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EPOXIDATION OF CATFISH OIL USING ZEOLITE SYNTHESIZED FROM PHU YEN DIATOMITE

D.Q. Do✉, S. M. Hoang, H.T. Tran, Q.C. Khuu, and C. T. Vo
Faculty of Chemical Engineering, Industrial University of Ho Chi Minh, 71423,
Ho Chi Minh City, Vietnam
✉Corresponding author: doquydiem@iuh.edu.vn

ABSTRACT
In this work, catfish oil was epoxidised using a zeolite catalyst synthesised from Phu Yen diatomite. The silica precursor was extracted from acid-treated diatomite via sodium hydroxide calcination, utilizing an optimal NaOH-to-diatomite weight ratio of 1.25:1 at 600 °C. Zeolite formation employed hexadecyltrimethylammonium bromide as a structure-directing agent at pH 12, followed by UV irradiation and hydrothermal treatment at 170 °C for six hours. XRD, SEM, and FTIR analyses confirmed the production of crystalline sodium P-type zeolite with a hierarchical structure and a 48.57 m^2/g surface area. The catalytic performance of zeolite was demonstrated by epoxidising catfish oil triglycerides at 80 °C, resulting in the conversion of carbon–carbon double bonds to epoxide rings. Successful epoxidation was indicated by the appearance of a distinct FTIR band at 833 cm^{-1} and the disappearance of the olefinic band at 3009 cm^{-1}. These findings show that zeolite from natural diatomite is an efficient, low-cost, and sustainable catalyst for converting aquaculture byproducts into valuable chemicals. This research advances SDG 12 by promoting the circular use of minerals and aquaculture residues in green chemistry.
Keywords: Diatomite, Zeolite, Catfish Oil, UVC Irradiation, Hydrothermal Crystallisation.
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INTRODUCTION

The chemical and materials industry relies on petroleum feedstocks for basic chemicals, base oils, resins, and organic intermediates. The IEA estimates that 12–13% of global oil demand comes from petrochemical feedstocks, a share expected to rise as demand for polymers and additives grows.[1] This dependence raises greenhouse gas emissions and oil price volatility, as well as sustainability issues and environmental concerns for the industry.[2] The search for renewable, carbon-neutral feedstocks is growing. Vegetable oils and animal fats have been promising alternatives to petroleum but have limited use due to poor oxidation resistance and thermal stability from unsaturated fatty acids.[3] Epoxidation converts double bonds into epoxy rings, improving these properties and enabling their use in bioplastics, resins, polyols, and additives—thereby reducing reliance on non-renewable resources and adding value to byproducts such as fish fat.[4] Vietnam, a major catfish producer, generates significant by-products such as fish oil, skin, and bones, accounting for about 50–60% of the total fish weight. Rich in protein and lipids, these by-products are valuable renewable resources. Catfish oil, high in unsaturated fatty acids, is now being recognised as a promising feedstock for biofuels, bio lubricants, and epoxidised oils, in addition to its traditional low-value uses. Effective catalysts are crucial for efficient, eco-friendly epoxidation. Zeolites and metal-modified zeolites, known for their stability, tunable properties, and high selectivity, are ideal for green chemical processes due to their low side reactions and good reusability.[5-7] Zeolite synthesis is challenging due to the need for pure materials, stringent conditions, lengthy processes, high costs, and difficulty in controlling structure and composition.[8,9] Thus, finding cheap, abundant, and eco-friendly raw materials is important for advancing zeolite production. Diatomite is a promising, cost-effective natural silica source for zeolite synthesis, offering high SiO_2 content and a porous structure. Zeolites made from diatomite show suitable crystal structures and strong adsorption and catalytic properties.[10] However, its variable purity and mineral composition require pretreatment to ensure consistent zeolite quality. Although interest in diatomite-derived zeolites and bio-oil epoxidation is increasing, few studies have used natural Vietnamese diatomite as a low-cost silica source to synthesize hierarchical sodium P-type zeolite catalysts for epoxidation of catfish oil. The connection between synthesis conditions, structural

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properties, and catalytic performance is still not fully understood. This study synthesises sodium P-type zeolite from Phu Yen diatomite and tests its catalytic performance in catfish oil epoxidation. The work supports SDG-12 by promoting the circular use of natural minerals and aquaculture byproducts to produce sustainably valuable chemicals.

EXPERIMENTAL

Materials

Diatomite from Phu Yen, Vietnam, was ground and treated with a 1:1 mixture of 3 M HCl and 4 M H_2SO_4 at 60°C for 30 minutes to eliminate impurities. The mixture was filtered, then washed until the filtrate reached pH 7. The solid part was dried at 105°C for 2 hours and then calcined at 700°C for 3 hours to yield acid-treated diatomite (TDAT), which was then stored for catalyst synthesis. Aluminium nitrate ($Al(NO_3)_3 \cdot 9H_2O$, 99 %) from Shanghai Macklin (China). Sodium hydroxide (NaOH, 96%) from Tianjin Dingxin Chemical Co., Ltd. (China). Hydrogen peroxide (H_2O_2, 30 %) was purchased from Arkema (China). Sulfuric acid (H_2SO_4, 98 %), hydrochloric acid (HCl, 36-38 %), and hexadecyltrimethylammonium bromide (CTAB, 99 %) from Sinochem (China). Acetic acid (CH_3COOH, 99%) and acetone 99.5% from Xilong, China. Ethyl acetate ($CH_3COOC_2H_5$, 99.5 %) from GHtech (China). Catfish oil from the An Phu Gia company (Vietnam).

Silica Extraction

TDAT (5 g) was mixed with NaOH at various ratios and heated at 400, 500, 600 and 700 °C for 2 hours. Following cooling, each mixture was then stirred in water and incubated for 16 hours. The filtered sodium silicate solution (SS) was then added dropwise to 1% CTAB. After the CTAB solution was adjusted to pH 9, the mixture was stabilised for 16 hours. It was then filtered and washed until the filtrate reached pH 7. The solid was first dried at 100 °C for 2 hours, followed by calcination at 600 °C for 2 hours to get silica.

Zeolite Synthesis

An aluminium nitrate solution (AN) was prepared by dissolving 1.8 g of $Al(NO_3)_3 \cdot 9H_2O$ in 10 mL of distilled water. A sodium silicate solution—prepared by mixing 5 g of TDAT with 6.25 g of NaOH—was added dropwise to a 1% CTAB solution under continuous stirring, together with the AN solution. The resulting mixture was then stirred at 80 °C for 2 hours. The pH was then adjusted to pH values of 10, 11 and 12 using 1 M HCl, followed by incubation under UVC light (254 nm) for 24 hours. Following a 6-hour hydrothermal treatment at 170 °C. The mixture was filtered and then washed until the filtrate reached neutral pH. The resulting solid was dried at 105 °C for 2 hours and subsequently calcined at 600 °C for 5 hours to obtain the zeolite catalyst.

Catfish Oil Epoxidation

A zeolite catalyst of 0.5 g was activated in a mixture containing 20 mL of hydrogen peroxide and 11.2 mL of acetic acid under magnetic stirring for 30 min. Then, 10 g of catfish oil (CFO), pretreated with bentonite to remove impurities, was added to the reaction vessel containing the activated catalyst system, along with 20 mL of ethyl acetate. The reaction was conducted under continuous stirring at 80 °C for 3 hours. Afterwards, the catalyst was allowed to settle, and the reaction mixture was transferred to a separatory funnel for phase separation. The solvent was then removed by rotary evaporation to obtain epoxidised catfish oil (ECFO).

Characterization

The catalyst was characterised for crystallinity by XRD (Shimadzu 6100 diffractometer, Cu Kα radiation), morphology by SEM (LEO 1530), and the surface area was determined by nitrogen adsorption using a Quantachrome Autosorb 1 analyser (Ronkonkoma, NY, USA). The ATR-FTIR (400–4000 cm^{-1}) was used to analyse the structure, composition, and functionality of the catalyst and epoxidised catfish oil.

RESULTS AND DISCUSSION

Effect of Temperature and DA/NaOH Ratios on Silica Extraction

Before zeolite catalyst synthesis, the silica extraction yield from acid-treated diatomite (TDAT) was evaluated using different NaOH/TDAT ratios (0.5:1, 1:1, 1.25:1, and 1.5:1 g/g) and calcination

temperatures (400 °C, 500 °C, 600 °C, and 700 °C). The extracted silica yield was utilised to optimise calcination and alkalization conditions for subsequent zeolite synthesis. The results are shown in Fig.-1 and Fig.-2. As shown in Fig.-1, increasing the calcination temperature from 400 to 600 °C increased the silicate yield from 0.80 g (19.6%) to 1.98 g (39.6%). However, the yield decreased to 1.74 g (34.8%) at 700 °C. The maximum yield at 600 °C is attributed to the effective cleavage of Si–O–Si bonds by NaOH, resulting in the formation of soluble sodium silicate (Na_2 SiO_3).[11,12] There was insufficient thermal energy for this reaction, whereas excessive heating at 700 °C promoted the formation of crystalline quartz and insoluble sodium silicate phases (e.g., $Na_2Si_2O_5$ and $Na_2Si_3O_7$), thereby reducing silica recovery.[13,14] These observations agree with previous studies on Phu Yen diatomite, which reported that moderate thermal activation enhances silica accessibility and removes mineral impurities, whereas excessive calcination leads to less reactive crystalline phases.[15,16] Using the optimum calcination temperature of 600 °C, the effect of the NaOH/TDAT mass ratio was further investigated. As shown in Fig.-2, increasing the NaOH/TDAT ratio from 0.5:1 to 1.25:1 increased the silicate extraction yield from 0.97 g (19.4%) to 2.84 g (56.8%). However, the yield decreased to 1.81 g (36.2%) at a ratio of 1.5:1. Although increasing the NaOH dosage enhanced silica extraction up to the optimum ratio, excess alkali promoted the formation of insoluble silicate compounds, thereby reducing the recovery of soluble silica. This trend is consistent with previous studies on diatomaceous earth.[17,18] Therefore, a calcination temperature of 600 °C and a NaOH/TDAT ratio of 1.25:1 were selected as the optimum conditions for subsequent zeolite synthesis.

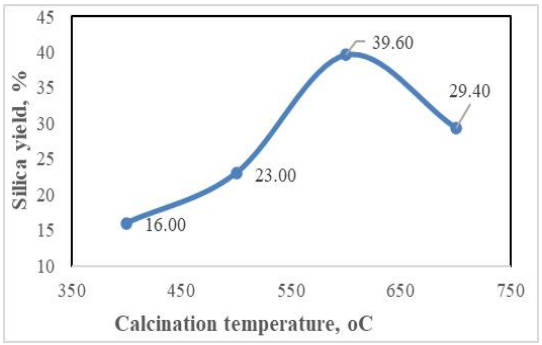


Fig.-1: Impact of Calcination Temperatures on Silicon Yield at the NaOH/TDAT = 1:1 (g/g)

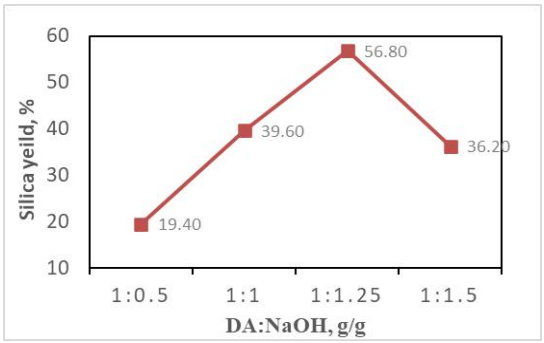


Fig.-2: Impact of the NaOH/TDAT Ratios on Silicon Yield at 600 °C.

Zeolite Characterization

The synthesis of zeolite catalysts from TDAT for catfish oil epoxidation involved three steps: silica extraction, zeolite formation by co-precipitation in 1% CTAB, and crystallisation via hydrothermal treatment at 170 °C for 6 hours. XRD analysis (Fig.-3) showed that the precipitation pH strongly influenced zeolite structure. At pH 10, the product was mostly amorphous with minimal crystallinity. At pH 11, some weak diffraction peaks appeared, indicating partial crystallisation. At pH 12, sharp, high-intensity peaks were observed at 2θ ≈ 12.8°, 18.1°, 22.1°, 28.5°, and 33.8° values characteristic of Na-P zeolite[19], confirming a highly ordered aluminosilicate framework with high phase purity. These results demonstrate that optimal zeolite crystallisation and structure were achieved at a precipitation pH of 12. It is anticipated that the increased crystallinity at pH 12 will increase the number of accessible active sites

and enhance catalytic activity during epoxidation. To further confirm the formation of the aluminosilicate lattice and identify key functional groups, FTIR analysis was conducted on the material samples. FTIR spectra (Fig.-4) exhibit the characteristic absorption bands of the synthesised aluminosilicate zeolite. The broad band at 3600–3400 cm^{-1} is due to O–H stretching of surface hydroxyls and adsorbed water, while the 1630–1650 cm^{-1} band arises from H–O–H bending of physisorbed water. The presence of surface hydroxyl groups can promote the adsorption and activation of polar oxidising species via hydrogen-bond interactions, which may enhance the epoxidation reaction.[20] A strong 1000–1100 cm^{-1} band is attributed to asymmetric T–O–T (Si, Al) stretching, [21] confirming the aluminosilicate framework. Bands at 790–800 cm^{-1} (symmetric Si–O–T stretching) and 450–470 cm^{-1} (T–O bending) further evidence the zeolite structure.[22] The absence of impurity bands suggests high purity. Together with XRD, the FTIR analysis confirms successful Na-P zeolite crystallisation.[23] The aluminosilicate framework may contribute to the improved catalytic performance observed during catfish oil epoxidation.[24] SEM images (Fig.-5) revealed that the zeolite synthesised from Phu Yen diatomite consisted of well-defined polyhedral crystals with relatively uniform particle sizes (5-10 μm), sharp edges, and minimal aggregation. High-magnification images further revealed a hierarchical porous structure composed of aggregated nanocrystallites, which can be attributed to the structure-directing effect of CTAB during crystallisation.[25] Combined with the XRD and BET results, these observations confirm the successful formation of hierarchical Na-P zeolite.[26] The hierarchical porous architecture is expected to enhance mass transport and facilitate the diffusion of bulky triglyceride molecules, while improving access to catalytically active sites.[27] Such characteristics are advantageous for catalytic epoxidation reactions and may contribute to improved epoxidation performance of catfish oil.[16] BET analysis showed that the synthesised zeolite has a hierarchical porous structure with a specific surface area of 48.57 m^2/g and a total pore volume ranging from 0.056 to 0.060 cm^3/g. BJH and NLDFT analyses revealed a predominantly mesoporous structure with pore diameters of 2.6–7.6 nm and a limited fraction of micropores, consistent with SEM observations. This micro/mesoporous structure may enhance active-site accessibility and triglyceride diffusion, supporting improved epoxidation of catfish oil and highlighting the zeolite's catalytic potential.

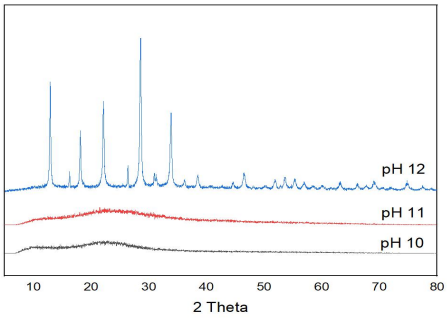


Fig.-3: Zeolite XRD Patterns at pH=12, 11, 10 of the Precipitation Solution

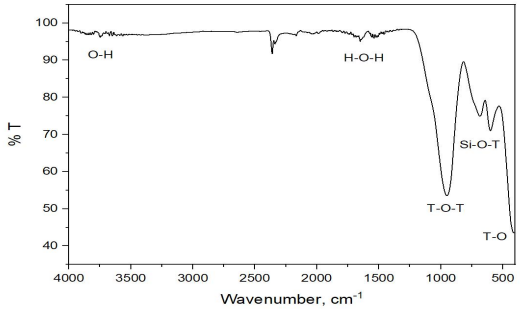


Fig.-4: FTIR Spectra of Zeolites (T = Si, Al)

Evaluation of the Catalytic Activity of Zeolite in the CFO Epoxidation Reaction

After optimising the synthesis conditions and confirming the hierarchical zeolite structure through XRD, FTIR, SEM, and BET analyses, the catalyst's activity was evaluated in the epoxidation of catfish oil (CFO). FTIR analysis was used to identify changes in chemical bonds, directly demonstrating the catalyst's ability to convert C=C double bonds in triglycerides into oxirane rings.

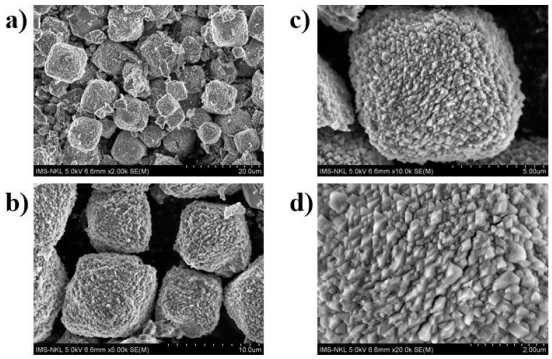


Fig.-5: SEM Surface Morphology of Synthesized Zeolites

Figure-6 compares the FTIR spectra of the original CFO and the epoxidised CFO (ECFO). The FTIR spectrum of CFO reveals the typical absorption bands found in triglyceride oils. A weak band at around 3009 cm^{-1} is attributed to the stretching vibration of olefinic C=C–H bonds present in unsaturated fatty acid chains. There are also strong bands at 2920 and 2850 cm^{-1}, which correspond to the asymmetric and symmetric C–H stretching vibrations of aliphatic $–CH_2$ and $–CH_3$ groups. Additionally, a prominent absorption at approximately 1740 cm^{-1} is associated with the ester carbonyl (C=O) stretching vibration, while intense bands in the 1150–1050 cm^{-1} region are related to ester C–O stretching.[28] After epoxidation, significant changes appeared in the ECFO sample's FTIR spectrum. The notable reduction or disappearance of the olefinic band at ~3009 cm^{-1} indicates the consumption of carbon–carbon double bonds during the reaction.[29, 30] More importantly, the appearance of a new absorption band at ~833 cm^{-1}—characteristic of oxirane ring vibrations—confirms the successful formation of epoxide groups.[31,32] Meanwhile, the ester C=O band (~1740 cm^{-1}) and C–O bands (~1100 cm^{-1}) remained largely unchanged, suggesting that the triglyceride backbone is preserved during the epoxidation process. These spectral changes indicate that the synthesised hierarchical zeolite efficiently catalysed the conversion of carbon–carbon double bonds to epoxy groups while maintaining the triglyceride structure. This highlights its potential as an effective heterogeneous catalyst for converting catfish oil into value-added bio-based intermediates.

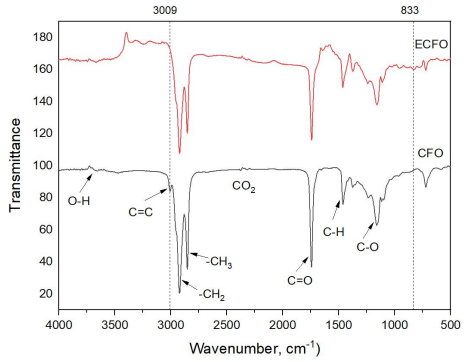


Fig.-6: FTIR Spectra of CFO and ECFO

CONCLUSION

This study developed a process to synthesise zeolite from acid-treated Phu Yen diatomite, using NaOH calcination for sodium silicate production, followed by zeolite formation with CTAB, UVC light, and hydrothermal treatment at 170°C for 6 hours. Optimal silicate extraction was achieved at 600°C with a NaOH: TDAT ratio of 1.25:1. Zeolite crystallisation was optimised at pH 12 under UVC irradiation, producing highly crystalline, hierarchical micro-mesoporous zeolite with a BET surface area of 48.57 m^2/g and uniform 5–10 µm polyhedral crystals, as confirmed by XRD, FTIR, SEM, and BET analyses. The synthesised zeolite efficiently catalysed the epoxidation of catfish oil, as shown by the loss of the olefinic FTIR band at 3009 cm^{-1} and the appearance of the oxirane band at 833 cm^{-1}, while preserving the triglyceride backbone. These results demonstrate that natural diatomite is a low-cost and sustainable precursor for hierarchical zeolite synthesis, while the synthesised zeolite serves as an effective

heterogeneous catalyst for converting aquaculture by-products into value-added bio-based intermediates. Future work should investigate catalyst reusability, optimise the epoxidation process, and evaluate the scalability of the synthesis route for practical applications. Additionally, this study advances Sustainable Development Goal (SDG) 12: Responsible Consumption and Production by encouraging the circular use of natural minerals and aquaculture by-products in sustainable catalytic processes.

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CONFLICT OF INTERESTS

The authors affirm that they have no competing interests.

AUTHOR CONTRIBUTIONS

This work contributes to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

D. Q. Do https://orcid.org/0000-0001-9497-5332
H.T. Tran https://orcid.org/0000-0001-5047-1423
S.M. Hoang https://orcid.org/0000-0001-6851-3927
Q. C. Khuu https://orcid.org/0000-0002-6743-1839
C. T. Vo https://orcid.org/0000-0002-0718-8750?lang=en

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