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Epoxidation of Cooking Oil Using Natural Zeolite as Heterogeneous Catalyst Via Peracids Mechanism

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ABSTRACT

The increasing demand for sustainable and environmentally friendly materials has directed attention towards the epoxidation of vegetable oils for biopolymer and plasticizer production. This study investigates the use of natural zeolite as a heterogeneous catalyst in the epoxidation of cooking oil via the in situ peracids mechanism. The epoxidation was carried out using hydrogen peroxide and formic acid at 80°C, with varying catalyst loadings to examine their effect on oxirane yield and reaction kinetics. The oxirane oxygen content (OOC) was monitored through titration, while Fourier Transform Infrared Spectroscopy (FTIR) confirmed structural changes in the oil. Results showed that 0.25 g of natural zeolite yielded the highest relative conversion to oxirane (RCO) of 68.6% within 10 minutes, with higher catalyst amounts resulting in lower efficiency due to possible side reactions. FTIR analysis revealed the successful conversion of unsaturated fatty acids into epoxides, as indicated by the disappearance of alkene (C=C) peaks and the appearance of epoxy (C-O-C) peaks. The study confirms the catalytic potential of natural zeolite as a greener and reusable alternative to conventional homogeneous catalysts, supporting sustainable practices in bio-based chemical processes.

Keywords: *Epoxidation, corn oil, oxirane ring, kinetic study*

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1. INTRODUCTION

The epoxidation of cooking oil has become a crucial step in the production of bio-based materials, offering sustainable alternatives to petroleum-derived products. As global industries increasingly focus on environmentally friendly practices, the demand for efficient and green chemical processes has never been greater [1]. According to Wai *et al.* [2], epoxidized vegetable oils play a vital role in the production of plasticizers, stabilizers, and biopolymers. It also contributes significantly to reducing the reliance on non-renewable resources. The catalyst selection is important to this process performance since it directly influences reaction efficiency, product yield, and environmental impact [3]. Sulfuric acid, a widely used homogeneous catalyst, offers high reactivity but poses challenges such as product contamination and separation difficulty. On the other hand, heterogeneous catalysts like natural zeolite provide recyclability and reduced contamination risks, aligning with sustainable production goals [4] [5].

In the epoxidation of oils, catalyst efficiency plays a vital role in optimizing conversion rates and ensuring sustainable industrial applications. The use of natural zeolite as a heterogenous catalyst in the epoxidation of vegetable oils has been explored extensively due to its eco-friendly and reusable nature. The findings journals demonstrated the effectiveness of natural zeolite in the in situ epoxidation of oleic acid, highlighting its catalytic activity in promoting high epoxide yields under mild reaction conditions [5]. Furthermore, natural zeolite not only facilitates cleaner production methods but also offers enhanced sustainability by minimizing the generation of harmful by-products [6]. The catalytic reaction in the epoxidation process using natural zeolite and sulphuric acid as a catalyst has been studied to understand their different benefits and mechanisms [7]. Natural zeolite, a heterogeneous catalyst, improves reaction efficiency and is environmentally friendly because it can be reused and is not corrosive [8]. On the other hand, sulphuric acid is a homogeneous catalyst, was found to be highly effective and produce better epoxide yields, but it is corrosive and creates waste that needs careful disposal [9]. The comparison for both types of catalyst found that natural zeolite is better for the environment and reusable, while sulphuric acid works faster under specific conditions [10].

Despite extensive studies on natural zeolite as a heterogeneous catalyst for vegetable oil epoxidation, the effect of catalyst loading on oxirane formation under in situ peracid conditions remains insufficiently explored. Therefore, this study investigates the epoxidation of cooking oil using natural zeolite with hydrogen peroxide and formic acid. The influence of catalyst loading on oxirane yield and reaction performance is evaluated, while FTIR analysis is used to confirm epoxy ring formation.

Overall, natural zeolite is more sustainable and eco-friendly option compared to sulphuric acid. The choice of catalysts, which affects reaction efficiency, selectivity and sustainability, has a big impact on this problem. Homogeneous catalysts, such as sulphuric acid, are typically used as due to their high catalytic activity. However, their use is associated with challenges, such as environmental issues, difficulties in separation, and product contamination. On the other hand, heterogeneous catalysts, such as natural zeolites provide benefits including lower contamination risks, ease of separation, and reusability which are align with sustainable industrial practices [11][12].

2. EXPERIMENTS

2.1 Materials

Cooking oil that we use for this study is palm oil from EcoSafa. The Oxidising agent is 30- 32% hydrogen peroxide brand from Chemiz. The oxygen carrier is 85% formic acid brand from Chemiz, and the acetic acid is 100% glacial brand from Chemiz. In titration process and boost its efficiency, catalysts such sulphuric acid 96–97% brand from Chemiz, and catalyst of NaOH and HCl is used for this process. For the titration procedure, this process used crystal violet and hydrogen bromide 48-50% brand from Chemiz.

2.2 Epoxidation method

The process began by preparing a water bath on a hot plate to maintain consistent reaction conditions as shown in Figure 1. A measured amount of 50 g of cooking oil was mixed with Hydrogen Peroxide and Formic Acid with the molar ratio 1:1:1. A magnetic stirrer was added to ensure the mixture was well blended, and the beaker was set in the water bath, with the temperature maintained at 80°C and stirring at 350 rpm for uniformity. In this experiment the temperature and stirring need to be monitored to make sure it maintained at 80°C and stirring at 350 rpm to monitor the progress, samples were taken every 10 minutes, mixed with hydrogen bromide, and analyzed through titration to measure the oxirane content. This step helped assess the reaction's effectiveness and the impact of different catalyst concentrations.

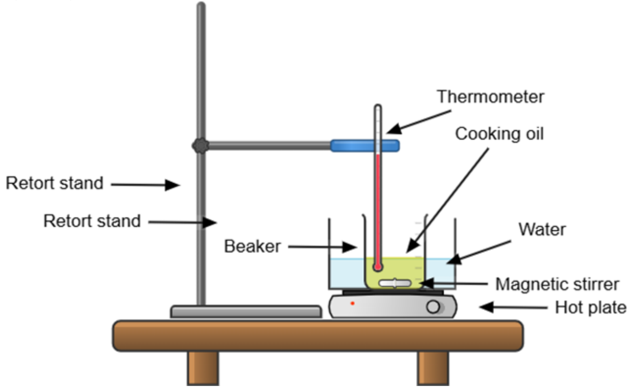

Figure 1. Apparatus set up for epoxidation process

3. RESULTS AND DISCUSSION

3.1 Effect of Heterogeneous Catalyst (Natural Zeolite) in Cooking Oil Epoxidation

The data analysis for the epoxidation of cooking oil using natural zeolite as a heterogeneous catalyst focuses on evaluating the relative conversion to oxirane (RCO%) across varying catalyst weights (0.25 g, 0.50 g, 0.75 g and 1.0 g) and reaction times at (10 to 70 minutes). By analysing the experimental oxirane oxygen content (OOCexp) and compare it with theoretical oxirane oxygen content (OOCtheo), the reaction efficiency was determined. The optimal conditions were identified based on the maximum RCO achieved at specific catalyst weights and reaction time. This analysis provides insights into the catalytic performance of natural zeolite, assessing its efficiency in leading the epoxidation reaction and identifying the most effective catalyst concentration for achieving high conversion rates.

From Figure 2, the graph shows the relative conversion to oxirane (RCO%) over reaction time during the epoxidation of cooking oil using varying weights of natural zeolite catalysts weights at 0.25 g, 0.50 g, 0.75 g and 1.0 g. The results show that the catalyst weight significantly influences the reaction kinetics and conversion efficiency. For 0.25 g, the RCO% peaks at approximately 68.6% at 10 minutes, indicating that this is the optimal condition for maximum conversion. As for 0.50 g of natural zeolite catalyst that expressed in Figure 3.2, the RCO% reached a maximum of 68.16% at 10 minutes, showing the comparable initial performance but slightly lower conversion rates at extended reaction times. As for 0.75 g of natural zeolite catalyst, it resulted in a lower maximum RCO of 58.99% at 10 minutes, indicating that higher catalyst amounts might not enhance the reaction efficiency due to potential side reactions. Lastly, at 1.0 g of natural zeolite catalyst, the RCO peaked at 48.53% at 10 minutes and decreased consistently, suggest that excessive catalyst concentration might hinder the performance, that leads to produce the highest oxirane conversion at shortest reaction time.

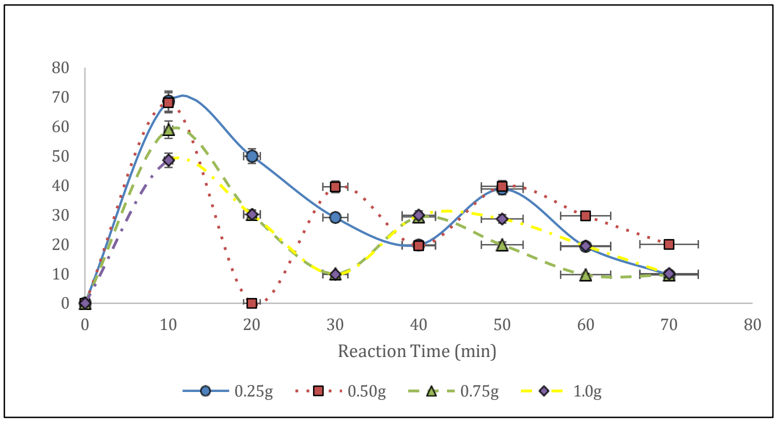

Figure 2. Relative conversion (RCO%) against reaction time (min) for epoxidation of cooking oil with natural zeolite catalyst

3.2 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis helps in identify key functional groups and bonds that make up its framework. Natural zeolite is primarily composed of silica (Si) and alumina (Al) bonds, which form its unique porous structure. These features are crucial for its catalytic performance. The specific vibrations of bonds, such as Si-O-Si and Al-O-Si, which confirm the stability and functionality of the zeolite can be detect by analysing FTIR spectrum. Figure 3 shows the analysis of FTIR spectrum of raw cooking oil against epoxide cooking oil for natural zeolite catalyst.

The hydroxyl group (O-H) peak was observed in the range of (3200-3600 cm^{-1}) in both raw and epoxidized cooking oil. However, its intensity decreased in epoxidized oil, suggesting that some hydroxyl groups reacted with the epoxide during the reaction. This reduction aligns with previous findings that indicate a decrease in free fatty acids upon epoxidation [13]. The alkyl group (C-H) peak, detected between (2800-3000 cm^{-1}), remained strong in both raw and epoxidized oil, implying that the hydrocarbon backbone of the triglycerides remained intact. This result was expected since epoxidation primarily targets the (C=C) bonds rather than (C- H) bonds [14].

A key transformation was observed in the ester carbonyl group (C=O) at range (1700- 1750 cm^{-1}). In raw cooking oil, this peak was prominent due to the presence of triglycerides. However, a decrease in intensity was recorded in epoxidized oil, indicating that some ester groups underwent transformation, potentially due to secondary reactions with the epoxide ring [6].

The alkene (C=C) peak at (1600-1680 cm^{-1}) was strong in raw cooking oil but disappeared in epoxidized cooking oil. These disappearances confirms that the double bonds were successfully converted to epoxide groups, verifying the efficiency of the epoxidation process [15]. The formation of epoxy groups was validated by the epoxy (C-O-C) peak in the range of (850-1250 cm^{-1}), which was absent in raw cooking oil but appeared strongly in the epoxidized sample. The presence of this peak provides direct evidence of epoxidation, demonstrating the conversion of unsaturated fatty acids into oxirane rings [4].

Additionally, the role of natural zeolite catalyst was confirmed by the intensification of (Si-O-Si) range at (1000-1250 cm^{-1}) and (Al-O-Si) range at (450-900 cm^{-1}) peaks in epoxidized cooking oil. These peak indicate the interaction between the epoxide and zeolite framework, suggested that zeolite facilitated the reaction by providing an active surface for oxidation.. In summary, FTIR analysis confirms the successful conversion of unsaturated fatty acids into epoxides, as indicated by the disappearance of the (C=C) peak and the emergence of the (C-O- C) peak. The interaction between the oil and the zeolite catalyst is evidenced by changes in the (Si-O-Si) and (Al-O-Si) peaks. These findings support the effectiveness of natural zeolite as a catalyst in epoxidation reactions.

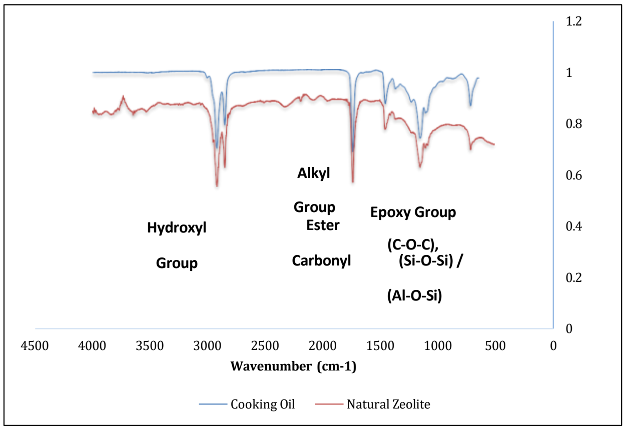

Figure 3. FTIR spectrum of raw cooking oil against epoxide cooking oil for natural zeolite catalyst

4. CONCLUSION

This study demonstrates that natural zeolite is an effective and sustainable heterogeneous catalyst for the epoxidation of cooking oil via the in situ peracids mechanism. Among the catalyst loadings tested, 0.25 g provided the highest oxirane conversion at the shortest reaction time, indicating an optimal balance between catalyst activity and reaction efficiency. FTIR analysis further confirmed the successful formation of oxirane rings and the transformation of unsaturated bonds in the oil. The findings highlight the environmental and practical advantages of using natural zeolite, including reusability, reduced contamination, and alignment with green chemistry principles. This work contributes to the advancement of eco-friendly epoxidation processes and supports the development of renewable resources for industrial applications.

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