

# Precipitation Method for LTA Zeolite Synthesis and Structural Characterization

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## ABSTRACT

The demand for large-scale industrial applications of zeolite has driven the development of synthetic zeolite as an alternative to natural zeolite, which is limited by availability and production constraints. This study investigates the synthesis of LTA (Linde Type A) zeolite using a precipitation method, focusing on the effects of stirring time and aging time on zeolite yield and crystallinity. The synthesis process involved preparing sodium aluminate and sodium silicate solutions, followed by controlled crystallization at 80°C for 8h. The resulting zeolite was analyzed using X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) to determine its phase purity, morphology, and Si/Al ratio. The results showed that optimal zeolite yield (24.75%) was obtained with 3h of stirring and 24h of aging. SEM analysis confirmed the cubic morphology characteristic of LTA zeolite, while EDS analysis determined an Si/Al ratio of 1.44, classifying the product as LTA zeolite. These findings highlight the significance of controlled stirring and aging conditions in optimizing zeolite synthesis for industrial applications.

**Keywords:** aging time, crystallization, LTA zeolite, precipitation, stirring time, synthetic zeolite.

## 1. INTRODUCTION

Zeolite, particularly LTA-type, has a growing demand in various industries, including catalysis, water purification, and gas separation. According to IMARC Group, zeolite market reached 2.20 million tons in 2024, with an annual growth rate of 2%. However, the production of high-purity zeolite faces several challenges, such as raw material availability, synthesis complexity, and energy-intensive processes. The limited availability of zeolite can hinder its industrial application, driving the need for optimized synthesis methods to improve yield and cost-efficiency [1]. This study aims to address these challenges by investigating the effect of stirring and aging time on the synthesis efficiency of LTA zeolite. The first zeolite synthesis was carried out in the late 19th century to find a solution to these limitations [2].

Compared to natural zeolite, synthetic zeolite offers superior selectivity, higher stability, and a more efficient production process with optimal results, making it a preferred substitute [3].

Zeolite consists of an orderly crystalline network with controlled porosity, making it highly valuable for various industrial applications. Its structure is primarily composed of silicon and aluminium, meaning industrial residues containing these elements can be repurposed for zeolite synthesis [4]. However, zeolites cannot be identified solely through chemical composition analysis; instead, they must be characterized using X-ray diffraction (XRD) [5]. A more precise classification of zeolite types is based on their secondary building units, which determine their distinct framework structures [6].

The synthesis of synthetic zeolites typically involves the use of aluminosilicate gel, prepared from sodium aluminate, sodium

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silicate, and sodium hydroxide solutions. Among various synthesis methods, precipitation is widely used because it ensures high-purity zeolite production while maintaining consistent product quality across batches. Additionally, precipitation allows precise control over particle size [7]. This method involves carefully adjusting reaction conditions to control nucleation and crystal growth, resulting in zeolites with desirable properties for specific applications.

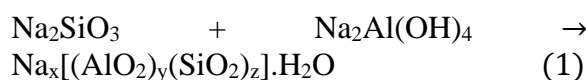
A crucial factor in zeolite synthesis is the selection of raw materials containing silica and alumina [8]. Silica can be obtained from water glass, while aluminium can be sourced from alum or other compounds. Water glass is often chosen as a substitute for sodium silicate solution due to its high silicate content and relatively low cost [9]. The reaction rate, nucleation, and crystal growth during synthesis can be influenced by temperature and concentration [10]. Heat treatment is often applied to study the effects of temperature variations on particle size and crystallinity, with the final products typically characterized using XRD and scanning electron microscopy (SEM) [11].

Precipitation, as a key step in zeolite synthesis, involves adding specific chemicals to induce the formation of solid particles from a solution. This process is widely used to remove or neutralize suspended solids and dissolved pollutants. For instance, phosphates can be precipitated by adding  $\text{Al}^{3+}$  ions, while heavy metals can be removed by adjusting the pH to form insoluble hydroxides [5]. A solution is considered supersaturated if, after slow cooling, excess solute remains dissolved. Higher saturation levels encourage the aggregation of solute particles, leading to crystal formation, while lower saturation results in smaller and less stable crystals. Precipitation occurs more efficiently in supersaturated solutions, as the excess solute naturally forms solid deposits [12].

During zeolite synthesis, NaOH plays a crucial role as an activator, facilitating the formation of silicate and aluminate salts. The  $\text{Na}^+$  ions in NaOH are particularly important

in stabilizing the framework-building units of zeolite [13]. In an alkaline environment, the polymerization of silica and alumina ions occurs, forming a supersaturated solution [14]. These phases exist in equilibrium, with the amorphous gel dissolving and restructuring to form initial crystal nuclei [5]. The process of zeolite formation is driven by the dissolution and reaction of silica and alumina, leading to the development of a silica-alumina framework with active sites capable of trapping heavy metal ions.

The overall reaction for zeolite formation can be summarized as follows [15]:



Stirring duration plays a crucial role in enhancing the contact surface area by increasing stirring speed, which improves the homogeneity of a mixture [16]. In the precipitation process, prolonged stirring increases the reaction temperature and particle collisions, which further influence the crystallization process [17]. Stirring also accelerates the nucleation rate of amorphous gel crystals, leading to the formation of crystal nuclei with a higher silica content. Consequently, more silica is incorporated into the zeolite structure, resulting in an increased silica-to-alumina framework ratio in the synthesized zeolite.

Apart from stirring time, aging time also significantly affects zeolite formation. Aging at ambient temperature enhances the interaction between precursor species, leading to an increased concentration of these species during nucleation. This, in turn, accelerates the nucleation process [18]. The crystallinity of synthetic NaX zeolite depends on the duration of the aging process, with maximum crystallinity achieved after 24h of aging at room temperature [19,20].

Temperature also influences the crystallization process. An increase in temperature accelerates crystal growth and results in larger crystal sizes [21]. Higher synthesis temperatures lead to increased crystal intensity, though they do not

significantly alter the final product composition. Instead, elevated temperatures primarily enhance the intensity of the crystalline phase while reducing the amorphous phase [15].

In general, zeolite synthesis involves dissolving silica, alumina, and a cation in water, where the crystallization process is influenced by heating over a specific period [22]. Earlier studies have explored different raw materials, such as palm kernel shell ash and palm fly ash, with synthesis variations focusing on reactant volume ratios, fly ash/NaOH mass ratios, stirring speed, aging time, and stirring duration [23-25]. This research refers to the findings of Asia et al. (2016), who concluded that the optimal synthesis conditions for zeolite using fly ash were achieved at a volume ratio of 55/45 mL and a synthesis temperature of 80°C [26].

Previous studies have shown that optimizing the stirring and aging time significantly impacts the crystallinity and morphology of LTA zeolite. For instance, research by Siregar et al. (2016) demonstrated that a stirring time of 3h and an aging time of 24h resulted in the highest crystallinity [23]. Additionally, Yelmida et al. (2012) determined that the ideal stirring speed for zeolite synthesis is 200 rpm, as effective mixing is achieved when the material flows turbulently [25].

However, excessive stirring speeds can lead to inefficient mixing, as more material moves tangentially, forming vortices that reduce mixing efficiency [27]. Additionally, excessively high stirring speeds may cause previously bonded ions to detach due to intense particle collisions [24]. The controlled conditions in this study are based on optimized parameters established in previous research to ensure consistency and reproducibility in zeolite synthesis.

## 2. RESEARCH METHODS

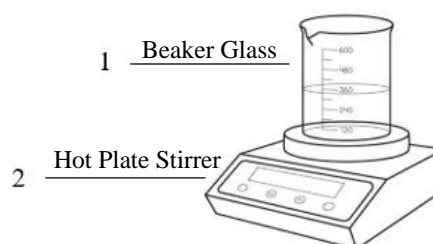
The materials used in this study, including sodium silicate ( $\text{Na}_2\text{SiO}_3$ ), aluminum hydroxide ( $\text{Al}(\text{OH})_3$ ), and sodium hydroxide ( $\text{NaOH}$ ), were of analytical grade with a purity of  $\geq 99\%$  and were purchased from

Bratachem, Surabaya. Distilled water was used as the solvent to ensure the absence of impurities that could affect the synthesis process.

The research consists of three main stages: the preparation of sodium aluminate solution, the dilution of sodium silicate, and the synthesis of synthetic zeolite.

### Preparation of Sodium Aluminate Solution

A sodium aluminate solution was prepared by dissolving 30.5 grams of NaOH in 100 mL of distilled water under constant stirring until fully dissolved as shown in Figure 1. Then, 21.65 grams of  $\text{Al}(\text{OH})_3$  was gradually added while continuously stirring to form a homogeneous solution with a final concentration of 2.77 M. This solution serves as the aluminium source for zeolite formation.



**Figure 1.** Equipment Setup.

### Dilution of Sodium Silicate

A total of 92.95 mL of sodium silicate (waterglass) was diluted with distilled water to reach a final volume of 250 mL, resulting in a solution with a concentration of 2.77 M. This solution provides the silica source necessary for zeolite synthesis.

### Synthesis of LTA Zeolite

The final stage was the synthesis of synthetic zeolite. The sodium silicate solution was stirred into the sodium aluminate solution at a speed of 200 rpm for varying durations of 1, 2, 3, 4, and 5h. This was followed by an aging process with variations of 0, 12, 24, 36, and 48h. The synthesis was then carried out at a temperature of 80°C for 8h. The resulting precipitate was filtered and washed with distilled water (3–5 drops) until it reached a

neutral pH. The precipitate was then dried in an oven at 120°C for 3h.

### Characterization of Synthesized Zeolite

The crystalline structure of the synthesized LTA zeolite was characterized using Powder X-ray Diffraction (PXRD). The morphology and elemental composition of the synthesized LTA zeolite were analysed using Scanning Electron Microscopy (SEM) equipped with Energy Dispersive X-ray Spectroscopy (EDS).

### Yield Calculation

The yield calculation of LTA zeolite was performed to determine the synthesis efficiency based on the amount of the obtained product compared to the total

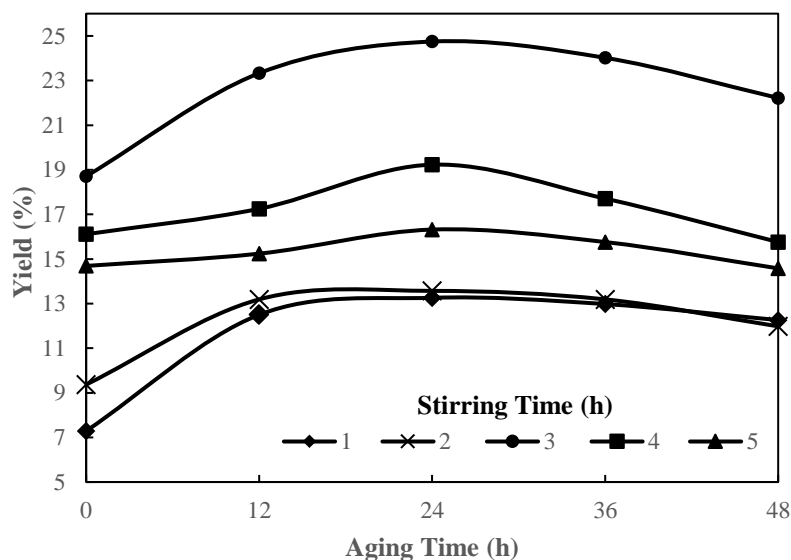
precursor used. The yield was calculated using the following equation:

$$\% \text{yield} = \frac{\text{Actual Mass}}{\text{Theoretical Mass}} \times 100\% \quad (2)$$

Where the actual mass refers to the weight of the synthesized LTA zeolite, and the theoretical mass represents the total mass of both precursor materials used in the synthesis.

## 3. RESULTS AND DISCUSSION

The research data indicates that stirring time and aging time significantly influence the formation of zeolite. The graphical representation of this data is presented in Figure 2.



**Figure 2.** The Effect of Stirring Time and Aging Time on Zeolite Yield.

Based on Figure 2, there is a relationship between stirring time, aging time, and zeolite yield. The yield increases with initial aging, reaching a peak at 24h before declining at 48h. Similarly, longer stirring durations generally result in higher zeolite yield, particularly at shorter aging times. The optimal yield of approximately 24.75% was achieved with 3h of stirring and 24h of aging, whereas shorter stirring durations, such as 1 hour, only produced a maximum yield of around 13.26%. This indicates that the interaction between stirring time and aging

time significantly influences zeolite formation efficiency, where prolonged stirring enhances the formation of zeolite structures during the aging process [28].

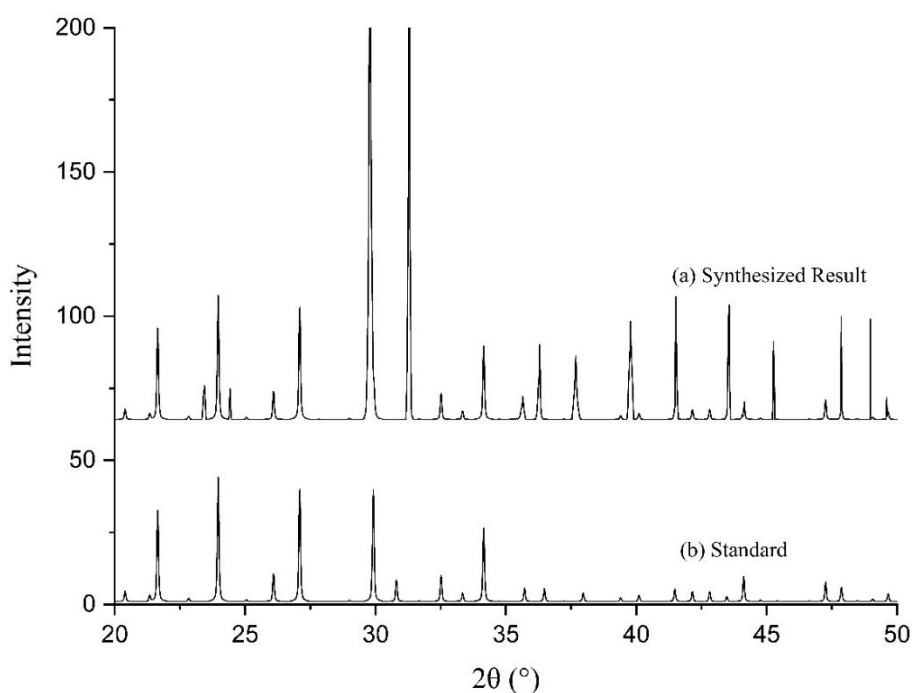
The figure also shows that the variation in stirring time significantly affects the zeolite yield, highlighting the crucial role of stirring in zeolite formation. Longer stirring durations enhance precursor homogeneity, ensuring a more uniform chemical reaction during aging, which in turn results in better zeolite structure and higher yield [29]. This is evident in the 3h curve, which exhibits the

highest yield compared to other stirring durations. However, excessive stirring time can reduce effectiveness, as excessive mechanical energy may disrupt the initial zeolite structure or accelerate the dissolution of the formed material [30].

The statistical significance of these trends was analysed by comparing the relative yield differences across stirring and aging variations. The sharp increase from 1h to 3h stirring (from 13.26% to 24.75%) confirms a strong correlation between stirring efficiency and zeolite formation. However, after 24h of aging, the yield decline suggests a potential saturation point where extended aging may favour secondary reactions over zeolite crystallization.

Additionally, aging time significantly affects the zeolite formation yield. During the initial

aging phase (0h - 24h), there is a sharp increase in yield, particularly for longer stirring times. This is likely due to the improved crystallization of zeolite structures during aging [18]. Aging provides the precursors time for structural reorganization, enhancing zeolite crystal formation efficiency. However, beyond 24h, the yield starts to decline gradually, which could be attributed to decomposition or partial dissolution of the formed product, especially under prolonged aging conditions [31]. This factor can reduce zeolite crystal formation efficiency, leading to a lower yield as aging time increases. Thus, finding the optimal combination of stirring and aging time is essential for maximizing yield, as demonstrated by the peak at 24h of aging with 3h of stirring.



**Figure 3.** Comparison of Standard and Synthesized LTA Zeolite XRD Pattern.

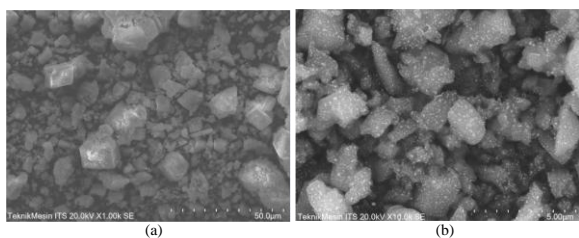
Stirring time is one of the key factors influencing crystallization kinetics, particle size, particle size distribution, and the final silica-to-alumina framework ratio of zeolites. Stirring accelerates crystal formation from the liquid phase onto the growing zeolite crystal surface, fostering the formation of viable nuclei to enhance crystallization and crystal growth rates [32]. On the other hand,

excessive stirring can also lead to the dissolution of nuclei or viable zeolite crystals, reducing the overall crystallization rate and crystal growth. The effect of stirring time is particularly significant in determining the rate at which viable zeolite nuclei transition into fully formed crystals [33].

The synthesized zeolite was characterized using XRD to determine its crystalline

structure. The XRD analysis provides information about the microstructure of a crystal, including its phase purity and lattice structure changes based on the  $2\theta$  angle. As shown in Figure 3, in addition to crystalline phases, amorphous and luminosilicate phases were also detected. The diffractogram indicates that both materials predominantly consist of sodium silicate and sodium alumina silicate, as evidenced by the matching diffraction peak patterns.

The zeolite obtained in this study was analysed by comparing its characteristic  $2\theta$  peaks and d-spacing values with the Collection of Simulated XRD Powder Patterns for Zeolites. The synthesized zeolite showed characteristic  $2\theta$  and d-spacing values similar to those of standard LTA zeolite. Thus, the synthesized zeolite was identified as LTA (Linde Type A) zeolite.



**Figure 4.** (a) SEM Image at 1000x Magnification, (b) SEM Image at 10000x Magnification.

In addition to XRD analysis, Scanning Electron Microscopy (SEM) was conducted to examine the morphology of the synthesized LTA zeolite, including its shape, texture, and microstructural features, as shown in Figure 4. Furthermore, chemical composition identification was performed using Energy Dispersive Spectroscopy (EDS) to determine the Si/Al ratio, providing further insight into the material's structural characteristics. Based on SEM analysis, the morphology of the synthesized zeolite exhibits a tendency toward the cubic shape characteristic of LTA zeolite. However, it should be noted that the cubic morphology is not perfectly defined, as some irregularities are observed in the crystal structure. Despite this, the overall shape aligns with the

expected morphology of LTA zeolite. Additionally, the chemical composition was analysed using Energy Dispersive Spectroscopy (EDS) to determine the Si/Al ratio.

**Table 1.** Result of Energy Dispersive Spectroscopy (EDS) Analysis.

| Element Atomic | %     |
|----------------|-------|
| O              | 61.80 |
| Si             | 18.75 |
| Na             | 6.50  |
| Al             | 12.95 |

The EDS analysis revealed that the atomic percentage of Si was 18.75% and that of Al was 12.95% as shown in Table 1. The calculated Si/Al ratio was 1.44, confirming that the synthesized zeolite belongs to the LTA type. According to literature sources such as Introduction to Zeolite Science and Practice and Handbook of Zeolite Science and Technology, the Si/Al ratio for zeolite A typically ranges between 1.0 and 1.5.

LTA zeolite is highly valuable due to its extensive applications in both laboratory and industrial settings. It serves as an adsorbent, catalyst, membrane material, and ion exchanger. Owing to its low silica content, LTA zeolite exhibits a high affinity for water molecules.

In 1974, Henkel introduced zeolite A in detergents as an environmentally friendly substitute for phosphate-based additives. Studies have shown that, despite being insoluble, LTA zeolite does not cause excessive fiber buildup due to its optimized particle shape (rounded edges) and particle size (average size of 3.5  $\mu\text{m}$ ). LTA zeolite plays a crucial role as an ion exchanger in detergents, making it a significant component in modern cleaning products [34].

#### 4. CONCLUSION

The synthesis of LTA zeolite using the precipitation method was achieved by optimizing stirring duration and aging time. The highest zeolite yield of 24.75% was

obtained with 3h of stirring and 24h of aging. X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses confirmed the crystalline structure and morphology of LTA zeolite, with a Si/Al ratio of 1.44, aligning with standard values. The results emphasize the critical role of process parameters in enhancing zeolite formation efficiency. With its well-defined structure, the synthesized LTA zeolite holds significant potential for industrial applications as an adsorbent, catalyst, and ion-exchange material. Further research could explore additional synthesis conditions and large-scale production feasibility.

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