

Green Zeolite Synthesis Assisted with Plants Extract

Dee Tee¹, Faiz Hafeez Azhar¹, Zawati Harun^{1*}, Rosniza Hussin^{1,2}, Syamsutajri Syamsol Bahri¹, Nur Hanis Hayati Hairom³, Ainun Rahmahwati Ainuddin², Hatijah Basri⁴, Zakiah Kamdi²

¹Advanced Manufacturing and Materials Centre (AMMC), Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia (UTHM), 86400, Parit Raja, Batu Pahat, Johor, MALAYSIA

²Nano Structure and Surface Modification Research Group, Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia (UTHM), 86400, Parit Raja, Batu Pahat, Johor, MALAYSIA

³Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia, Hab Pendidikan Tinggi Pagoh, KM1, Jalan Panchor 86400, Muar, Johor, MALAYSIA

⁴Faculty of Applied Sciences and Technology, Universiti Tun Hussein Onn Malaysia, Pagoh Campus, 84600, Pagoh, Muar, Johor, MALAYSIA

*Corresponding Author

DOI: <https://doi.org/10.30880/emait.2023.04.01.007>

Received 5 June 2023; Accepted 20 July 2023; Available online 31 July 2023

Abstract: The study explores the hydrothermal synthesis of zeolite using kaolin clay from Perak in the presence of two different reduction mediums, namely turmeric extract and ginger extract, and a 2M NaOH solution, measuring their efficiency and potential for green synthesis. Before the zeolite synthesis and transformation, the kaolin clay was heated at a temperature of 650 °C for 4 hours in the furnace to produce the metakaolin phase. The synthesized zeolite involves the reaction of NaOH with potential reduction plant extracts to assist the transformation of zeolite. The characterization of the synthesized particles after being ground into a fine powder and subsequently undergoing testing and characterization via X-ray diffraction (XRD) revealed the existence of zeolite phases in both plants with slightly different zeolite compositions. Synthesis mixtures with turmeric plants show better composition of the zeolite phase as compared to ginger mixtures. Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray Analysis (EDX) also revealed that the synthesized product using turmeric plant extract was able to produce zeolite LTA with a good grade. The current experimental work shows the possibility of obtaining a well crystalline zeolite from Malaysian kaolin, assisted with plant extract reduction medium.

Keywords: Zeolite particle, plant extract, synthesis

1. Introduction

In general, zeolites are crystalline solid structures made of silicon, aluminium, and oxygen that have cavities and channels that can hold cations, water, or small molecules [1]. The geometry of zeolites is a highly ordered microporous structure containing an aluminosilicate structure and extensively used as adsorbents, catalysts, separations, and many other critical industry applications [2]. Mesoporous and micropore structures provide a huge surface area and a large number of active sites, which allows them to adsorb a wide spectrum of molecules, including gases, liquids, and dissolved ions [3]. Thus, zeolites are widely employed as adsorbents to remove pollutants from liquids or gases, such as in water treatment and air purification. Zeolites can be used as catalysts to accelerate chemical reactions by providing

active reactants and sites [4]. This important structure is able to minimize the amount of energy required to start the reaction and increase the rate of the process.

The geometry of zeolites, which is consistent with microspore structure and is crucial in many applications, has led to the improvement of zeolite synthesis technique and zeolite structure, making zeolite synthesis an active field of research until now [5]. The hydrothermal synthetic technique has emerged as the primary method for synthesizing zeolite as compared to other techniques. The term "hydrothermal synthesis" typically refers to processes that take place in closed systems at high temperatures and high pressures [6]. In this present work, hydrothermal techniques will be used in different reaction mediums as a controlled parameter to observe the ability of plant extract as a reduction medium.

2. Methodology

2.1 Materials

At the beginning of the experiment, the kaolin clay was activated at 650°C to produce metakaolin [7]. The plant extract solution was produced by diluting 20g of pineapple extract powder, ginger extract powder, and turmeric powder into deionized water.

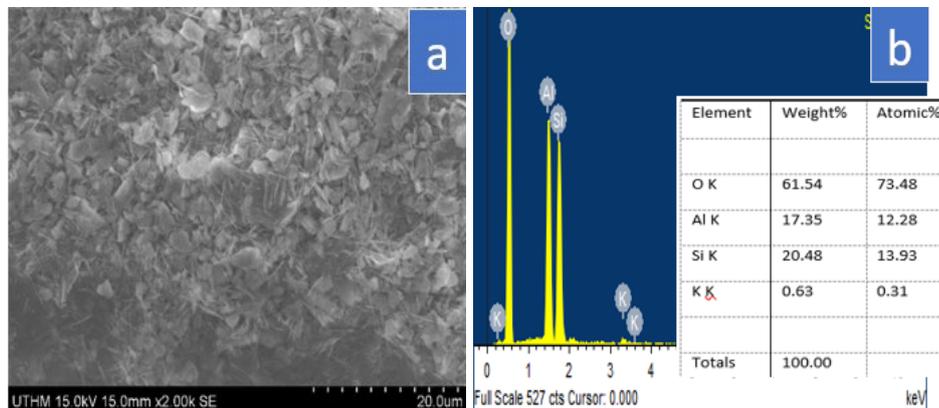
2.2 Synthesis of the Zeolite Particles

Before the crystallization, a part of the activated kaolin was used to prepare the reaction mixture in a 100 mL glass vessel containing a 15 mL plant extract solvent of pineapple extract, turmeric extract, and ginger extract, and a 2 M NaOH solution. The reaction mixture was aged for 12 h at 40°C.

Immediately after the end of the ageing process, the reaction mixtures were transferred into Teflon-lined autoclaves. The samples were crystallized in an oven at a temperature of 100 °C for 12 h. After the crystallization process, the white precipitates were collected and washed several times with deionized water until the pH was below 10. At the end of the washing, the product obtained is dried in the oven at 100°C for 12 h. The final product is ground to a fine powder and subsequently undergoes testing and characterization via x-ray diffraction(XRD) and scanning electron microscope (SEM).

3. Results and Discussions

The SEM images shown in Fig. 1(a) describe the raw kaolin material prior to thermal activation. The sample consists of a small flaky morphology kaolin particle [8]. The EDX spectra in Fig. 1(b) show the peak corresponding to the element of the kaolin powder. From the spectra, Al and Si elements were found at high, strong peaks, with confirm the existence of kaolinite in the sample [9]. The metakaolin Fig. 1(c) obtained at the end of the hydrothermal activation operation was used for the crystallization process. The crystallization was carried out at 100 °C for 12 hours. The purity and grade of the synthesized product vary accordingly with the suitability of the reaction solvent.



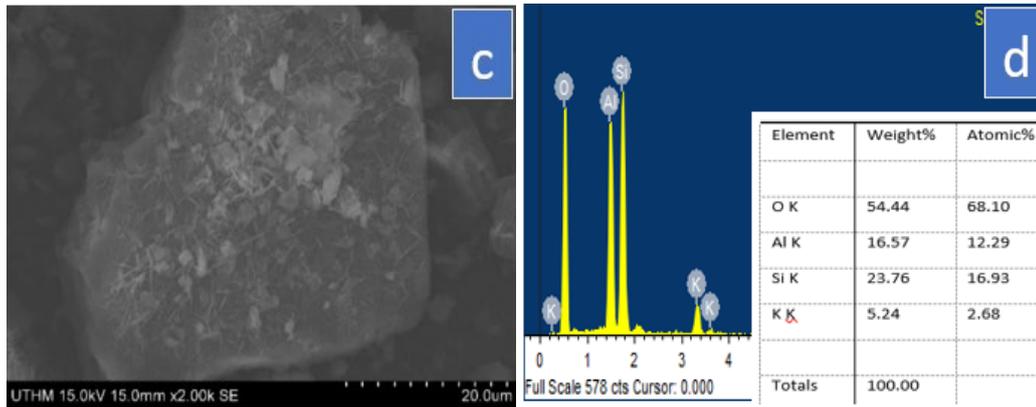


Fig. 1 - SEM image and EDX spectra for (a-b) kaolin (c-d) metakaolin

Fig. 2(a) describes the result obtained for the XRD analysis of sample D (3 g of metakaolin + turmeric extract solution + 2 M of NaOH (60 mL)), with one main composition present, which is Zeolite LTA (100%). At the sharp peak, $2\theta = 29.987^\circ$ indicates a match with Zeolite LTA, ICDS 98-001-2640 with cubic structure [10]. Fig. 2(b) describes the result obtained for the XRD analysis of sample F (3 g of metakaolin + ginger extract solution + 2 M NaOH (60 mL)), where there are two main compositions present, which are Zeolite A (99.6%) and Quartz (0.4%). At the sharp peak of $2\theta = 26.217^\circ$, it indicates a match with Zeolite A, ICDS 98-004-6965 with cubic structure [11]. A sharp peak of $2\theta = 26.524^\circ$ indicates a match with Quartz, ICDS 98-010-7204 with cubic structure. It is obviously shown that the sample made with the 15 mL turmeric plant extract was able to develop the crystallization reaction that can yield high quality Zeolite LTA with purities of 100 %, while the sample made with ginger extract can yield Zeolite A with purities of 99.6 % as shown in Fig. 2(b).

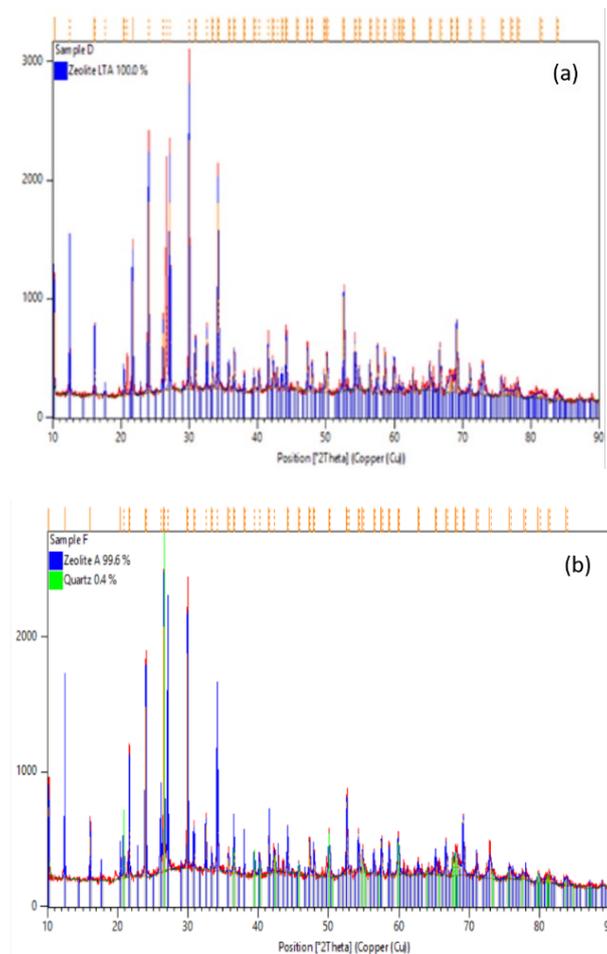


Fig. 2 - XRD Diffractogram of the synthesized product (a) sample using turmeric plant extract (b) sample using ginger plant extract

Considering Fig. 2(a), the XRD pattern shows that the zeolite obtained from the sample of turmeric plant extract has a stable yield of zeolite LTA with no presence of quartz thus, Fig. 3(a) revealed that the shape of the synthesized zeolite under this condition is almost fully cubic in shape [11]. This indicates that turmeric medium is able to assist the zeolite transformation with a nearly diminishing interaction with zeolite growth with NaOH medium as compared to ginger, which has a slight interaction with the zeolite transformation.

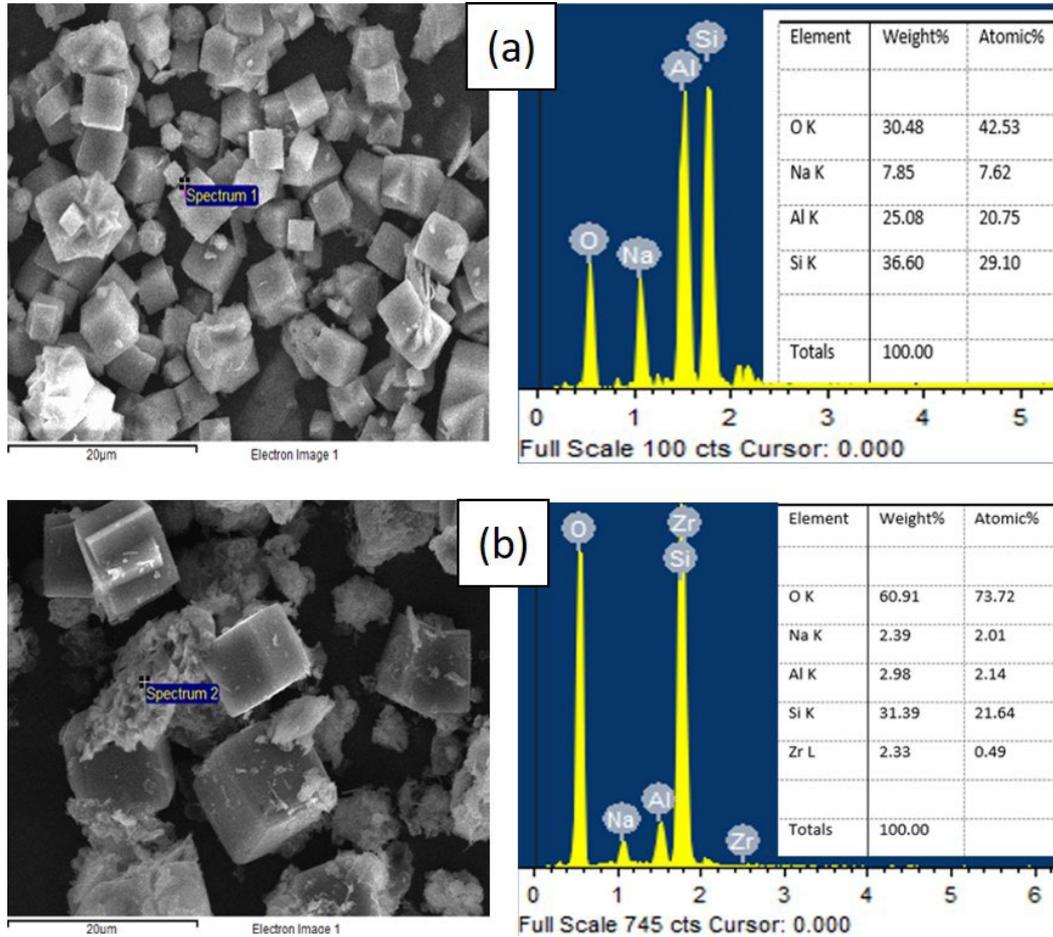


Fig. 3 - SEM and EDX result of the synthesized zeolite for (a) sample using turmeric plant extract; (b) sample using ginger extract

4. Conclusion

The synthesis process using a plant extract medium from turmeric plant extract was able to produce zeolite LTA with a good grade compared with ginger plant extract. The result also portrays the possibility of obtaining a well-crystalline zeolite from low-grade kaolin in Malaysia using a zeolite synthesis mixture with plant extract. Therefore, it is highly recommended to further study the influence of the concentration of plant extract on assisting the zeolites' synthesis and growth using the conventional technique.

Acknowledgement

The authors would like to thank the Yayasan Pelajaran Johor (YPJ) for supporting this research under the Scholarship of Sultan Ibrahim or Biasiswa Sultan Ibrahim (BSI). Also, an acknowledgement to the Advanced Manufacturing and Material Centre (AMMC) and Faculty of Mechanical and Manufacturing Engineering (FKMP) for providing the facilities to conduct experimental work and lab testing.

References

[1] T. Abdullahi *et al.*, "Synthesizing of Zeolite Particle Using Alkaline Plant Extract," *Emerg. Adv. Intergrated Technol.*, vol. 2, no. 1, pp. 1–6, 2021.

- [2] N. Sazali *et al.*, “The Route of Hydrothermal Synthesis Zeolite-A from the Low-Grade Perak kaolin, Malaysia,” *Silicon*, vol. 14, no. 12, pp. 7257–7273, 2022.
- [3] A. A. Vasconcelos *et al.*, “Zeolites: A Theoretical and Practical Approach with Uses in (Bio)Chemical Processes,” *Appl. Sci.*, vol. 13, no. 3, 2023.
- [4] M. Moliner and M. Boronat, “Towards ‘enzyme-like’ zeolite designs to maximize the efficiency of catalysts by molecular recognition: Fine-tuning confinement and active site location,” *Microporous Mesoporous Mater.*, vol. 358, no. September 2022, p. 112354, 2023.
- [5] F. H. Azhar *et al.*, “Simple Approach in Measuring the Synthesized Zeolite from Kaolin as Nutrient Retention for Mung Bean Growth,” *Emerg. Adv. Integr. Technol.*, vol. 3, no. 2, pp. 52–59, 2023.
- [6] Y. X. Gan *et al.*, “Hydrothermal Synthesis of Nanomaterials,” *J. Nanomater.*, pp. 1–3, 2020.
- [7] I. Bošković *et al.*, “Characterization of red mud/metakaolin-based geopolymers as modified by $\text{Ca}(\text{OH})_2$,” *Mater. Tehnol.*, vol. 53, no. 3, pp. 341–348, 2019.
- [8] M. A. Fadzil *et al.*, “Characterization of Kaolin as Nano Material for High Quality Construction,” in *MATEC Web of Conferences*, 2017, vol. 103, p. 09019.
- [9] S. Mustapha *et al.*, “The role of kaolin and kaolin/ZnO nanoadsorbents in adsorption studies for tannery wastewater treatment,” *Sci. Rep.*, vol. 10, no. 1, pp. 1–22, 2020.
- [10] N. Sazali and Z. Harun, “One Shot of the Hydrothermal Route for the Synthesis of Zeolite LTA Using Kaolin,” *J. Inorg. Organomet. Polym. Mater.*, vol. 32, no. 9, pp. 3508–3520, 2022.
- [11] N. Sazali *et al.*, “Adsorption of Sodium Ion (Na^+) onto Synthesized Zeolite-A from Malaysian Kaolin by Hydrothermal Method: Equilibrium and Kinetic Studies,” *J. Adv. Res. Appl. Sci. Eng. Technol.*, vol. 31, no. 1, pp. 53–67, 2023.
- [12] S. Hu *et al.*, “Influence of alkalinity on the synthesis of hierarchical LTA zeolite by using bridged polysilsesquioxane,” *RSC Adv.*, vol. 9, no. 5, pp. 2551–2558, 2019.