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Innovation and Characterization of Zeolite from Matoa Fruit for Adsorption of Heavy Metals Cu(II)

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ABSTRACT

Matoa fruit is abundant in Papua and has not been optimally utilised. Matoa fruit consists of ash approximately 3.74%. The high ash and silica content make it highly suitable as a raw material for zeolite production. Zeolite can be used as a zeolite material to adsorb heavy metal Cu(II). Heavy metal pollution continues to increase with industrial growth. Heavy metals can harm human health. Innovation must continue addressing heavy metal pollution, such as Cu(II), to produce more efficient, cost-effective, and environmentally friendly technologies. This study aims to determine the ability of matoa fruit zeolite to adsorb heavy metal Cu(II). The characterization of zeolite was done using XRD (X-Ray Diffraction) and SEM (Scanning Electron Microscope). The synthesis process involved preparing sodium aluminate and sodium silicate solutions, followed by controlled crystallisation at 105°C for 24 hours. The zeolites were tested for adsorption with the independent variable being zeolite mass, while the dependent variables were contact time of 48 hours and stirring time of 4 hours. SEM analysis confirmed the cubic morphological characteristics of matoa fruit zeolite, classifying the product as matoa fruit zeolite. The main findings of the study showed that matoa fruit zeolite can reduce Cu metal levels in sewage-polluted water with 88.85% adsorption at 0.5 g in 50 ml of test solution.

Keywords: adsorption, Cu metal, matoa fruit, silica, zeolite.

1. INTRODUCTION

The matoa plant is a plant that grows a lot and is very abundant in Papua [1]. Matoa plants have not been optimally utilised. The high silica ash content in matoa fruit has potential for making zeolite.

Heavy metals such as Cu(II), Ni, Co, and Fe(II) result from industrial activities that can pollute waters and harm living organisms [2-4]. Previous research in 2023 reported that the waters of Puri Bridge in Sorong City have been polluted with heavy metal Cu(II), causing adverse effects on the environment [5]. This is due to the extensive use of Cu(II) metal in various fields such as copper used as a mixture of paint materials, pesticides and the Cu(II) in refining and smelting industry [6,7]. The presence of Cu(II) metal in the environment can also negatively impact human health. It can inhibit the work of enzymes in the body, preventing metabolic processes from running normally. In addition, it can cause carcinogenic properties in the human body.

Adsorption is one of the methods used to solve problems related to heavy metal pollution in water [8,9]. Adsorption is the process of accumulation of substances at the interface of two phases [10]. One method in wastewater treatment is adsorption. This process is a chemical process where all bonds are formed between zeolite acceptors and heavy metals [11,12]. The effectiveness of wastewater treatment using this method is simple and cost-effective. One of the widely used adsorption materials is zeolite [13,14].

Zeolites can be synthesised from natural materials containing high silica ash. Zeolites can degrade heavy metal content in industrial wastewater [15]. Zeolite is a material that can adsorb substances on its surface due to the attractive force between adsorbate molecules and the adsorbent surface. Zeolite is an aluminosilicate crystal with the elements Si

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and Al as the main constituents [16]. Heavy metals can substitute cations in zeolites through ion exchange [17]. Zeolites can be utilised widely as heavy metal adsorbents. Alternative adsorbents of natural origin that are often used are rice husk [18], banana peel [19] and corn cob [20]. This study will use matoa fruit as a raw material for making zeolite. Matoa fruit has a high enough silica ash content so that it has the potential as a raw material for making zeolite [21]. Based on this background, research was conducted on the innovation and characterisation of zeolite from matoa fruit as an adsorber of heavy metal Cu which is expected to degrade environmental pollution.

2. RESEARCH METHODS

The materials used in this study, namely matoa fruit waste, aluminium hydroxide ($Al(OH)_3$), and sodium hydroxide (NaOH), have analytical quality with purity ≥99%. Distilled water is used as a solvent to ensure the absence of impurities that can affect the synthesis process.

2.1. Matoa Fruit Preparation

A total of 1 kg of matoa fruit was first cleaned and then dried under the sun and then heated in an oven at 105°C for 2 hours.

2.2. Activation of Matoa Fruit Ash

The prepared matoa fruit was activated in a furnace at 550°C for 4 hours. The resulting ash was sieved with a 100-mesh size. Next, 80 g of ash was weighed, activated with 250 mL of 2 M HCl, and soaked for 4 hours. Then, neutralisation was done with distilled water. The result was dried at 105°C for 8 hours and analysed by XRD.

2.3. Preparation of Sodium Silicate (Na_2SiO_2)

A total of 50 grams of activated matoa fruit ash was reacted with 300 mL of 6 M NaOH, then heated at 80°C for 2 hours. The solution was then filtered and sodium silicate was obtained.

2.4. Preparation of Sodium Aluminate ($NaAl_2O_3$)

NaOH powder weighing as much as 36 g was dissolved with 300 mL of distilled water, then heated, and 15 g of $Al(OH)_3$ gradually added until a sodium aluminate solution was formed.

2.5. Zeolite Preparation and Characterisation

50 mL of sodium silicate was reacted with 50 mL of sodium aluminate. Then, it was stirred for 4 hours to obtain a white gel, which was then heated in an autoclave at 120°C for 2 hours. The solid was filtered and reheated in the oven for 5 hours. The zeolite was characterized using XRD (X-Ray Diffraction) and SEM (Scanning Electron Microscopy).

2.6. Zeolite Adsorption Test on Cu Metal

A 20 ppm Cu solution was made and tested by zeolite adsorption powder to Cu metal by putting 0.5 g of zeolite into 50 mL of 20 ppm Cu solution, stirring with a magnetic stirrer for 40 minutes. The solution was then decanted, and the filtrate was analysed for concentration by AAS (Atomic Absorption Spectrophotometer).

3. RESULTS AND DISCUSSION

The removal of Cu metal in the waste can be done through the adsorption method using adsorbent from the matoa fruit zeolite. This can be done because the analysis in this study shows that the zeolite structure has a free electron pair content on the O atom that can bind heavy metals [22].

The adsorption mechanism involves binding Cu metal ions from the waste to the surface of the adsorbent as shown in Figure 1 [23]. The principle of metal adsorption on matoa fruit zeolite is the formation of coordination covalent bonds, where oxygen groups (-O-) on zeolites that have free electron pairs bind metal ions from the wastewater mixture. Metals in electrolyte solutions are positively charged particles, while the oxygen atom group is an anionic polyelectrolyte that binds positively charged atoms.

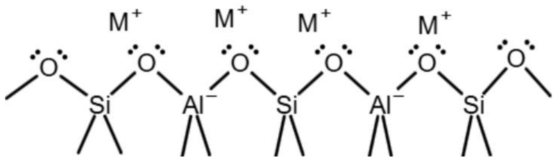


Figure 1. Adsorption of metal ions on zeolite.

FTIR analysis and characterization were conducted to determine the functional groups of matoa fruit zeolite from the synthesis process. The results of FTIR analysis are presented in the form of spectra in Figure 2. IR spectra of matoa fruit zeolite show the presence of several functional groups similar to the IR spectra of commercial zeolites, such as in the fingerprint region >1000 cm^{-1}. Matoa fruit zeolite has an abundance of Al-Si groups more than commercial zeolites. The identified functional group data can be seen in Table 1.

The peak at wave number 2836 cm^{-1} is the result of stretching vibrations of the OH group, indicating the presence of water content in the sample, and is reinforced by peaks at 1642 and 1533 cm^{-1} which appear due to bending vibrations of the -OH group [20]. Peak at 578 cm^{-1} indicates the presence of Si-O-Si or Si-O-Al asymmetry which are alumina adsorption bands.

Table 1. Result of FTIR analysis of matoa fruit zeolite.

Wavelength (cm^{-1})	Functional Groups
2836	OH
1642	OH
578	Si-O, Al-O

The synthesised zeolite was characterized using XRD to analyse its crystal structure. XRD analysis offers insights into the crystal microstructure, including phase purity and changes in lattice structure based on the 2θ angle. As shown in Figure 3, amorphous and aluminosilicate phases were also detected in addition to the crystalline phase. The diffractograms show that both materials are mainly composed of sodium silicate and sodium alumina silicate, as evidenced by the matching of diffraction peak patterns.

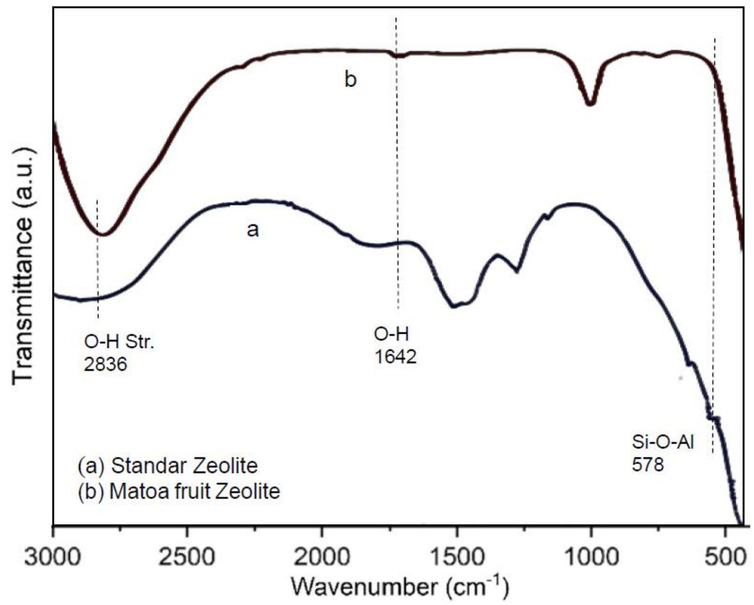


Figure 2. IR spectra of a) standard zeolite and b) matoa fruit zeolite.

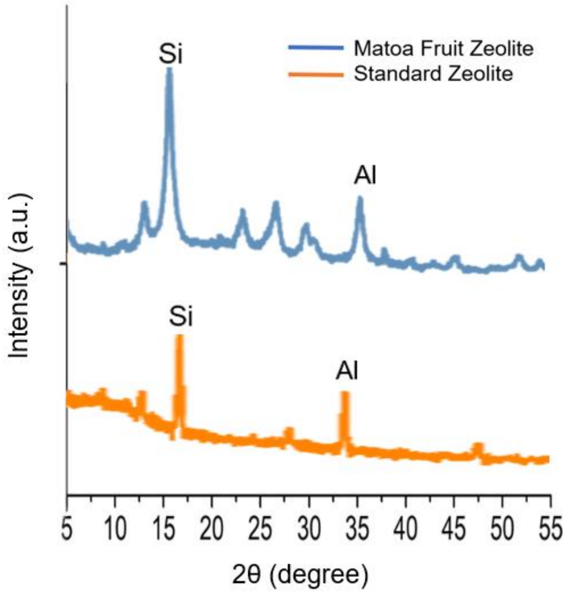
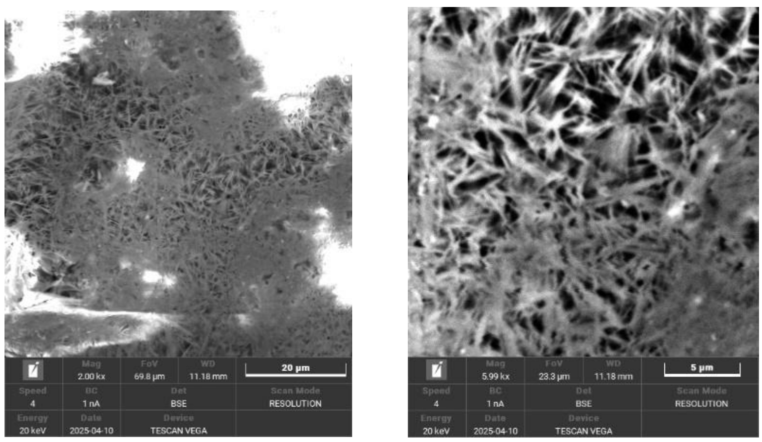


Figure 3. XRD test results of matoa fruit zeolite and standard zeolite.

This study analysed the obtained zeolites by comparing the characteristic 2θ peaks and d-spacing values with the collections data from powder XRD simulations. Matoa fruit zeolite shows adsorption bands at wave numbers around 11, 16, 26, 33° in Figure 3. The adsorption band at 33° indicates the presence of Al in $Al[(OH)]_6$. While the adsorption bands at 16° shows the presence of Si in SiO_4 bonds. The adsorption bands of matoa fruit zeolite and commercial zeolite show accurate patterns. This result shows that zeolite can be synthesised from matoa fruit.

In addition to XRD analysis, Scanning Electron Microscopy (SEM) was performed to examine the morphology of the synthesised matoa fruit zeolite, including its shape, texture, and microstructure, as shown in Figure 4. Based on SEM analysis, the morphology of the synthesised zeolite tends towards a cubic shape, which is characteristic of matoa fruit zeolite. However, it should be noted that the cubic morphology was not perfectly defined, as some irregularities were observed in the crystal structure.



a b

Figure 4. SEM test results of matoa fruit zeolite at zoom of (a) 6000x and (b) 2000x.

In the adsorption capacity test, matoa fruit zeolite was dissolved in a 20 ppm Cu solution. Table 2 shows the results of the adsorption ability test, which shows that the best treatment is achieved by treating 0.1 grams, resulting in 88.85% adsorption.

Table 2. Adsorption test results of matoa fruit zeolite against Cu metal.

No	Zeolite (gram per 50 mL)	Adsorption Ability (%)
1	0.1	88.85
2	0.2	88.71
3	0.7	88.25
4	1.0	87.97

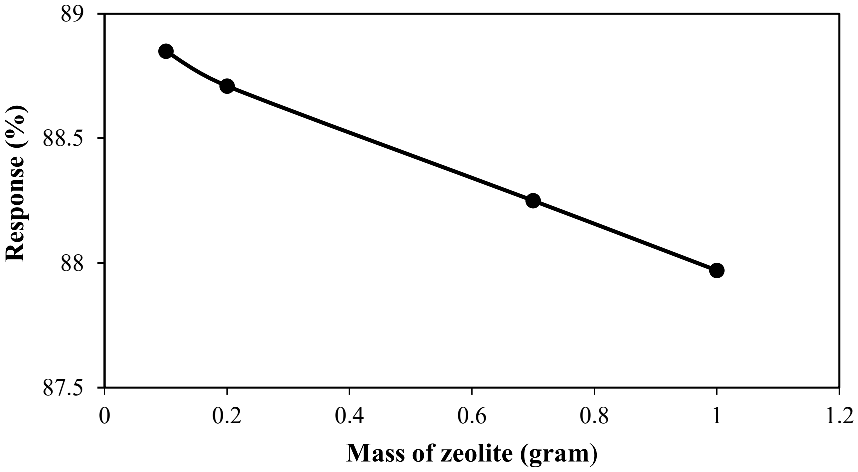


Figure 5. Adsorption power of matoa fruit zeolite on heavy metal Cu(II).

Figure 5 shows that the more matoa fruit zeolite used, the percentage of adsorption will decrease. In contrast to the natural zeolite used, where the more concentration of natural zeolite used, the percentage of adsorption of Cu metal increase from 0.5 gram to 1.0 gram. The highest adsorption power in matoa fruit zeolite was 88.85% at the addition of 0.1 gram.

The addition of zeolite concentration will affect the adsorption power of heavy metals [24]. Meanwhile, matoa fruit zeolite does not affect adsorption power. This is because matoa fruit zeolite has been able to provide sufficient surface area for the interaction between the adsorbent and Cu metal closely and the availability of active groups of matoa fruit zeolite in adsorbing heavy metal Cu has been maximised. The active group of matoa fruit zeolite in adsorbing heavy metal Cu is optimum at 0.1 gram per 50 ml of test solution.

The research by Mohamad, et al. concluded that this decrease in adsorption power can occur due to changes in the structure of the adsorbent material or adsorbent [25]. In the study, a decrease in adsorption power was observed due to the chemical structure of matoa fruit zeolite. This change weakened the bonds between the active groups of matoa fruit zeolite and copper (Cu) metal ions, making it easier for these bonds to break. The process of copper metal adsorption into matoa fruit zeolite occurs through the hydroxyl groups (-OH) on its surface. The adsorbed heavy metals eventually reach a specific concentration, at which point saturation occurs, leading to desorption. The binding mechanism of Cu metal ions by matoa fruit zeolite is due to the presence of active sites, namely hydroxyl groups on matoa fruit zeolite, so that it can adsorb heavy metal ion cations. The interaction between metal ions and matoa fruit zeolite occurs through the formation of *chelates* or coordination bonds by Cu metal ions with OH groups. This active site on zeolite surface is called a selective metal-binding ligand.

4. CONCLUSION

From this research, it can be concluded that matoa fruit waste can be processed into zeolite. Matoa fruit zeolite can reduce Cu metal levels in waste-polluted waters. The best dose that can reduce Cu metal for matoa fruit zeolite is 0.1 g in 50 ml of test solution with an adsorption ability of 88.85%.

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