported on bentonite. Methyl acrylate was created by esterifying acrylic acid with methanol using the catalyst.The purpose of this work is to prepare and investigate KOH-bentonite catalysts in a variety of ratios, including 1:20 and 1:4, in order to assess their effectiveness as catalysts. The study focuses on increasing reaction efficiency and selectivity and inexpensive. The study is interesting since it optimizes the KOH loading on bentonite and evaluates its effect on the catalyst's performance.

1.1.Materials
Bentonite was purchased from S. D. Fine Chemicals, and potassium hydroxide was obtained from Molychem Pvt. Ltd. To clean the bentonite, it was first soaked in 30% hydrogen peroxide at 30°C for 24 hours (2:1 ratio of bentonite to peroxide). Then, it was gently heated in a boiling water bath to remove extra peroxide. After washing and drying at 110°C, the bentonite was ground into a fine powder. Methanol and acrylic acid were used as raw materials for the reaction. The prepared KOH-bentonite catalyst helped speed up the reaction and improve the product yield.

1.2.Catalyst Preparation
1.2.1. Cleaning and Drying of Bentonite
The bentonite was cleaned by immersing it in 30%

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hydrogen peroxide at 30°C for 24 hours (2:1). The excess peroxide was then removed by heating it in water. The mixture was then cleaned with distilled water (1:4), allowed to settle, and the water was drained. It was then dried at 110°C to reduce moisture to 10%, and then ground into powder form.Methanol and acrylic acid were used as reactants. KOH supported on bentonite was employed as a catalyst to increase reaction speed and output.[1][2][3]

1.2.2. Materials and Experimental Setup

The catalyst was produced by mixing high-quality bentonite with KOH. Bentonite has good ion exchange properties, and KOH acts as the active catalyst. A 500 mL conical flask containing a reflux condenser and magnetic stirrer was used. The system made it easier to control temperature and mixing, which are critical for good catalyst preparation. To ensure uniformity in the catalyst mixture, the preparation process was repeated three times.

1.2.3. KOH Impregnation Process

The catalyst's effectiveness is determined by the degree to which KOH is placed onto bentonite. Inadequate temperature or pH management may result in uneven distribution or low activity. A proper KOH concentration prevents overloading or under-loading. The bentonite was mixed with KOH in various ratios, including 1:20, 1:5, and 1:4.These ratios influence catalyst characteristics. A larger ratio (1:4) enhances reaction pace, but a lower ratio (1:20) promotes selectivity.For 24 hours, the mixture was stirred at 60 degrees Celsius. Constant stirring helped to properly distribute the KOH. This enhances the reaction and decreases unwanted byproducts, making the process more environmentally friendly and less wasteful.

1.2.4. Drying and Removal of Water

After impregnation, the slurry was vacuum-filtered to remove water. Then it was dried in the oven approximately 5 hours. This step ensures that no moisture remains, which could compromise catalyst performance. Proper drying also maintains structural stability. After drying, the catalyst was prepared for thermal treatment to increase its activity.

1.3.Calcination of The Catalyst

Calcination is the final step, in which the dried KOH-bentonite catalyst is heated to 400°C in a muffle furnace for 5 hours. This process eliminates any remaining organic materials and stabilizes the KOH within the bentonite. Heat increases surface area, thermal stability, and reactivity. The end result is a powerful and efficient catalyst that works well in the esterification of acrylic acid and methanol to form methyl acrylate. This catalyst is also stable in acidic and basic environments, making it appropriate for repeated industrial application. However, several difficulties, such as prolonged deactivation or excessive energy consumption while heating, must be addressed.[4][5]

1.4.Performance and Application of the Catalyst

The step-by-step preparation method develops a catalyst with high activity. It accelerates the esterification of methanol and acrylic acid, resulting in more efficient methyl acrylate production. To produce a high-quality catalyst, every step was carefully controlled, including material selection, impregnation, drying, and calcination. The finished catalyst has a large surface area and good porosity. It works well in the reaction and is inexpensive, non-toxic, and biodegradable. These properties make KOH/bentonite catalysts ideal for industrial applications in sustainable chemical manufacturing.

2. Material and Methods

2.1.Characterization of Catalysts

The powder X-ray diffraction patterns of bentonite and KOH/bentonite catalysts were determined using a Rigaku Miniflex Goniometer at 30 kV, 15 mA, and 0.01° steps. To identify bentonite and catalysts, a comprehensive understanding of their crystal structures is required. As catalysts are prepared, an XRD pattern can be used to identify phase changes, crystallinity, and structural modifications. A diffraction pattern indicates how KOH impregnation and calcination impact bentonite structural properties. To fully understand the results, XRD data must be interpreted by identifying characteristic peaks, determining phase composition, and examining any shifts and changes in peak intensity and position. These patterns can be compared to conventional reference patterns for bentonite and KOH to further understand structural changes.

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Additionally, comparing XRD results to KOH/bentonite catalyst performance provides the light on how chemical transformations affect the catalyst's efficiency during the esterification reaction for methyl acrylate production. [6][7][8]

2.2.Esterification of Acrylic Acid

For esterification, we used a 250 mL round-bottom flask with three necks, a reflux condenser, a temperature indicator, and a magnetic stirrer needle. The reactor was heated in a regulated hot oil bath. A defined volume of methanol was added with a known amount of catalyst (3–27 g). Once the mixture had achieved the desired temperature of 90 °C, it was fried in an oil bath at a controlled temperature. Acrylic acid esterification was carried out in a 250 mL three-neck round-bottom flask with vigorous stirring (at 350 rpm).A reflux condenser minimizes reactant evaporation, a temperature indicator allows precise temperature control, and a magnetic stirrer thoroughly combines reactants. A hot oil bath heater kept the temperature of the flask constant and correct while the reaction continued.According to the experimental criteria, an appropriate quantity of methanol was mixed with a specific amount of catalyst, ranging from 3 to 27 grams. It was then heated in an oil bath at 90°C until it reached that temperature. When the mixture reached the specified temperature, acrylic acid was added. The mixture was continuously stirred at 350 rpm during this addition to ensure uniform mixing and an effective reaction. As a result of controlling the temperature of the oil bath and maintaining consistent stirring conditions, acrylic acid and methanol were efficiently converted into ester in an efficient manner. During the reaction, acrylic acid was mated with methanol at a molar ratio of 1:4 for one to three hours.

2.3.Determination of Methyl Acrylate Produced

This study used volumetric titration to investigate the composition of methyl acrylate produced by the esterification of acrylic acid and methanol with Bentonite as a solid catalyst. The esterification reaction converts acrylic acid in the following way: % Conversion = (moles of A reacted / moles of A fed) × 100, where A is acrylic acid. To compute the % conversion, first find the initial moles of acrylic acid (A0) using the following formula: Moles of A0 = Molecular Mass/Mass of Acrylic Acid. To calculate the moles of unreacted acrylic acid, use the volumetric titration formula: Moles of Unreacted A = Titrant Concentration × Titrant Volume × Stoichiometric Ratio.Calculate the moles of acrylic acid that reacted by subtracting the moles of acrylic acid that did not react from the initial moles. Finally, the percentage conversion is calculated by applying the formula: % Conversion = (Moles of A reacted / Moles of A fed) × 100.[9]

3. Results and Discussion

3.1.XRD Measurement of Bentonite and Catalyst

As a result of the impregnation process, potassium hydroxide (KOH) molecules were introduced into the pores of bentonite, reducing the catalyst's surface area. With a higher KOH-to-bentonite ratio, KOH filled more pores on the catalyst, reducing its surface area significantly. As a result, KOH is likely to be converted to potassium oxide (K2O) during calcination. This process may have been facilitated by the presence of KOH on its surface as well as within the pores.As shown in figure 1, KOH/bentonite catalysts with ratios of 1:10 and 1:10 exhibit X-ray diffraction (XRD) patterns that differ from raw bentonite in terms of D100 as shown in figure 1.The catalyst's crystallinity improved with higher KOH loading. The catalyst's XRD pattern resembled that of raw bentonite at 1:20, but a new K2O phase was discovered. With a 1:20 KOH ratio, the catalyst had a D100 value of 1.67 nm. It may be inferred that, while the structure of the bentonite remains recognizable, KOH is not equally distributed across its surface. This clearly indicates that the surface or structure of the bentonite did not distribute KOH evenly, leading to the emergence of K2O. According to the XRD patterns, there are reflections observed at 31°, 39°, 51°, 55°, and 62° angles, which are consistent with the K2O phase. This indicates that the KOH phase was then calcined to become the K2O phase.In this study, it is found that KOH loading ratios influence both the surface area of the bentonite catalyst as well as its ability to produce K2O.

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Therefore, it is critical to carefully control KOH loading ratios to ensure that the catalyst has the best surface area and the highest K2O formation. This will result in a better performance and a more efficient catalyst. Figure 1 Shows XRD Measurement of Bentonite and Catalyst.

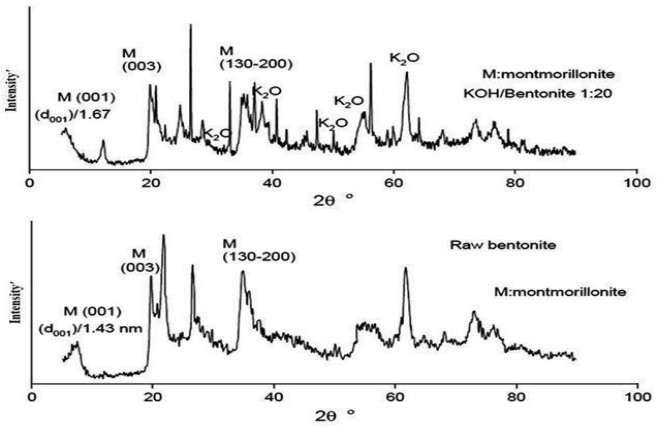

Figure 1 XRD Measurement of Bentonite and Catalyst

3.2.Catalytic Evaluation of KOH/Bentonite

To investigate the effects of the KOH/bentonite catalyst on the esterification of acrylic acid, a series of experiments were conducted. It was observed that incomplete miscibility at 90°C hindered the esterification process, which hindered the reaction. Because of this, the stirrer speed was adjusted and maintained at 350 rpm in order to improve the dispersion of the mixture and its overall reaction. As a result of the higher stirring speed, better mixing of the reactants and catalyst was achieved, which resulted in higher conversion rates and a higher yield of the desired product due to the better mixing process.[13]

3.2.1. Effect of Molar Ratio

A modified KOH/Bentonite catalyst was used during the esterification process to assess the impact of the molar ratio of acrylic acid (AA) to methyl acrylate (MA) on catalytic activity. As the acrylic acid to methanol ratio increased from 2:1 to 1:4, the conversion of acrylic acid was significantly improved. The results indicate that the molar ratio of acrylic acid to methanol improved significantly with increasing molar ratios of AA to MA. According to Figure 2, the conversion rate increased from 65% at a 2:1 ratio to 91% at a 1:4 ratio (Fig. 2), suggesting that acrylic acid can be converted to methyl acrylate with greater efficiency when methanol is added to it. As the ratio was adjusted to 1:6, the conversion rate remained stable or even decreased slightly, but further increases in the molar ratio did not result in substantial improvements. Despite an increase in methanol-to-acrylic acid ratio, the conversion of acrylic acid decreased to 84% at a ratio of 1:16.The 1:4 molar ratio of acrylic acid to methanol yielded the highest conversion rate of 91%, making it the most effective ratio for maximizing the production of methyl acrylate among the tested ratios. Figure 2 Shows Effect of Molar Ratio.[10][11][12]

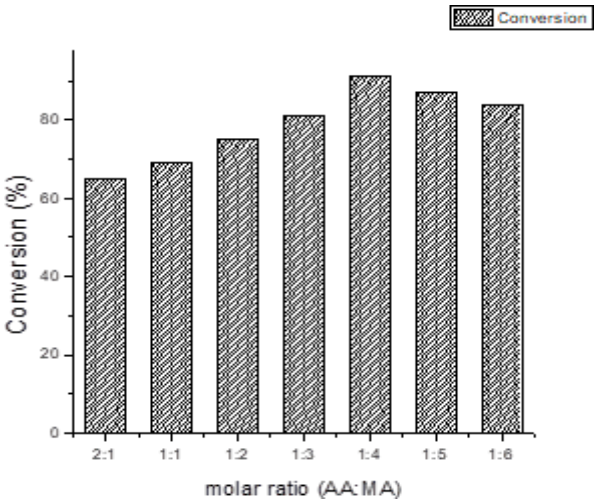

Figure 2 Effect of Molar Ratio

It was therefore decided that a 1:4 molar ratios would be optimal for the subsequent reactions. According to the study, the esterification process was most efficient at a molar ratio of 1:4 (acrylic acid to methanol), with an overall conversion rate of 91%. In order to ensure optimal KOH/Bentonite catalytic performance, this ratio was used for all subsequent reactions.

3.2.2. Effect of Catalyst Loading

The aim of the present study was to investigate the effects of catalyst loading on the esterification reaction by varying the catalyst loading from 2% to 16% by weight in comparison to acrylic acid (AA). When the catalyst loading was increased from 2% to

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10%, a significant increase in acrylic acid conversion was observed. With 2% catalyst loading, the conversion rate increased to 94% when the catalyst loading was increased by 10%. As a result, it indicates that adding more catalysts up to a certain point will increase reaction efficiency, likely by creating more active sites for esterification. During the experiment, the conversion rate of acrylic acid decreased when the catalyst was loaded at 10%. When the catalyst loading was increased to 16%, the acrylic acid conversion rate dropped from 94% to 71%. Thus, excessive catalyst loading can reduce the reaction's efficiency. As a result of high concentrations of catalyst, over aggregation or deactivation of active sites can occur, reducing the conversion rate. Based on these findings, it has been determined that a catalyst loading of 10% is optimal when it comes to achieving the highest conversion rate of acrylic acid. Following the experimentation, this optimal catalyst loading was used in order to ensure that the esterification process would be as efficient as possible. Figure 3 Shows Effect of Catalyst Loading

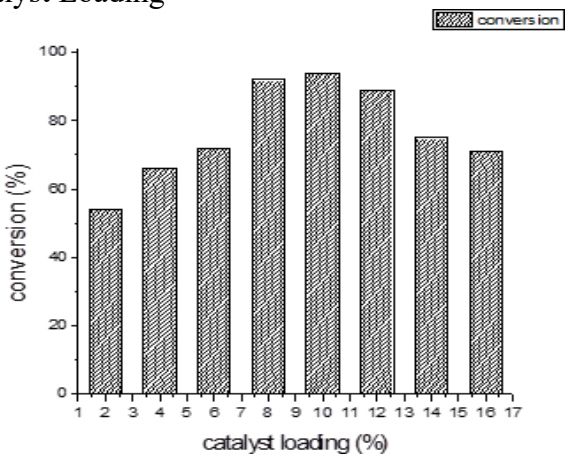

Figure 3 Effect of Catalyst Loading

In order to achieve this optimal loading, the amount of catalyst required to drive the reaction efficiently must be balanced with the avoidance of excessive catalyst concentrations.[14][15]

3.2.3. Effect of Reaction Time

To evaluate the catalytic performance of KOH/Bentonite, a detailed study was conducted to assess the effect of reaction time and temperature on acrylic acid conversion to evaluate the catalytic performance of KOH/Bentonite. A 200 minute extension of the reaction time was used to examine the impact of reaction time. The results indicated that over time acrylic acid conversion rates increased significantly, with significant improvements observed up to 100 minutes after the test. The rate of increase subsequently became marginal after this period had ended. The results showed that the conversion rate of acrylic acid improved from 79% at the shorter times to 94% at 180 minutes, which indicates that 180 minutes is the optimal reaction time to achieve the highest conversion of acrylic acid. In addition to reaction time, the effect of reaction temperature has also been investigated. The temperature was increased from 90°C to 160°C while keeping the reaction duration constant at 120 minutes. As a result of higher temperatures, there was a significant increase in acrylic acid conversion from 47% at 90°C to 94% at 160°C. Figure 4 shows Effect of Reaction Time

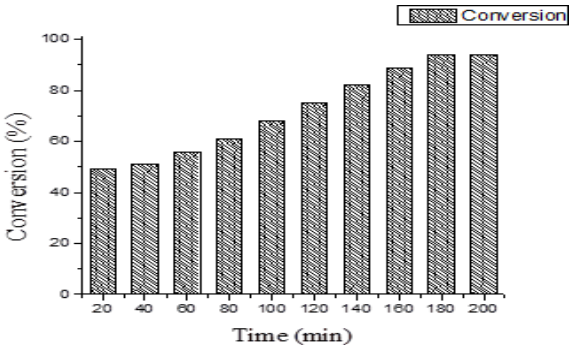

Figure 4 Effect of Reaction Time

This result highlights how temperature plays a critical role in the optimization of the reaction. In this study, the significant improvement in the conversion rate at 160°C is demonstrated as evidence that this temperature is ideal for maximizing the conversion of acrylic acid into acrylamide at this temperature. The results of this study show that reaction times as well as temperatures play a crucial role in maximizing the performance of KOH/Bentonite catalysts in general. In order to achieve the highest conversion rate of acrylic acid, a reaction time of 180 minutes and a temperature of 160°C are the optimal conditions. For catalytic processes to operate at the maximum

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https://doi.org/10.47392/IRJAEH.2025.0498

efficiency, it is extremely important to control the reaction parameters carefully. Under these optimized conditions, the results demonstrate that it is possible to obtain efficient esterification reactions using a KOH/Bentonite catalyst under these optimized conditions.[16][17][18].

3.2.4. Effect of Reaction Temperature

During a period of 120 minutes, the reaction temperature was increased from 90°C to 160°C and the conversion rate of acrylic acid significantly improved. The conversion rate of acrylic acid at 90°C was only 47% at the initial temperature, suggesting that the reaction was not proceeding efficiently under such conditions. This indicates that the reaction was not proceeding efficiently under these conditions. When the temperature was increased to 160°C, the conversion rate increased significantly to 94% (Fig. 4). As a result of this notable improvement, it could be concluded that the higher the temperature, the more effective the reaction is.Several factors contribute to the improvement in conversion rate with higher temperatures. For example, higher temperatures generally increase the kinetic energy of molecules, which in turn leads to more frequent, energetic collisions between reactants at higher temperatures. As a result of this increased molecular activity, the esterification process is enhanced, resulting in higher levels of acrylic acid conversion as a result. Additionally, elevated temperatures can be beneficial to the conversion process because they reduce the activation energy required for the reaction, which further speeds up its progress.In this study, it is demonstrated that 160°C is the optimal temperature for the maximum conversion of acrylic acid with a 94% conversion rate. As a result, this finding emphasizes the importance of optimizing reaction conditions in order to achieve efficient chemical transformations. It is important to keep the reaction temperature at 160°C so that the conversion of acrylic acid is maximized, leading to increased yields and improved synthesis efficiency.Ultimately, the experiment revealed that increasing the temperature from 90°C to 160°C significantly increased acrylic acid conversion from 47% to 94%. Figure 5 shows Effect of Reaction Time

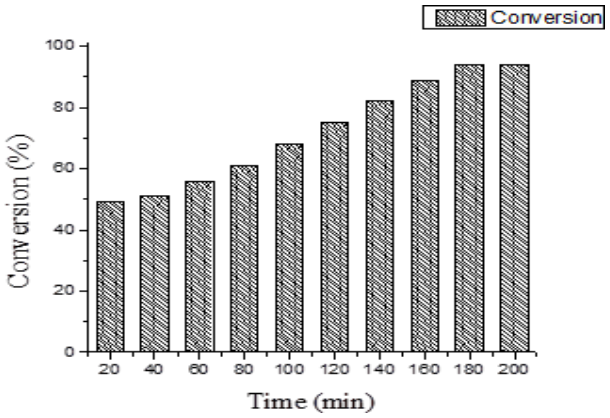

Figure 5 Effect of Reaction Time

This result highlights the importance of temperature control in optimizing chemical reactions and achieving desired conversion rates. For maximizing the efficiency of the reaction and the yield of the final product, a temperature of 160°C is crucial[19][20].

Conclusion

It has been shown that the KOH-Bentonite catalyst is able to synthesize acrylic ester via esterification with higher efficiency than traditional bentonite clay at synthesizing acrylic ester. In order to evaluate the efficiency of this hybrid heterogeneous catalyst, specifically the KOH-Bentonite formulation with 5% KOH, a series of tests were conducted. The study involved systematic adjustments to reaction time, temperature, molar ratios, and catalyst loading to determine the optimal parameters that would maximize the efficiency of the reaction. A preliminary study was conducted to determine the effect of reaction time on the conversion of acrylic acid into methyl acrylate. Reaction times were varied between 20 minutes and 200 minutes, and the results indicated that with increased reaction times, the conversion efficiency significantly improved. After 180 minutes, the acrylic acid conversion rate increased gradually, reaching a peak of 94%. However, the further extending reaction time only resulted in minimal gains in conversion beyond this point. Based on these results, it appears that 180 minutes has been determined to be the optimal reaction time in achieving the highest conversion rate; this indicates that prolonged reaction times beyond this period do not have a significant impact

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https://doi.org/10.47392/IRJAEH.2025.0498

on the outcome. During the study, the temperature was also a crucial factor in the process of converting acrylic acid into a solid. It was observed that the conversion rate of acrylic acid improved substantially with the increase of temperature from 90°C to 160°C while maintaining the reaction time constant at 120 minutes. At 90°C, the conversion rate was only 47%, but when the temperature was raised to 160°C, it improved to 94%. As a result of this substantial increase, it can be concluded that 160°C is the ideal temperature for maximizing conversion efficiency, as it promotes a greater level of molecular activity, reduces activation barriers, and enhances the rate of the overall reactions.The molar ratio of acrylic acid to methanol also played a vital role in determining the reaction efficiency. Various ratios of acrylic acid and methanol were tested, ranging from 2:1 to 1:5. This study found that a 1:4 ratio of acrylic acid and methanol produced the highest amount of methyl acrylate. Based on this result, it is clear that a ratio of 1:4 is the optimal ratio to achieve the highest conversion efficiency. Ratios outside this range did not perform as effectively, confirming that precise control of the molar ratio is essential for optimal esterification.As a second parameter, catalyst loading was a significant factor influencing the reaction outcome. In this study, the catalyst loading was varied from 2% to 20% relative to the limiting reactant. It was found that a 10% catalyst loading resulted in the maximum yield of methyl acrylate and achieved a 94% conversion rate. Thus, in order to ensure efficient performance, catalyst loading needs to be carefully optimized. There is a possibility that an insufficient catalyst loading could lead to incomplete reactions, while an excessive catalyst loading may result in uneconomical and inefficient reactions.As a conclusion, the KOH-Bentonite catalyst has proven to be an extremely efficient and environmentally friendly option for esterification processes as well as an extremely environmentally friendly one. According to the study, a 10% catalyst loading, a reaction temperature of 160°C, a 180-minute reaction time, and a 1:4 acid-to-alcohol ratio were found to be the optimal conditions for achieving maximum performance with this catalyst. Using these conditions, it is possible to convert acrylic acid to methyl acrylate very effectively and efficiently. The results of the study demonstrate that the KOH-Bentonite catalyst has the potential to be a sustainable and efficient catalyst for industrial applications involving esterification in the near future.

Acknowledgements

I would like to express my heartfelt gratitude to Gharda Institute of Technology, Lavel, for providing the required consumables and laboratory support for carrying out this research work.I am deeply thankful to my research guide, Dr. Ujwal D. Patil,KBC-NMU Jalgao(Maharashtra) for his kind guidance, valuable suggestions, and continuous encouragement throughout the study.I also sincerely appreciate the support and help provided by Miss Sanjana Pawar and Miss Disha Kamble during the experimental work. Their assistance was truly helpful in completing this work successfully.

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