Synthesis of Zeolite A from Bangka Kaolin as a High-Performance Host for Fluorescent Compounds

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Abstract. This study aimed to synthesize Zeolite A from Bangka kaolin and evaluate its potential as a high-performance host material for fluorescent compounds in latent fingerprint identification. Zeolite A was synthesized using the hydrothermal method and characterized by X-ray diffraction (XRD), Fourier Transform Infrared (FTIR), and Scanning Electron Microscopy (SEM). The characterization results confirmed the successful formation of Zeolite A with an optimum crystallization time of 5 h, producing a crystallinity of 47%. Structural analysis indicated the development of a uniform aluminosilicate framework suitable for hosting fluorescent molecules. Fluorescence spectroscopy was employed to investigate the optical performance of the zeolite-based fluorescent material under different dye concentrations and contact times. The results showed that the highest fluorescence intensity was achieved at 100% dye concentration with a contact time of 30 min. The enhanced fluorescence emission demonstrated Zeolite A's ability to serve as an efficient host matrix for fluorescent compounds.

1 Introduction

The development of porous aluminosilicate materials has attracted significant attention in materials chemistry and photonic applications due to their ability to regulate molecular interactions at the microscopic scale [1]. Among these materials, zeolites are particularly promising because of their well-defined pore structures, high surface area, and excellent thermal and chemical stability [2]. These properties make zeolites widely applicable not only as adsorbents, catalysts, and ion exchangers, but also as host materials for optical and fluorescent compounds [3]. In fluorescent systems, the porous zeolite framework can

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immobilize fluorescent dye molecules in a highly dispersed, stable state, thereby enhancing emission intensity, reducing molecular aggregation, and improving photochemical stability [4]. Zeolite A is one of the most important synthetic zeolites due to its uniform pore size and low Si/Al ratio, which provide suitable cavities and channels for hosting fluorescent molecules [5]. The regular aluminosilicate framework of Zeolite A enables efficient confinement of dye molecules, thereby improving optical performance. However, most previous studies have relied on commercial chemical precursors such as sodium silicate and sodium aluminate, which are relatively expensive and less sustainable. Therefore, the use of natural aluminosilicate minerals as alternative precursors for zeolite synthesis has become increasingly important for the development of environmentally friendly, cost-effective functional materials [6]. Kaolin is a natural material that provides greater ease of conversion and more uniform zeolite structure compared to other natural materials, including bauxite and fly ash [7].

Bangka kaolin is an abundant natural aluminosilicate mineral in Indonesia with high silica and alumina contents, making it suitable for zeolite synthesis [8]. The conversion of kaolin into Zeolite A not only increases the economic value of local mineral resources but also provides an alternative route for producing advanced porous materials from natural sources. Although the synthesis of zeolites from kaolin has been widely reported, studies focusing on the application of kaolin-derived Zeolite A as a host matrix for fluorescent compounds remain very limited. The novelty of this research lies in the utilization of Zeolite A synthesized from Bangka kaolin as a high-performance porous host material for fluorescent dyes in latent fingerprint detection applications. In this study, Zeolite A was synthesized using a hydrothermal method and characterized to evaluate its crystal structure, morphology, and optical properties after interaction with fluorescent dyes. This work is expected to contribute to the development of zeolite-based fluorescent materials for forensic identification and advanced photonic applications.

2 Methods

This study employed an experimental approach to synthesize Zeolite A from kaolin and evaluate its performance as a host matrix for fluorescent Rhodamine B molecules. The synthesis procedure began with the metakaolinization of kaolin via calcination to activate the aluminosilicate structure and produce reactive metakaolin. The obtained metakaolin was subsequently mixed with an alkaline solution containing NaOH and deionized water under continuous stirring until a homogeneous gel was formed. The precursor gel was then aged for 2 h to promote aluminosilicate rearrangement and nucleation. Following aging, hydrothermal crystallization was conducted with crystallization time variations of 3, 5, 7, 9, and 12 h to investigate their effects on the formation and crystallinity of Zeolite A. The synthesized zeolite products were separated, washed, and dried prior to further application. To prepare fluorescent powders, Zeolite A was used as a porous host material for Rhodamine B dye at concentrations of 10%, 30%, 50%, 70%, and 100% (w/w). The dye adsorption process was further evaluated at contact times of 30 min, 1 h, 12 h, 1 day, 2 days, and 3 days to optimize dye incorporation into the zeolitic framework. The resulting fluorescent composites were subsequently dried in an oven before characterization and application. Rhodamine B was employed as a model fluorescent molecule to evaluate the hosting capability of the synthesized Zeolite A.

3 Results

The transformation of kaolin to metakaolin was validated by X-ray diffraction (XRD) spectroscopy. Measurements were made at an angle range of 2θ 2˚ - 60˚. The analysis of Bangka kaolin revealed several mineral mixtures; the highest peaks, characteristic of kaolin, appeared at angles of 12.30˚, 24.84˚, and 38.38˚. This is in accordance with the reference database software used in the XRD characterization. The diffractogram of Figure 1 shows the difference between the peaks of kaolin and metakaolin. The disappearance of some of these peaks is due to dehydroxylation, which changes the crystal structure to an amorphous state. In the metakaolin diffractogram, there are peaks at 20.83˚ and 26.58˚. These peaks are quartz peaks. The crystal structure of quartz cannot be damaged at a temperature of 750˚C. The diffractogram in Fig.1 shows that metakaolin is amorphous. This is indicated by the sloping background baseline and the few peaks observed. The amorphous phase of quartz can be confirmed by measuring its degree of crystallinity using OriginPro software. The calculated degree of crystallinity of metakaolin is 1.86%, which is smaller than the degree of crystallinity of kaolin, which is 19.4%. This is because metakaolin is amorphous, while kaolin is crystalline. The zeolite A synthesized in this study exhibited higher crystallinity and improved structural uniformity compared to previously reported samples, which demonstrated relatively low crystallinity and uniformity [9].

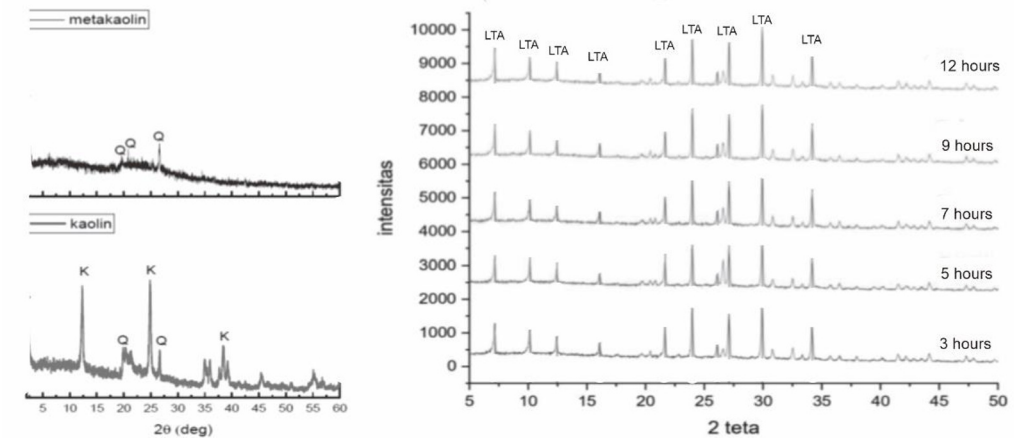

Fig 1. Diffractogram of kaolin, metakaolin and variation of crystallization time of zeolite A.

The morphology shown in Figure 2 is that of a cubic zeolite A structure. This is in line with the statement that the zeolite A crystal system is cubic. In the 12-hour crystallization variation, the degree of crystallinity decreases because the nucleation of sodalite has a higher density than zeolite A. Nucleation occurs in the amorphous phase when the cubic crystal system particles receive pressure. Before zeolite A crystals are formed, sodalite crystals begin to grow within the core of the cubic crystal system [10]. Figure 2 SEM demonstrates that zeolite A exhibits a highly uniform morphology, with particle sizes approximately 2 mm and a minor presence of residual reactant impurities as smaller flakes. Such structural uniformity is critical for the effective incorporation of fluorescent compounds, as it facilitates a consistent distribution of fluorescent molecules within the zeolite A framework.

In this study, rhodamine B was used as the dye. This dye was chosen because it is fluorescent, so it is expected to provide better visual results than traditional fluorescent powder. This study also used the cationic surfactant cetrimonium bromide (CTAB). The surfactant was used to contact the oil components in fingerprints. The powder was then dried at 100°C and sieved. Sifting was done to avoid clumps when applied to fluorescent media. The hypothesis of using a surfactant in the synthesis of fluorescent powder is that the dye is expected to be trapped within the cavities of zeolite A. The reversible nature of cation

exchange can make the dye short-lived, so the surfactant was used to close the cavities of zeolite A and act as a hydrophobic materials.

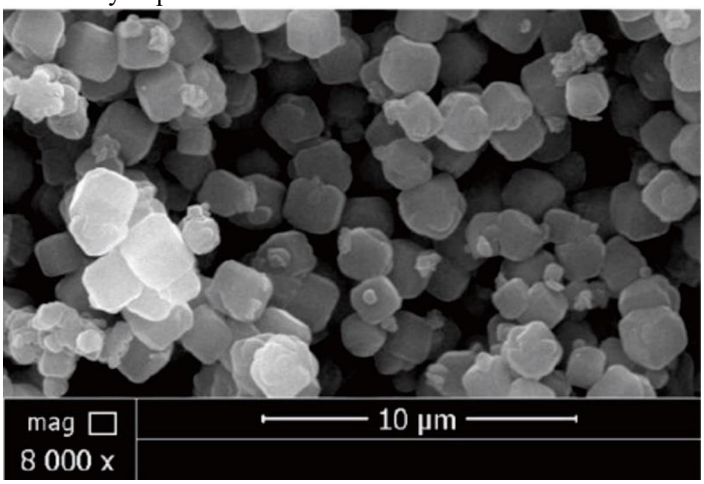

Fig 2. SEM of as synthesized Zeolite A

Fluorescence spectroscopy can be used to validate the effect of using CTAB or rhodamine B in the fluorescent powder composition. The emission wavelength of rhodamine B was 556 nm. Variations in the rhodamine B dye percentage and contact time were then performed to determine the optimum fingerprint powder composition. The dye percentages were 10%, 30%, 50%, 70%, and 100%. The effects of these variations were also observed with and without CTAB. The image shows the effect of the dye variation without surfactant, with the highest intensity at 100%. This occurs due to the principle of fluorescence spectroscopy: the greater the amount of fluorescent dye, the higher the intensity. The same is also shown in the image of the dye percentage variation with CTAB, with the highest intensity at 100%.

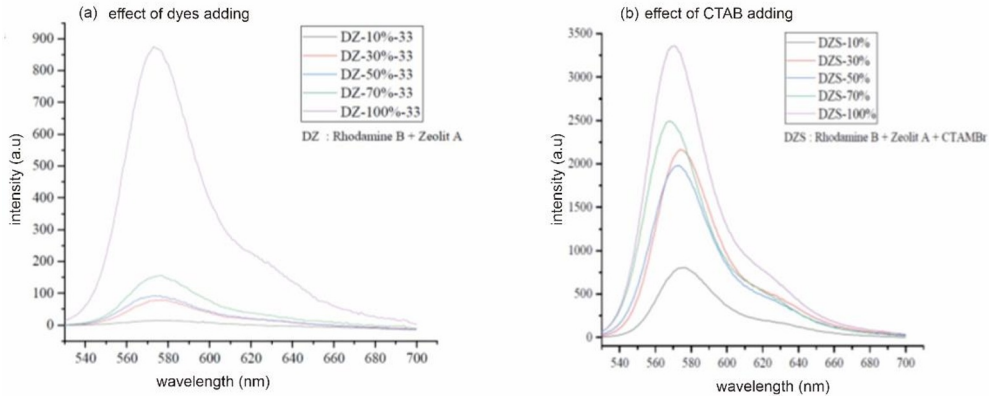

Fig 3. Fluorescence graph of variation in % of dye without CTAB, (b) with CTAB

The role of CTAB is evident in its high intensity; in graphs without CTAB, the intensity is lower, due to the washing process. Fluorescent that does not use CTAB loses a lot of rhodamine B during the washing process. Meanwhile, those using CTAB rhodamine B will remain in the zeolite A matrix. The reversible nature of cation exchange in zeolite A is what makes rhodamine B not last long in the zeolite A matrix, so that other molecules are needed to maintain rhodamine B in the zeolite A matrix. Figure 3 shows the combined spectra of the fluorescence without CTAB and with CTAB. Figure 3 illustrates the synergistic interaction between rhodamine B and CTAB. The introduction of CTAB to zeolite loaded with

rhodamine B increases the luminescence intensity to nearly four times the original value, resulting in a narrower emission profile and a blue shift.

4 Conclusions

Zeolite A was successfully synthesized from Bangka kaolin through a hydrothermal method and demonstrated significant potential as a high-performance host material for fluorescent compounds. Structural characterization by XRF, XRD, FTIR, and SEM confirmed the formation of a crystalline aluminosilicate framework, with the optimal crystallization time at 5 h, yielding a crystallinity of 47%. The uniform porous structure of Zeolite A enabled effective incorporation of Rhodamine B molecules within the zeolitic framework. Fluorescence studies revealed that the highest emission intensity was obtained at 100% dye concentration with a contact time of 30 min, indicating efficient interaction between the fluorescent molecules and the zeolite host matrix. The enhanced fluorescence performance demonstrates that the synthesized Zeolite A can stabilize and disperse fluorescent compounds effectively within its porous structure.

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