Indonesian Journal of Material Research
***e*-ISSN: 2987-1654**
Vol. 3, No. 3, November 2025

Indonesian Journal of
MATERIAL RESEARCH

Research Paper

Evaluation of Natural Zeolite and Bentonite as Catalysts in Cyclohexanone Oxidation with Hydrogen Peroxide

Muhammad Hidayatullah[1], Sahrul Wibiyan[2,3], Risfidian Mohadi[2], Aldes Lesbani[2,3*]

[1] *Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sriwijaya, Indralaya, 30882, Indonesia*
[2] *Master Program of Materials Science, Graduate School, Universitas Sriwijaya, Palembang, 30139, Indonesia*
[3] *Research Center of Inorganic Materials and Coordination Complexes, Universitas Sriwijaya, Palembang, 30139, Indonesia*
[*] **Corresponding author**: aldeslesbani@pps.unsri.ac.id

Abstract

The catalytic oxidation of cyclohexanone using environmentally benign oxidants remains a key challenge in sustainable organic synthesis. In this study, natural zeolite (clinoptilolite and mordenite) and bentonite were evaluated as heterogeneous catalysts for the oxidation of cyclohexanone with hydrogen peroxide under reflux at 90°C. Structural characterization by X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) confirmed the aluminosilicate frameworks, where clinoptilolite exhibited the highest crystallinity followed by bentonite and mordenite. The oxidation products were analyzed using FTIR, melting point measurements, and gas chromatography–mass spectrometry (GC-MS). Despite the catalytic activity of all materials, FTIR and melting point analyses revealed that the expected adipic acid was not formed under the applied conditions. GC-MS results indicated the formation of partially oxidized oxygenated intermediatessuch as alcohols, ethers, and carbonyl derivatives whose distribution strongly depended on the catalyst's pore structure and surface properties. Clinoptilolite promoted confined partial oxidation due to its microporous structure, whereas bentonite facilitated non-selective oxidation owing to its open layered framework. These findings emphasize that the interplay between molecular confinement and oxygen accessibility governs the selectivity of cyclohexanone oxidation and provide insights for the rational design of improved zeolite and clay-based catalytic systems for green oxidation reactions.

Keywords
Catalytic Oxidation, Zeolite, Bentonite, Cyclohexanone, Characterization

Received: 19 August 2025, Accepted: 10 October 2025
https://doi.org/10.26554/ijmr.20253369

1. INTRODUCTION

Among various oxidation methods, catalytic oxidation processes are particularly effective, as they can degrade hazardous compounds without producing polycyclic or aromatic by-products. Cyclohexanone, an important intermediate in the chemical industry, plays a key role in the synthesis of ε-caprolactam, adipic acid, and nylon-based polymers. Traditionally, its oxidation to adipic acid involves multi-step reactions using nitric acid, which produce large amounts of nitrogen oxide by-products that are harmful to the environment. Therefore, the development of greener and more sustainable oxidation routes has attracted considerable interest in recent years. Nonetheless, studies on α-substituted cyclohexanones have mainly focused on compounds with minimal steric hindrance, such as 2-methylcyclohexanone and 2-phenylcyclohexanone, which are widely used in the production of polyamide resins (Gajbhiye et al., 2025; Ma et al., 2021; Umam et al., 2025).

Hydrogen peroxide (H_2O_2) is recognized as a green and sustainable oxidant owing to its high active oxygen content and the formation of water as its sole by-product. Compared to peracids, H_2O_2 offers several advantages, including lower cost, wider availability, and greater safety during storage and transportation. However, despite these benefits, its oxidizing ability at low concentrations remains weak, requiring the presence of efficient catalysts to promote selective oxidation reactions. In recent years, H_2O_2 and O_2 have gained significant attention as environmentally friendly oxidants for the oxidation of cyclohexanol to cyclohexanone. Nonetheless, the conventional production of H_2O_2 via the anthraquinone process remains energy-intensive and environmentally detrimental, and its tendency to decompose exothermically introduces safety risks during handling and storage. Therefore, from both environmental and economic perspectives, the development of catalytic oxidation processes utilizing molecular oxygen (O_2) as an alternative to H_2O_2 for cyclohexanol oxidation is considered a promising and more sustainable approach (Liang et al., 2025; Tu et al., 2025).

Research on catalytic processes continues as scientists seek more efficient materials for chemical conversion, especially through catalytic oxidation reactions using synthesized catalysts such as layered double hydroxide base material (Ahmad et al., 2023; Mohadi et al., 2023a,b). Zeolites are crystalline hydrated aluminosilicates, either natural or synthetic, possessing a well-defined microporous framework. Natural zeolites, with their aluminosilicate frameworks, offer abundant acidic sites and well-organized microporous channels that promote molecular diffusion and adsorption, making them highly effective for catalytic reactions (Siora, 2025). Bentonite, a naturally occurring layered aluminosilicate primarily composed of montmorillonite, exhibits a large specific surface area, high compressive strength, excellent thermal stability, and low cost. Its abundant surface hydroxyl groups, high cation-exchange capacity, and structural flexibility make it an effective catalyst support in heterogeneous catalysis. The utilization of bentonite, either alone or in combination with other natural catalysts, can enhance oxidation performance through synergistic effects arising from its surface activity and textural properties (Priatna et al., 2023; Zeng et al., 2025).

Despite many studies on the oxidation of cyclohexanone, most research focuses on achieving complete oxidation to adipic acid or other high-value products using transition metal-based catalysts (Mekala et al., 2022; Pan et al., 2021; Yan et al., 2020). However, oxidation over natural mineral catalysts such as zeolite and bentonite may lead to different reaction pathways, resulting in partial oxidation products such as cyclohexanol, cyclohexenone, or other oxygenated compounds (Mehta et al., 2016; Niu et al., 2023). Understanding these reaction pathways and product selectivity is crucial for optimizing green oxidation systems.

Therefore, this study aims to investigate the catalytic performance of natural zeolite and bentonite in the oxidation of cyclohexanone using hydrogen peroxide as a green oxidant. This study investigates the characterization of catalysts and the crystals produced through the catalytic oxidation of cyclohexanone using hydrogen peroxide, as well as identifies the types of crystalline compounds formed based on the characterization results obtained from FTIR, melting point, and GC-MS analyses.

2. EXPERIMENTAL

2.1 Chemical and Instrumental

Cyclohexanone (analytical grade, ≥99%) and hydrogen peroxide (H_2O_2, 30% w/w) were used as the main reactants without further purification. The catalysts employed in this study consisted of natural zeolites and natural bentonite. The natural zeolites were obtained from several regions in Indonesia, including clinoptilolite from Lampung, clinoptilolite from Bayah (Banten), and mordenite from Nanggung (Bogor, West Java). Natural bentonite was also utilized as a representative aluminosilicate material with high surface activity. All catalyst samples were oven-dried and sieved to obtain uniform particle sizes prior to use. Distilled water was used throughout the experimental work for reagent preparation and washing processes.

The experimental setup consisted of a 250 mL round-bottom flask equipped with a reflux condenser, magnetic stirrer, and mercury thermometer to control reaction temperature and mixing conditions. Additional laboratory equipment included beaker glass, filter paper, desiccator, and capillary tubes for product collection and physical measurements. Melting points of solid samples were determined using a melting point apparatus. The structural and functional characterization of catalysts and reaction products was performed using Fourier Transform Infrared Spectroscopy (FTIR, Shimadzu 8201 PC) in the spectral range of 4000-400 cm^{-1}. Product composition and selectivity were analyzed by Gas Chromatography–Mass Spectrometry (GC-MS, Shimadzu QP 2010 SE) equipped with an RTX-5MS capillary column. The crystalline structure of the catalysts was characterized using X-ray diffraction (XRD, Rigaku Miniflex 600, Cu Kα radiation, λ = 1.5406 Å).

2.2 Catalyst Characterization

Prior to use in the oxidation reaction, the natural zeolite and bentonite materials were characterized to determine their structural and surface properties. The characterization aimed to confirm the crystalline phases and functional groups present in the materials, which are relevant to their catalytic performance. The crystalline structure of the natural zeolite and bentonite was analyzed using X-ray diffraction. Fourier Transform Infrared Spectroscopy was used to identify the functional groups of the materials. These characterizations were conducted to provide fundamental information about the structure and surface chemistry of the catalysts before their application in the oxidation of cyclohexanone.

2.3 Synthesis of Adipic Acid from Cyclohexanone Using Natural Zeolite and Bentonite Catalysts

Initially, the oxidation mixture was prepared in a beaker glass using a magnetic stirrer to ensure uniform dispersion of the catalyst. Specifically, 1.0 g of natural zeolite or bentonite catalyst was added to 44 mL of 30% hydrogen peroxide (H_2O_2) and stirred for 15 minutes. Subsequently, 10 mL of cyclohexanone was added dropwise under continuous stirring to initiate the oxidation reaction. The resulting homogeneous suspension was then transferred into a 250 mL round-bottom flask equipped with a reflux condenser and magnetic stirrer. The reaction system was refluxed at a temperature of 90°C for 8 hours while maintaining constant stirring. Upon completion of the reaction, the mixture was allowed to cool, and the solid catalyst was separated from the liquid phase by filtration. The recovered catalyst was stored for possible reuse, while the filtrate was placed in a refrigerator and maintained at 0°C for 24 hours to promote crystal formation. The resulting crystals were collected by filtration, washed thoroughly with distilled water, and dried in a desiccator for 24 hours. The dried solid products were subsequently weighed and characterized by melting point determination, Fourier Transform Infrared Spectroscopy, and Gas Chromatography-Mass Spectrometry to identify the obtained oxidation products. An illustration of the experimental setup and reaction process is presented in Figure 1.

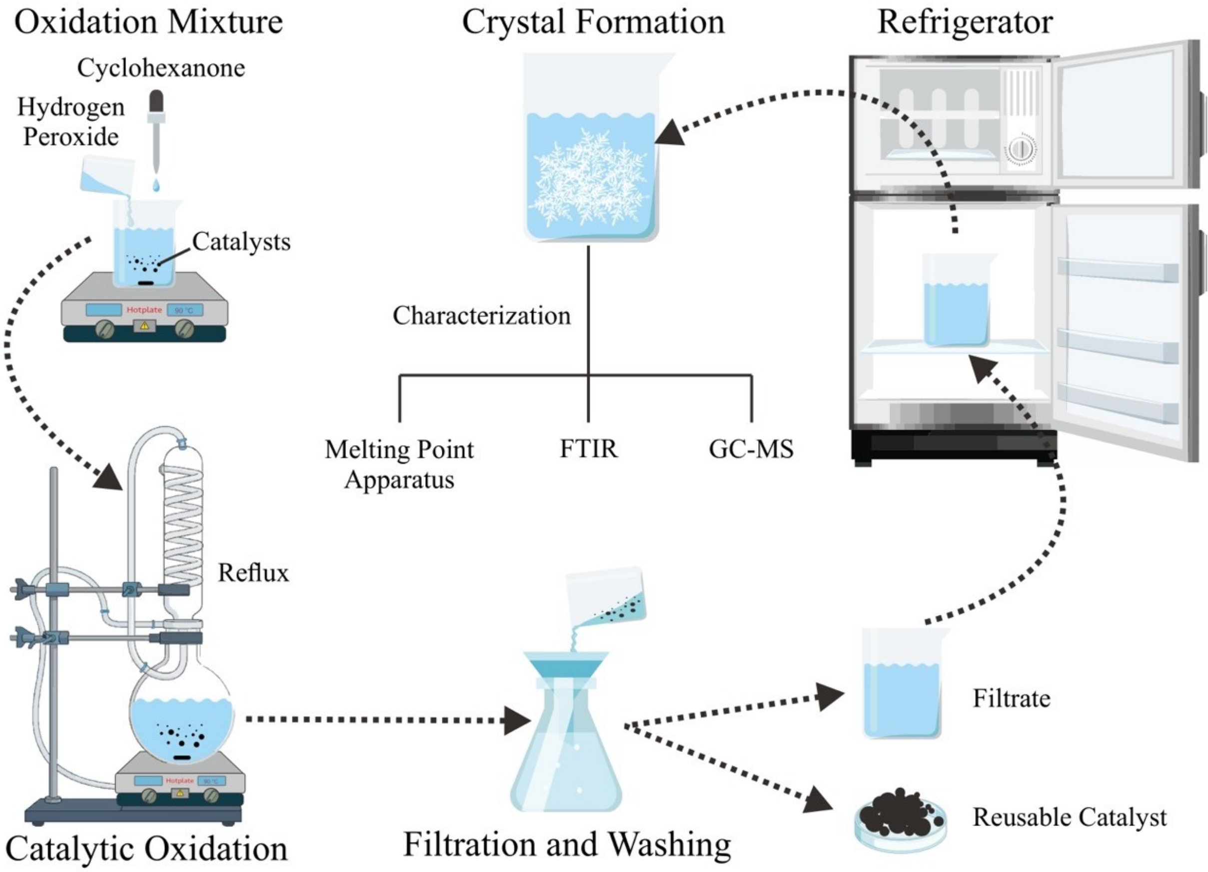

Figure 1. Schematic Illustration of the Oxidation Process of Cyclohexanone Using Natural Zeolite and Bentonite Catalysts with Hydrogen Peroxide as the Oxidant

3. RESULTS AND DISCUSSION

3.1 Catalyst Characterization

3.1.1 X-ray Diffraction (XRD)

The X-ray diffraction (XRD) analysis was conducted to determine the structural characteristics and crystallinity of natural zeolites (mordenite and clinoptilolite) and bentonite used as catalysts in the cyclohexanone oxidation reaction to adipic acid. The diffraction peaks of natural zeolites appeared in the range of 2–30° 2θ, which is consistent with the typical crystalline framework of aluminosilicate minerals. Clinoptilolite exhibited sharp and intense peaks below 30° 2θ, indicating a higher degree of crystallinity compared to mordenite (Huang et al., 2024; Pérez González et al., 2024; Yordanova et al., 2025). The prominent reflections at approximately 9.3° and 20.3–23.9° 2θ correspond to the characteristic planes of clinoptilolite, confirming its well-ordered microporous structure. This high crystallinity suggests greater structural rigidity and stability, making clinoptilolite more suitable for repeated catalytic applications in heterogeneous systems. In contrast, mordenite displayed broader and less intense diffraction peaks (broad features centered around 12–22° 2θ and weaker peaks at 2.1°, 6.8°, 8.1°, 12.7°, 17.8°), reflecting lower crystallinity and a more amorphous nature. This may influence its catalytic performance, as reduced crystallinity often leads to less-defined active sites and decreased structural stability. However, mordenite still retains its framework integrity, which contributes to its adsorption and ion-exchange capabilities (Güngör and Özen, 2021; Vaezi et al., 2025; Wahono et al., 2020).

Bentonite exhibited diffraction peaks in the low-angle and mid-angle regions, with notable reflections at 2.5° 2θ (low-angle/basal), 9.5–13° 2θ, and 20–23° 2θ, characteristic of layered silicate (smectite) structures. The presence of a sharp reflection near 2.5° 2θ indicates an observable basal spacing (interlayer) and moderate crystallinity, while additional peaks around 20–23° 2θ reflect layered/tetrahedral-octahedral framework features that remain after drying/processing. These structural features highlight bentonite's flexibility and swelling capacity, making it a suitable support or co-catalyst material in heterogeneous catalysis. Overall, the XRD results indicate that clinoptilolite possesses the highest crystallinity among the examined materials, followed by bentonite and mordenite (El-Khalafy et al., 2025; Seidi et al., 2024; Wang et al., 2025) This order of crystallinity (clinoptilolite >bentonite >mordenite) aligns with their expected catalytic performance and structural stability in the oxidation reaction system.

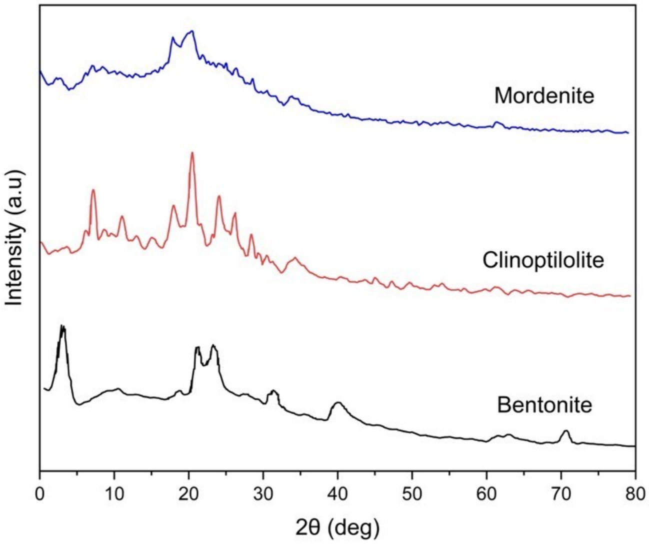

Figure 2. XRD Characterization of Catalysts of Mordenite, Clinoptilolite, and Bentonite

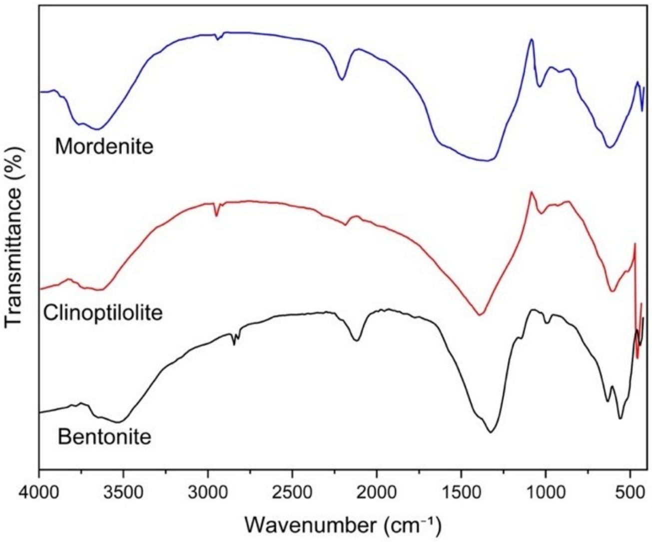

Figure 3. FTIR Characterization of Catalysts of Mordenite, Clinoptilolite, and Bentonit

3.1.2 Fourier Transform Infrared Spectroscopy (FTIR)

The functional groups and structural frameworks of the Mordenite, Clinoptilolite, and Bentonite catalysts were characterized using Fourier Transform Infrared (FTIR) spectroscopy, with the resulting spectra presented in Figure 3. The analysis confirms the characteristic aluminosilicate nature of all three materials, identifiable through distinct vibrational bands corresponding to their structural bonds. As shown in Figure 3, all catalysts exhibit a broad absorption band in the region of 3400-3600 cm^{-1} and a smaller peak at approximately 1630 cm^{-1}. The former is attributed to the O−H stretching vibrations of hydroxyl groups from physically adsorbed water molecules and surface silanol (Si−OH) groups. The latter peak corresponds to the H−O−H bending vibration of water, indicating the presence of moisture within the porous structure of the materials (Aghel et al., 2022; Gurdal and Yasar, 2023; Pan et al., 2025; Wahono et al., 2019).

The most informative region for identifying the aluminosilicate framework lies between 400 and 1200 cm^{-1}. A prominent and intense absorption band is observed for all three samples at approximately 1000-1050 cm^{-1}. This band is characteristic of the asymmetric stretching vibrations of the internal T−O−T linkages, where T can be either a silicon (Si) or aluminum (Al) atom within the TO_4 tetrahedral units that form the primary building blocks of zeolites and clay minerals. The presence of this strong peak confirms the integrity of the Si−O−Si and Si−O−Al network in Mordenite, Clinoptilolite, and Bentonite. Furthermore, additional peaks are observed in the lower wavenumber region. The bands between 420 and 800 cm^{-1} are associated with the symmetric stretching and bending vibrations of T−O bonds and the vibrations of external linkages between the tetrahedra. Specifically, the peaks appearing around 420-500 cm^{-1} are indicative of Si−O and Al−O bond vibrations, respectively. While all three materials display these fundamental spectral features, subtle differences in the exact peak positions and shapes reflect variations in their specific crystal structures, Si/Al ratios, and cation compositions. In conclusion, the FTIR analysis substantiates the structural identity of Mordenite, Clinoptilolite, and Bentonite as aluminosilicate materials, characterized by a robust tetrahedral framework and the presence of hydroxyl groups. These structural features are essential for their application as catalysts (Jiang et al., 2025; Khairuddin et al., 2025; Neolaka et al., 2022). However, based on the FTIR and GC-MS analyses of the oxidation products of cyclohexanone, no characteristic peaks or molecular fragments corresponding to adipic acid were detected. This finding indicates that adipic acid was not formed through the catalytic activity of Mordenite, Clinoptilolite, or Bentonite under the applied reaction conditions. The absence of adipic acid formation suggests that these catalysts did not promote complete oxidation of cyclohexanone, possibly leading instead to the formation of other intermediate or by-product compounds, as discussed in the following section.

3.2 FTIR Characterization of Synthesized Adipic Acid Using Catalysts

An initial identification of the crystalline products, which were obtained from the catalytic conversion of cyclohexanone using natural zeolite (clinoptilolite and mordenite) and natural bentonite catalysts, was performed using FT-IR spectroscopy. To determine the chemical structure of the synthesized crystals, their FT-IR spectra were compared with the spectrum of a standard adipic acid, as presented in Figure 4. Figure 4 illustrates the FT-IR spectra of the standard adipic acid and the crystals synthesized using bentonite, mordenite, and clinoptilolite catalysts. The adipic acid standard spectrum displays a characteristic broad absorption band between 2500-3300 cm^{-1}, which corresponds to the O-H stretching vibration of the carboxylic acid group. A sharp and intense peak is observed at approximately 1700 cm^{-1}, which is a definitive indicator of the carbonyl (C=O) functional group. Furthermore, absorption bands corresponding to the C−O stretch (1080-1300 cm^{-1}) and aliphatic C−H stretches (below 3000 cm^{-1}) are also present (Liu et al., 2024; Wu et al., 2025).

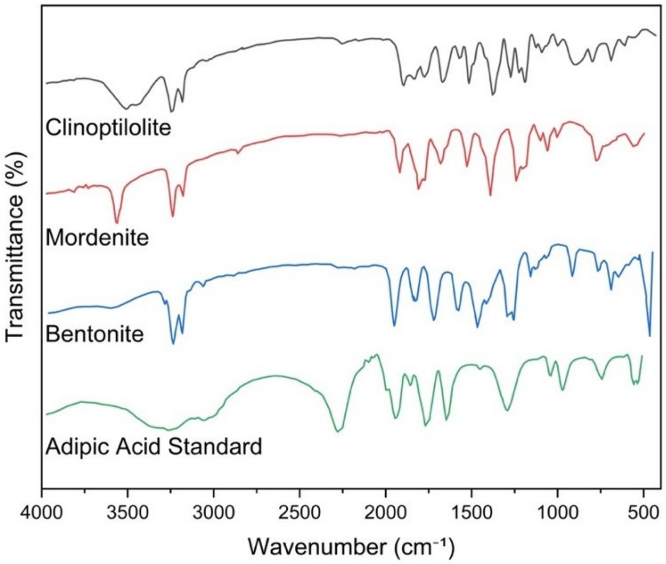

Figure 4. FTIR Characterization of Standard Adipic Acid and Synthesized Adipic Acid Using Catalysts Clinoptilolite, Mordenite, and Bentonite

In contrast, the FT-IR spectra of the crystals synthesized using clinoptilolite, mordenite, and bentonite, while similar to each other, show a significant deviation from the adipic acid standard. The most critical distinction observed in Figure 4 is the complete absence of the strong carbonyl (C=O) absorption peak around the 1700-1800 cm^{-1} region in all three synthesized samples (Wahono et al., 2019). Although broad O−H and aliphatic C−H absorption bands are present, the lack of the characteristic carbonyl peak strongly suggests that the resulting crystalline product is not adipic acid. This conclusion is further substantiated by a melting point analysis. The melting points of the crystals produced using clinoptilolite, mordenite, and bentonite catalysts were determined to be 80-85°C, 85-90°C, and 128-132°C, respectively. These values are considerably lower than the melting point of standard adipic acid, which is 145-152°C (Ganai et al., 2025). The significant discrepancy between the melting points of the synthesized crystals and standard adipic acid corroborates the FT-IR data, reinforcing the conclusion that the conversion of cyclohexanone under these catalytic conditions did not yield adipic acid.

3.3 GC-MS Identification of Products Formed during Catalytic Conversion of Cyclohexanone

The catalytic oxidation of cyclohexanone over clinoptilolite and bentonite catalysts exhibited distinct product distribution profiles, as clearly demonstrated by the GC-MS chromatograms in Figure 5. These findings underscore the profound influence of catalyst structure and surface characteristics on product selectivity and reaction pathways. The GC-MS chromatogram for the clinoptilolite-catalyzed reaction (Figure 5a) reveals a highly complex mixture containing up to 25 identifiable peaks, suggesting the occurrence of multiple oxidation and rearrangement steps. Nevertheless, the mechanistic scheme proposed in Figure 5c provides a crucial insight: the expected target product, adipic acid, was not formed under the investigated conditions. Instead, the detected compounds correspond predominantly to oxygenated intermediates containing aliphatic C−H, C−O, and O−H vibrations, indicating that oxidation proceeded only partially, stopping at intermediate alcohol or ketone stages. This observation suggests that the microporous zeolitic structure of clinoptilolite, despite offering high surface acidity and redox potential in the presence of hydrogen peroxide, may impose steric restrictions that hinder the full oxidation of cyclohexanone into the more extended dicarboxylic structure of adipic acid. The confined pore environment likely facilitates the adsorption and activation of the reactant molecules, but simultaneously limits molecular diffusion and product desorption, leading to incomplete oxidation. Moreover, the acid sites within the clinoptilolite framework may promote secondary reactions such as alcohol formation, ring-opening, or etherification, as evidenced by the detection of several minor oxygenated species in the chromatogram (Mouanni et al., 2019).

In contrast, the bentonite-catalyzed system (Figure 5b) exhibited a markedly different chromatographic profile, with ten prominent peaks corresponding to a diverse array of oxidation products. As illustrated in Figure 5d, adipic acid was again not detected, reaffirming that neither catalyst facilitated the desired deep oxidation to the dicarboxylic acid stage. The bentonite catalyst, characterized by its layered silicate structure and predominantly external active sites, provided a less sterically constrained reaction environment. This structural openness enabled a broader range of parallel oxidation pathways, resulting in the formation of numerous alcohols, ethers, and carbonyl compounds. The presence of multiple low-intensity peaks indicates a non-selective oxidation mechanism, where hydrogen peroxide may decompose rapidly, generating reactive oxygen species that indiscriminately attack various molecular sites on cyclohexanone and its intermediates. These results collectively highlight that the absence of adipic acid formation is not due to the lack of catalytic activity, but rather a consequence of structural and mechanistic mismatches between the catalysts and the reaction requirements. For clinoptilolite, the limitation arises from its microporous channels, which restrict diffusion and confine reactive intermediates, thus preventing further oxidation to adipic acid. For bentonite, the absence of well-defined micropores leads to uncontrolled oxidation, reducing selectivity and promoting the generation of numerous by-products (Gao et al., 2019).

From a broader catalytic perspective, these findings emphasize the pivotal role of catalyst architecture and microenvironment in governing oxidation selectivity. The comparison be-

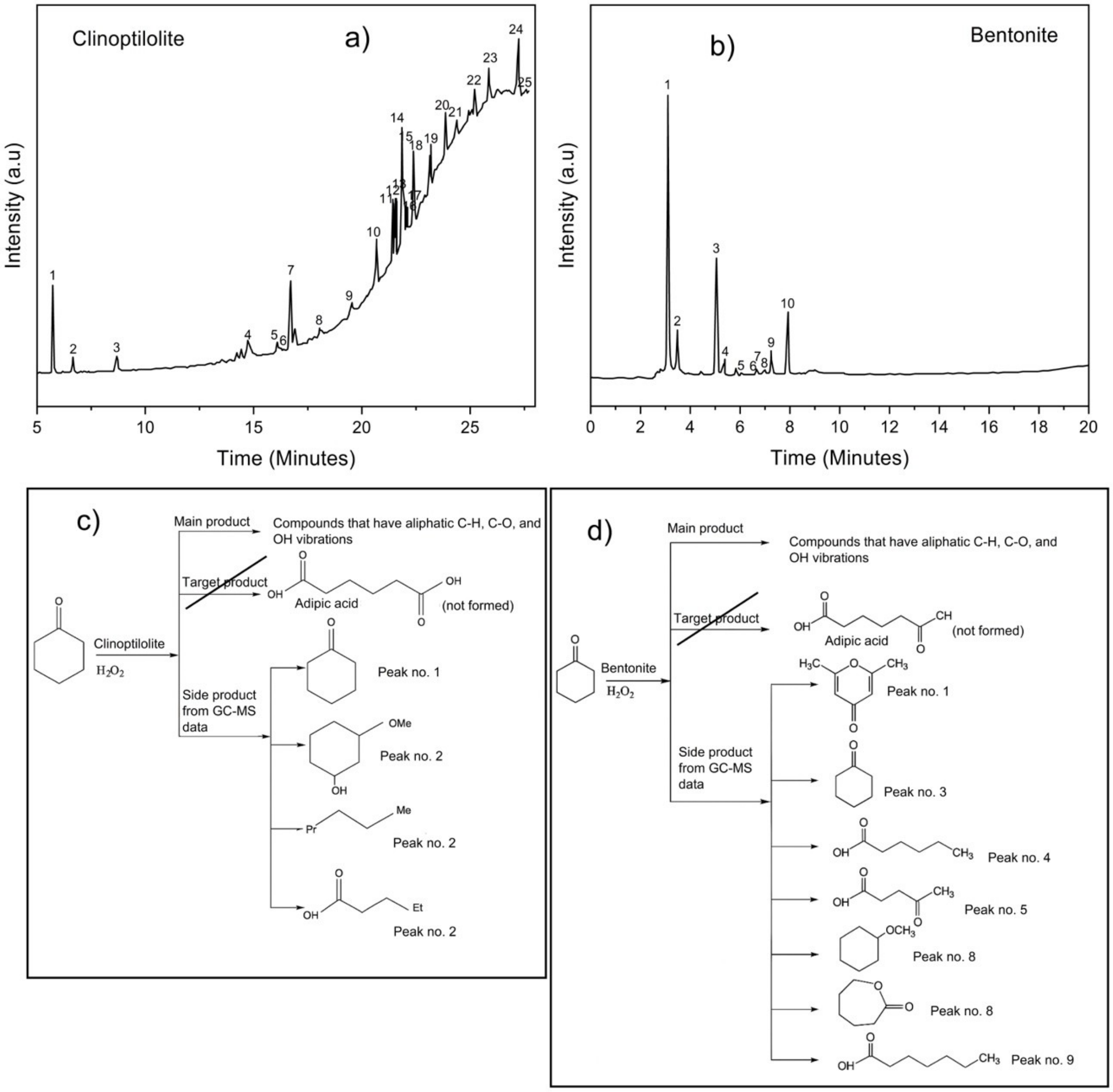

Figure 5. GC–MS Chromatogram and Identified Chemical Structures of the Products Formed During the Catalytic Conversion of Cyclohexanone

tween clinoptilolite and bentonite reveals that a balance between molecular confinement and oxygen accessibility is essential to drive the reaction towards the desired deep oxidation stage. Therefore, future catalyst design strategies could focus on modifying the pore structure, surface acidity, or introducing metal active centers to enhance the oxidative transformation efficiency and favor the formation of adipic acid from cyclohexanone. In summary, while both clinoptilolite and bentonite catalyze the oxidation of cyclohexanone in the presence of H_2O_2, the GC-MS results clearly demonstrate that adipic acid was not formed under the investigated conditions. The reactions predominantly yielded partially oxidized intermediates, the distribution of which is strongly dependent on the physicochemical characteristics of each catalyst. These outcomes not only provide a mechanistic understanding of the oxidation process but also establish a valuable framework for designing more effective catalytic systems for selective oxidation reactions in heterogeneous catalysis.

4. CONCLUSIONS

Natural zeolite (clinoptilolite and mordenite) and bentonite were successfully evaluated as green heterogeneous catalysts for the oxidation of cyclohexanone with hydrogen peroxide. XRD and FTIR analyses confirmed their crystalline aluminosilicate structures, with clinoptilolite exhibiting superior structural order and stability. However, FTIR, melting point, and GC-MS analyses collectively demonstrated that adipic acid was not produced under the studied conditions. Instead, both catalysts promoted partial oxidation reactions, yielding a variety of oxygenated intermediates. The microporous framework of clinoptilolite restricted the oxidation to intermediate stages due to limited molecular diffusion, while the layered and open structure of bentonite allowed uncontrolled oxidation, resulting in a wider but less selective product distribution. These observations highlight that catalytic selectivity in cyclohexanone oxidation is governed by the structural characteristics and surface chemistry of the catalyst. Future work should focus on structural modification, metal ion incorporation, or hybridization strategies to enhance oxidation depth and improve selectivity toward adipic acid formation under sustainable and mild conditions.

5. ACKNOWLEDGEMENT

The authors sincerely acknowledge the Research Center of Inorganic Materials and Coordination Complexes, Universitas Sriwijaya, for providing essential research facilities, laboratory instruments, and valuable technical support that greatly contributed to the completion of this study.

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