

Performance Test of Various Indonesian Natural Zeolites as Composite Components of NiMo/Al₂O₃-Zeolite Catalysts for Hydrocracking Used Cooking Oil into Biohydrocarbons

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Abstract

Due to increasing demand for alternative energy sources, nonedible used cooking oil is being converted into biohydrocarbons as an eco-friendly renewable option. This study explores the use of three Indonesian zeolites; Lampung, Bayah, and Tasikmalaya as a composite components of NiMo/Al₂O₃-Zeolite catalysts to enhance conversion and yields, promoting the use of sustainable domestic resources. The NiMo/ γ -Al₂O₃-zeolite catalyst, with alumina-to-zeolite ratios of 75:25 and 25:75, effectively converted used cooking oil into biohydrocarbons products—green diesel and gasoline. The NiMo/ γ -Al₂O₃ (75%)-Bayah Natural Zeolite (25%) catalyst exhibited a surface area of 194 m²/g, pore volume of 0.45 cm³/g, 7.01% Mo content, and a crystal size of 117.74 nm. At 370 °C, this catalyst achieved a 93% conversion, with GC-simdis analysis confirming 13% gasoline and 78% diesel fractions. This research demonstrates that Indonesian natural zeolites can be effectively used to convert used cooking oil into biohydrocarbons, achieving high conversion and desired product selectivity.

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1. Introduction

The development of renewable energy sources is required due to the depletion of fossil fuels and the environmental consequences of their consumption [1]. Because biohydrocarbons have qualities similar to those of regular hydrocarbons, they present a feasible alternative, especially when generated from non-edible sources like used cooking oil [2]. The National Energy Policy of Indonesia (Regulation No. 79 of 2014), which promotes greater use of renewable energy sources, serves as the foundation for this study.

Biohydrocarbons from the hydrocracking process are one of the most promising renewable energy sources since they can be easily incorporated into the current fuel infrastructure [3,4].

Biohydrocarbons can be produced from edible and non-edible oils; however, using edible oils as fuel can lead to competition with food supplies [5]. Used cooking oil (UCO) emerges as a viable feedstock for biohydrocarbon production, addressing both waste management and environmental pollution issues associated with its disposal [6]. The primary component of UCO, triglycerides, can be potentially converted into biohydrocarbons [7].

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Several studies have explored the use of various catalysts and feedstocks in biohydrocarbon production through hydrocracking, highlighting the importance of catalyst selection and modification. Wang *et al.* [8] investigated the hydrocracking of soybean oil using NiMo catalysts supported on different materials, including ZSM-5 and γ -Al₂O₃. Their findings revealed that zeolite-supported catalysts exhibited higher cracking activity, resulting in increased production of gasoline and hydrocarbon gas, while alumina-supported catalysts achieved higher overall hydrocarbon conversion. Similarly, Ishihara *et al.* [9] explored the effect of composite supports of zeolite and alumina on NiMo catalysts, achieving 90% conversion in hydrocracking processes. This study demonstrated that different types of zeolites could control the selectivity of products, ranging from gasoline to diesel, showcasing the versatility of zeolite-supported catalysts.

Further research by Aziz *et al.* [10] focused on the catalytic cracking of biodiesel derived from UCO using natural zeolite sourced from Lampung, Indonesia. The study found that a 1% NiO/zeolite catalyst offered the best performance, achieving a 60.79% conversion, indicating the effectiveness of local zeolites in enhancing catalyst activity. Irawan *et al.* [11] also utilized modified natural zeolite from Bayah, Indonesia, in the pyrolysis of palm oil waste, which improved the quality of hydrocarbon products by reducing acidity and viscosity. This study highlighted the potential of local zeolites as cost-effective and efficient catalysts in biofuel production, emphasizing their suitability as sustainable catalyst supports.

Previous studies have highlighted the potential of UCO as a feedstock for biohydrocarbon production, with the transesterification process being the most common pathway [12]. However, this process faces challenges, including poor storage stability and engine compatibility issues [13]. Hydrocracking, particularly with advanced catalysts such as zeolite-alumina composites, offers a solution to these challenges by enhancing the yield and quality of the biohydrocarbons [14,15].

The objective of this research is to explore the innovative use of underutilized Indonesian natural zeolites from regions such as Lampung, Bayah, and Tasikmalaya as catalyst promoters for the hydrocracking of used cooking oil (UCO) producing high-value biohydrocarbons. Specifically, the study aims to optimize catalytic performance by investigating zeolite-to-alumina ratios (75:25 and 25:75) to enhance biohydrocarbon yield and product selectivity. It also seeks to address limitations associated with traditional biodiesel production by employing

hydrocracking as an alternative to transesterification, overcoming challenges like storage stability and engine compatibility, while producing higher-quality biohydrocarbons such as green diesel and gasoline. Furthermore, the research aims to determine the optimal hydrocracking conditions by investigating the effects of reaction temperature in the range of 300 – 370 °C to achieve maximum conversion efficiency and desired product distribution. Another key objective is to promote environmental sustainability by repurposing waste materials and utilizing locally available natural zeolites, thereby reducing reliance on imported catalysts and supporting domestic production capabilities [16,17].

2. Materials and Method

2.1 Materials

The primary materials used in the study included UCO from fast food restaurant chain as the feedstock and natural zeolites sourced from local producer in Lampung, Bayah, and Tasikmalaya, known for their distinct mineral compositions. Alumina (boehmite, Catapalt) was used as the support material, combined with nickel nitrate hydrate [Ni(NO₃)₂·6H₂O] (Merck, 98-99%) and molybdenum trioxide [MoO₃] (Louyang, 99%) as the active catalyst components. Hydrochloric acid (HCl) (Sigma Aldrich, 37%) was utilised as a reagent for the dealumination of zeolites, while nitric acid (HNO₃) (Sigma Aldrich, 65%) and ammonium hydroxide (NH₄OH) (Merck, 99.98%) were used in the preparation of the support materials. Ultra-high purity (UHP) gases, including helium, hydrogen, and nitrogen from Surya Indotim Imex, were utilized in catalyst characterization and reaction processes. Iodine solution and titration reagents were used for the iodine number analysis.

2.2 Catalyst Preparation and Characterizations

The natural zeolites used in this study, sourced from regions with distinct compositions — Lampung, Bayah, and Tasikmalaya — were combined with alumina in weight ratios of 25:75 and 75:25 (zeolite to alumina). To enhance the catalytic properties, the zeolites were initially treated by refluxing with 9 M HCl at 100 °C for 3 hours, followed by thorough washing and drying at 110 °C. The zeolites were then mixed with alumina in the specified ratios and combined with 32 mL of deionized water (aquabides). Subsequently, HNO₃ and NH₄OH were added, and the mixture was thoroughly blended and cast before being calcined at 550 °C for 5 hours. In addition to the modified catalysts, an unmodified alumina catalyst was prepared as a reference for comparative analysis. This reference catalyst was

synthesized by mixing boehmite with deionized water, HNO_3 , and NH_4OH , followed by calcination at $550\text{ }^\circ\text{C}$ for 5 hours.

The prepared support was then impregnated with an aqueous solution of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and MoO_3 to introduce the active metal components. This mixture was once again dried and calcined at $550\text{ }^\circ\text{C}$ for 5 hours, resulting in the formation of the $\text{NiMo/zeolite-alumina}$ catalyst. The synthesized catalysts were characterized using X-ray fluorescence (XRF, Malvern PANalytical Axios series, 40 kV, 45 mA) and X-ray diffraction (XRD, Malvern PANalytical Empyrean series, Cu radiation, 40 kV, 15 mA, 2θ range $10\text{--}90^\circ$) for detailed compositional and structural analysis. Fourier-transform infrared spectroscopy (FTIR, Thermo Fisher Scientific Nicolet iS50) was performed to identify functional groups. Surface area and porosity assessments were conducted using Brunauer–Emmett–Teller (BET, Quantachrome AUTOSORB 6iSA.) analyzers.

2.3 Experimental Design

A pilot-scale trickle-bed reactor was used for the hydrocracking experiments (Figure 1). The reactor was loaded with 100 grams of catalyst and operated in a continuous flow mode. UCO, pre-treated to remove impurities and water content, was fed into the reactor. The hydrocracking temperatures were varied at $300\text{ }^\circ\text{C}$, $330\text{ }^\circ\text{C}$, $350\text{ }^\circ\text{C}$, and $370\text{ }^\circ\text{C}$. Each experiment was conducted under a constant pressure of 70 bars, H_2/Feed 300 mL/mL and Liquid Hourly Space Velocity (LHSV) of 1 h^{-1} . The conversion of triglycerides to biohydrocarbons was quantified using Gas Chromatography (GC) Agilent AT 7890A equipped with a Flame Ionization Detector (FID).

Product selectivity towards green diesel and gasoline was determined by analyzing the hydrocarbon range of the products using GC.

3. Results and Discussion

3.1 Characterization of Dealuminated Natural Zeolite and Catalyst Composites

XRF analysis was performed to determine the metal oxide content in natural zeolite before and after dealumination. The primary components in the untreated natural zeolites from Lampung, Bayah, and Tasikmalaya were silica (SiO_2) and alumina (Al_2O_3), with silica percentages of 73.4%, 71.5%, and 72.3%, respectively. After dealumination, silica content increased significantly, while alumina content decreased across all samples, reflecting the successful removal of aluminum from the zeolite framework (Table 1). The dealumination process also reduced impurities, such as K_2O , CaO , and Fe_2O_3 , enhancing the overall quality of the zeolite by increasing its silica content and decreasing its alumina content, which led to an increase in the Si/Al ratio (Table 2). A higher Si/Al ratio implies lower acidity, which is crucial for preventing over cracking in hydrocracking reactions targeting diesel and gasoline fractions [18].

BET analysis showed that acid treatment significantly increased the surface areas of the natural zeolites, indicating effective removal of impurities and enhanced porosity (Table 3). The surface area of Lampung zeolite increased from $41.0\text{ m}^2/\text{g}$ to $140\text{ m}^2/\text{g}$, while Bayah zeolite exhibit an increase from $59.5\text{ m}^2/\text{g}$ to $191\text{ m}^2/\text{g}$. This increase in surface area and pore volume is beneficial for catalytic applications, providing more active sites for reactions.

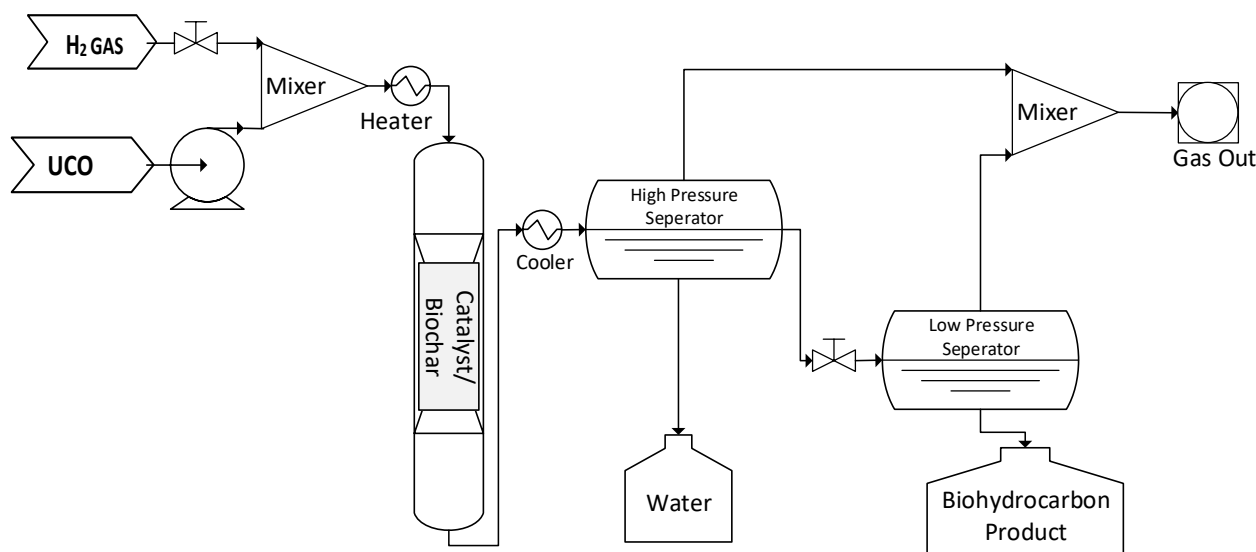


Figure 1. Schematic diagram of pilot plant experimental rig.

Figure 2 showed XRD analysis results, confirming Clinoptilolite as the main phase in Lampung zeolite, while Tasikmalaya and Bayah zeolites contained both Clinoptilolite and Mordenite phases. The crystal size of the zeolites was calculated using the Debye-Scherrer equation, which considers the XRD peak broadening at specific 2θ values. The larger the crystal size, the greater the surface area. The crystal sizes before and after acid treatment showed significant changes: for untreated Lampung zeolite at 2θ of 23°, the crystal size was 58.87 nm, increasing to 117.76 nm after treatment. Tasikmalaya zeolite at 2θ of 26° increased from 59.16 nm to 88.66 nm, while Bayah

zeolite at 2θ of 23° increased from 44.15 nm to 117.74 nm after acid treatment. These increases in crystal size indicate a reduction in defects and an improvement in the crystalline structure of the zeolites, which can contribute to enhanced catalytic stability and activity [19]. The greater the concentration, the greater the percentage of crystallinity which affects the magnitude of crystallinity, namely the area of the crystalline fraction and the amorphous fraction [19].

FTIR analysis was conducted to determine the types of acidic sites present in the natural zeolites, specifically the Brønsted and Lewis acid sites. Figure 3 showed the FTIR spectra of all zeolite samples, both before and after acid

Table 1. XRF analysis results of metal oxides in natural zeolite before and after acid treatment.

Compound	Lampung Natural Zeolite		Bayah Natural Zeolite		Tasik Natural Zeolite	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
SiO ₂ (wt.%)	73.4	87.6	71.5	89.0	72.3	87.8
Al ₂ O ₃ (wt.%)	14.9	7.54	15.3	5.77	13.4	6.91
K ₂ O (wt.%)	3.25	0.67	4.20	2.20	4.35	2.99
CaO (wt.%)	2.84	0.76	3.58	0.78	2.66	0.53
Fe ₂ O ₃ (wt.%)	2.36	1.52	2.40	0.49	2.17	0.27

Table 2. XRF analysis results of Si-Al compounds in natural zeolite

Elements	Lampung Natural Zeolite		Bayah Natural Zeolite		Tasik Natural Zeolite	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
Si (wt.%)	35.5	42.0	33.4	41.6	34.8	41.0
Al (wt.%)	7.92	3.91	8.12	3.06	7.37	3.66
Si/Al Ratio	4.48	10.7	4.11	13.6	4.72	11.2

Table 3. BET analysis results of natural zeolite before and after treatment

BET Result	Lampung Natural Zeolite		Bayah Natural Zeolite		Tasik Natural Zeolite	
	Untreated	Treated	Untreated	Treated	Untreated	Treated
SA (m ² /g)	41.0	140	59.5	191	54.3	141
PV (cm ³ /g)	0.20	0.27	0.18	0.44	0.19	0.25
PD (Å)	191	76.7	121	91.3	143	69.5

Table 4. XRF analysis results of natural zeolite after impregnation

Elements	Type of Catalyst						
	NiMo/γ-Al ₂ O ₃	NiMo/γ-Al ₂ O ₃ 75%-ZAL 25%	NiMo/γ-Al ₂ O ₃ 75%-ZAT 25%	NiMo/γ-Al ₂ O ₃ 75%-ZAB 25%	NiMo/γ-Al ₂ O ₃ 25%-ZAL 75%	NiMo/γ-Al ₂ O ₃ 25%-ZAT 75%	NiMo/γ-Al ₂ O ₃ 25%-ZAB 75%
wt.% Ni	2.34	2.14	2.58	2.56	2.21	2.22	2.44
wt.% Mo	3.23	6.00	7.13	7.01	6.54	6.24	7.03

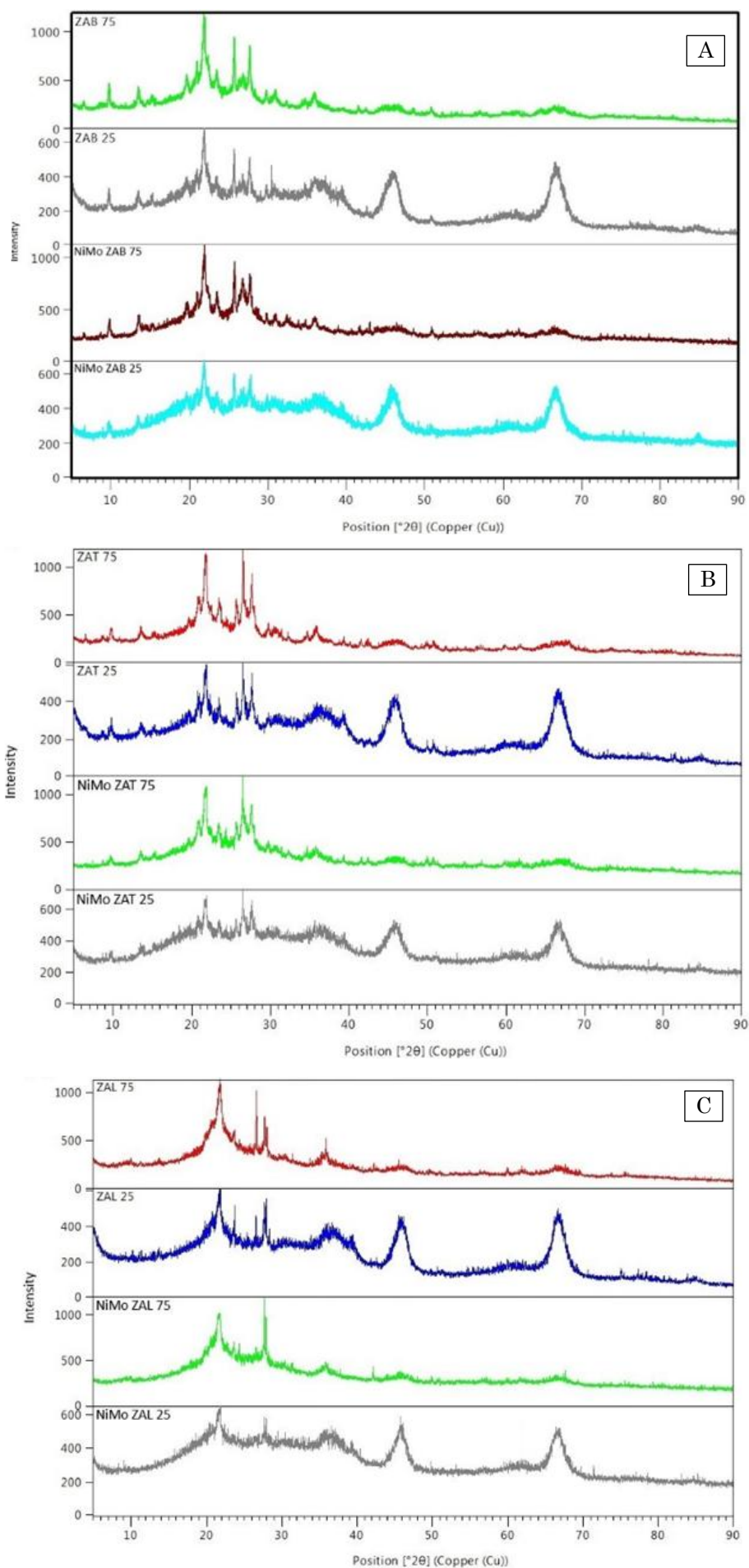


Figure 2. Diffractogram of crystalline phase analysis results with XRD from the sample ZAB catalyst (A), ZAT catalyst (B), and ZAL catalyst (C)

no significant bands observed in the range 1400–1480 cm^{-1} , which would indicate the presence of Lewis acid sites [20]. The absence of Lewis acid sites and the dominance of Brønsted acidity suggest that the acid treatment primarily affects

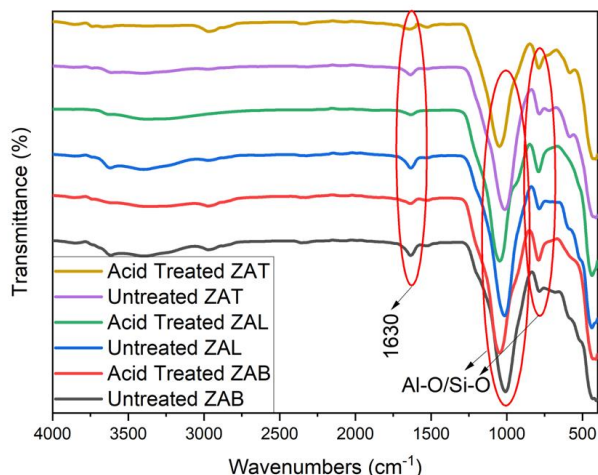


Figure 3. Natural zeolite FTIR results



Figure 4. Used cooking oil feedstock (left) and biohydrocarbon produced from different catalyst composites (left to right): NiMo/ γ - Al_2O_3 , NiMo/ γ - Al_2O_3 75%-ZAT25%, NiMo/ γ - Al_2O_3 75%-ZAB25%, NiMo/ γ - Al_2O_3 75%-ZAL25%, and Biochar.

the Brønsted sites within the zeolite framework. This acidity profile aligns well with the catalytic properties required for hydrocracking processes, where controlled acidity is essential to avoid excessive cracking of hydrocarbons into undesired lighter fractions.

After mixing the dealuminated zeolites with alumina at ratios of 25:75 and 75:25, the catalysts were impregnated with NiMo. XRF results indicated that zeolite-containing catalysts exhibit higher Mo absorption compared to the alumina-only catalyst, reflecting the superior adsorption capacity of zeolites due to their crystalline structure (Table 4). Based on previous research, zeolites are widely recognized for their superior Mo adsorption capabilities compared to alumina, attributed to their well-defined crystalline structure and orderly atomic arrangements [21,22]. This structure facilitates efficient metal dispersion, which is critical for catalytic performance in hydrocracking reactions [23].

3.2 Catalytic Activity Test and Product Characterization

Hydrotreating was conducted in a trickle bed reactor to convert UCO into biohydrocarbons. The product appearance varied depending on the catalyst used, with zeolite-containing catalysts producing clearer products compared to the yellow-tinted product from the alumina-only catalyst (Figure 4). This difference is attributed to the influence of double bonds on product color, with higher double bond content leading to darker color [24].

The data presented in Figure 5 showed that higher temperatures led to increased conversions, confirming that higher temperatures enhance

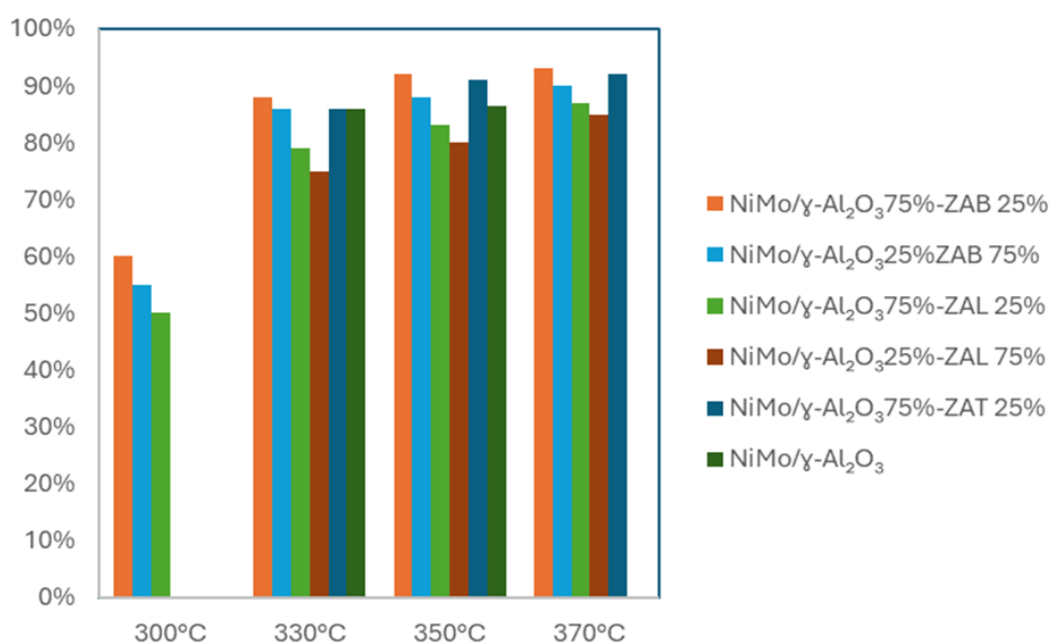


Figure 5. Effect of variation in natural zeolite types, zeolite/alumina ratios, and temperature on hydrotreating results

cracking efficiency. At 350 °C, catalysts containing Bayah and Tasikmalaya zeolites ($\text{NiMo}/\gamma\text{-Al}_2\text{O}_3$ 75%-ZAB 25% and $\text{NiMo}/\gamma\text{-Al}_2\text{O}_3$ 75%-ZAT 25%) achieved the highest conversions of approximately $92\pm2.7\%$ and $91\pm3.5\%$, respectively, outperforming the alumina-supported catalyst, which reached $87\pm2.5\%$ (Figure 6). The superior performance of $\text{NiMo}/\gamma\text{-Al}_2\text{O}_3$ 75%-ZAB 25% and $\text{NiMo}/\gamma\text{-Al}_2\text{O}_3$ 75%-ZAT 25% catalysts is attributed to their higher surface areas and better molybdenum uptake compared to other variants, which enhances their catalytic activity. The presence of zeolites in the catalyst promotes hydrocracking, leading to the formation of shorter hydrocarbon chains. According to Dik *et al.* [25], the addition of zeolites enhances catalyst activity but also reduces diesel fractions.

The feedstock, UCO, primarily contained oleic and palmitic acids, typical components of palm oil. The dominant fatty acids were palmitic acid (47.08%) and oleic acid (37.52%), which are key precursors in the formation of biohydrocarbons. GC Simdist analysis showed that the UCO feedstock contained long-chain hydrocarbons (C56–C66), indicating the presence of triglycerides. After hydrotreating, the longest detected carbon chains in the products were C37, with C16 and C18 being the most abundant, reflecting effective conversion to shorter hydrocarbons suitable for diesel and gasoline.

Figure 7 shows the percentage yield of diesel and gasoline for each catalyst. The data indicates that catalysts containing natural zeolites have higher selectivity for gasoline fractions, while

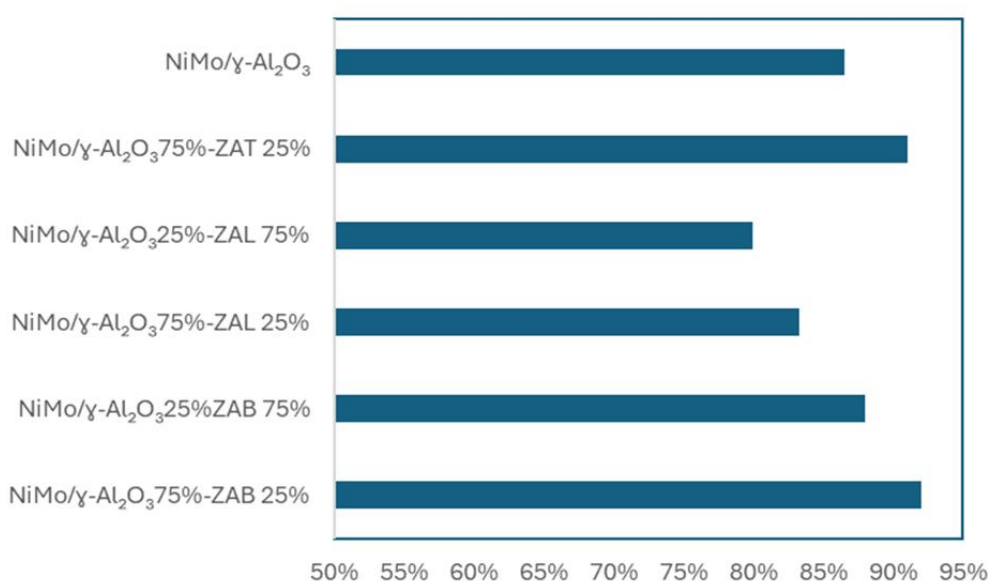


Figure 6. Comparison of conversion for different types of zeolites and zeolite/alumina ratios at 350 °C

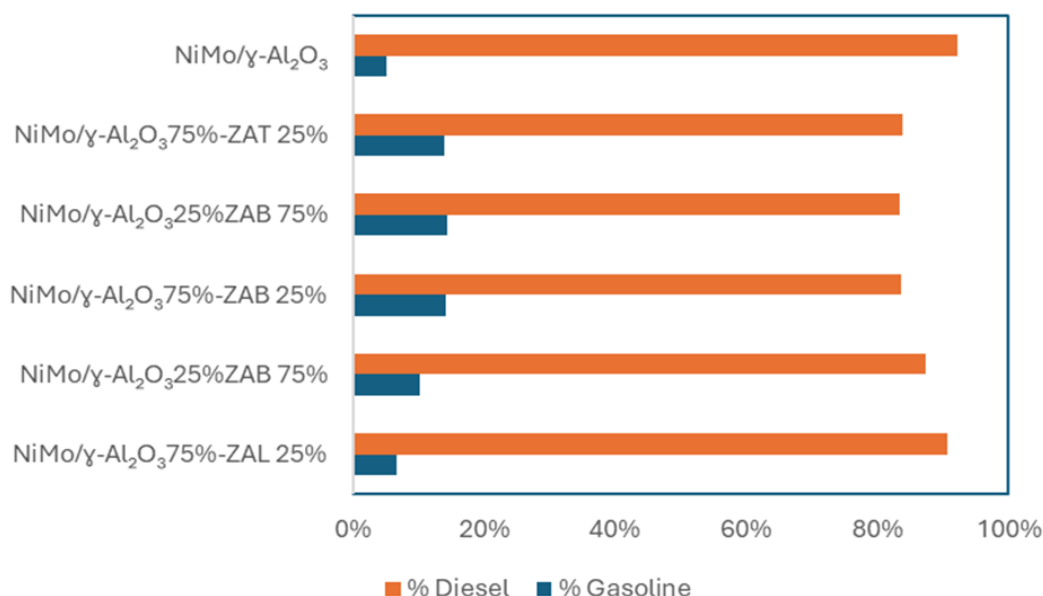


Figure 7. Percentage yield of diesel and gasoline for each catalyst at 350 °C

catalysts without zeolites are more selective for diesel fractions. This difference is due to the acidic nature of zeolites, which can donate hydrogen atoms, leading to further cracking into shorter fractions.

Density and viscosity analyses were conducted on the biohydrocarbon products to assess their fuel quality. An iodine number test was also conducted to determine the degree of unsaturation in the biohydrocarbons. Higher-density fuels may require adjustments in fuel injection timing and duration to ensure proper combustion. Properly adjusted fuel density can improve combustion efficiency and engine performance [26].

Table 5 compares the characteristics of the biohydrocarbon products with Standar Nasional Indonesia standards for gasoline, biodiesel, and diesel. The density measurements were taken at 40 °C for both NiMo/ γ -Al₂O₃ and NiMo/ γ -Al₂O₃-zeolite catalysts. In general, hydrotreating reduced the density of the biohydrocarbon products. The lower density compared to biodiesel and diesel standards can be attributed to the predominance of paraffinic hydrocarbons, mainly C16 and C18. These shorter hydrocarbons lack the longer chains found in traditional diesel or biodiesel, resulting in lower density and viscosity values within the acceptable range for fuel standards. The iodine number, which measures the degree of unsaturation, was highest for the NiMo/ γ -Al₂O₃ catalyst. Fuels with high iodine numbers are less stable against oxidation, potentially affecting performance and shelf life [27].

4. Conclusion

This study demonstrated the successful utilization of natural Indonesian zeolites (Lampung, Bayah, and Tasikmalaya) as components in NiMo/ γ -Al₂O₃-zeolite catalysts for hydrocracking used cooking oil (UCO) into biohydrocarbons. The dealumination process enhanced the catalytic properties of the zeolites, improving surface area, porosity, and selectivity toward desired products. Among the tested catalysts, NiMo/ γ -Al₂O₃ (75%)-Bayah Natural Zeolite (25%) showed the highest performance, achieving a 93±2.6% conversion rate at 370 °C and yielding significant amounts of diesel and gasoline fractions. These findings highlight the potential of locally sourced natural zeolites as cost-effective and efficient catalyst supports, contributing to national renewable energy goals and offering an environmentally friendly solution for converting waste into valuable biofuels.

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CRedit Author Statement

Authors Contributions: Amar Ariza Kurniawan: Formal analysis, investigation, writing original draft; Wawan Rustyawan: Supervision and reviewing; Muhammad Ibadurrohman: Supervision, editing and reviewing. All authors have read and agreed to the published version of the manuscript.

Table 5. Comparison of biohydrocarbon product characteristics at 350 °C with SNI standards

Parameter	UCO	SNI 8415:2017	SNI 7182:2015	SNI 8220:2017	Hydrotreating Product					
		(Gasoline)	(Biodiesel)	(diesel)	NiMo/ γ - Al ₂ O ₃	NiMo/ γ - Al ₂ O ₃ 75 %-ZAL 25%	NiMo/ γ - Al ₂ O ₃ 75 %-ZAT 25%	NiMo/ γ - Al ₂ O ₃ 75 %-ZAB 25%	NiMo/ γ - Al ₂ O ₃ 25 %-ZAL 75%	NiMo/ γ - Al ₂ O ₃ 25% ZAB 75%
Color	Dark Brown	Green, Clear and bright	Clear and bright	Clear and bright	Clear yellow	Clear	Clear	Clear	Clear	Clear
Density (40°C, kg/cm ³)	920	715-770	815-870	815-870	767	756	758	760	757	754
Cinematic viscosity (40°C, cSt)	31.2	0.95	2.3-6.0	2.0-4.5	2.91	2.6	2.53	2.52	2.54	2.49
Iodin Value (g iod/100 g)	48.56	-	max 115	-	9.83	5.72	6.08	6.32	5.96	6.53

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