

PHYSICAL, MECHANICAL AND MICROSTRUCTURE PROPERTIES OF METAKAOLIN BASED POROUS ALKALI ACTIVATED MATERIALS AS AN ADSORBENT FOR CU(II) ION REMOVAL

This study investigates the physical, mechanical, and microstructure properties of metakaolin-based porous alkali activated materials (AAM) at different surfactant concentrations as an adsorbent for Cu(II) ion removal. The AAM were synthesized using metakaolin as a precursor and alkali activators. Different surfactant contents ranging from 1 wt.% to 5 wt.% were incorporated into the AAM to enhance their adsorption capacity. The physical properties of the AAM were evaluated by determining their porosity, water absorption and density. The results showed that adding surfactant increased the AAM porosity, leading to higher water absorption. The highest porosity was observed at 3 wt.% surfactant content, indicating the optimal surfactant concentration for promoting a porous structure. The mechanical properties of the AAM were assessed through compressive strength tests. The microstructure analysis using scanning electron microscopy revealed that the AAM exhibited different microstructures depending on the surfactant content. The adsorption capacity and removal efficiency of the AAM for Cu(II) ion removal were determined. With a maximum adsorption capacity of 64.78 mg/g, the porous AAM could adsorb a significant quantity of Cu(II) ions. The outstanding removal efficiency of 97.17% showed the AAM maximum efficiency in removing Cu(II) ions from the solution at an adsorption condition of 25°C, pH 5, an initial concentration at 100 ppm and 60 minutes contact time.

Keyword: Alkali activated material; wastewater treatment; surfactants; copper ion removal; properties

1. Introduction

The contamination of water sources with heavy metal ions poses significant threats to human health and the environment. Among these pollutants, copper ions, Cu (II) are particularly concerning due to their widespread presence and potential adverse effects [1-5]. Developing efficient adsorbents capable of selectively removing Cu(II) ions from aqueous solutions has garnered considerable attention in water treatment.

In recent years, metakaolin-based porous alkali-activated materials (AAM) have emerged as promising candidates for various applications, including heavy metal ion removal. Metakaolin, obtained from the calcination of kaolin clay, exhibits excellent reactivity and pozzolanic properties, making it an attractive precursor for AAM synthesis [6,7]. Incorporating AAM as adsorbent offers several advantages, such as high surface area, adjustable porosity, and chemical stability [8-10].

Tween 80, known as polysorbate 80, is a commonly used surfactant in various industries and applications. It is a non-ionic

surfactant belonging to the polyoxyethylene sorbitan esters group. Tween 80 is a versatile and widely used surfactant due to its unique properties and characteristics. Tween 80 has excellent emulsifying properties, allowing it to disperse and stabilize immiscible substances like oil and water [11]. It helps form and stabilize emulsions, which can be useful in various applications. Besides, tween 80 can also act as a foaming agent, aiding in creating stable foam structures. It helps generate and stabilize bubbles, creating a porous structure in materials like the metakaolin-based alkali-activated materials in this study [12]. The incorporation of surfactants as pore stabilizers in AAM reduces the need for chemical additives, improves the effectiveness of copper ion adsorption, promotes cost-effective water treatment methods, and ultimately leads to a cleaner environment.

Therefore, this study aims to comprehensively investigate the impact of different surfactant content on the physical, mechanical, and microstructural properties of metakaolin-based porous AAMs, specifically focusing on their performance as adsorbents for Cu (II) ion removal. We can elucidate the correla-

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tion between the surfactant content, AAM characteristics, and adsorption efficiency by systematically varying the surfactant concentration. The investigation into the physical, mechanical, and microstructural properties of metakaolin-based porous materials adds valuable insights into the material science domain. This knowledge can be applied not only to water treatment but also to various construction and engineering applications, where understanding the properties of novel materials is crucial for innovation and improved performance.

The findings of this study are expected to contribute significantly to understanding how surfactant content influences the physical, mechanical, and microstructural properties of metakaolin-based porous AAM. Moreover, insights gained from the correlation between surfactant content and Cu(II) ion adsorption performance will guide the development of tailored AAM for efficient water treatment applications. Ultimately, this research endeavors to pave the way for sustainable and effective solutions in the remediation of heavy metal-contaminated water sources.

2. Experiment

2.1. Materials

The metakaolin was prepared by subjecting kaolin clay purchased from Kaolin (Malaysia) Sdn. Bhd., Bidor, Perak, Malaysia, to calcination at 850°C for 2 hours at a 5°C/min heating rate to achieve the desired reactive metakaolin. The metakaolin with a major component of ($\text{SiO}_2 = 56.84$, $\text{Al}_2\text{O}_3 = 35.60$, $\text{TiO}_2 = 3.13$, $\text{Fe}_2\text{O}_3 = 2.09$) was confirmed with chemical composition, XRF. The sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were mixed to produce the alkali activator until a homogeneous solution was obtained. The 10M sodium hydroxide solution was prepared by dissolving a 99% pure NaOH pellet weighing 400 g from Brenntag Sdn. Bhd. in Shah Alam, Selangor, Malaysia, was combined with the sodium silicate obtained from South Pacific Chemical Industries Sdn. Bhd. (SCPI) in Malaysia at composition of 30.1% SiO_2 , 9.4% Na_2O , and 60.5% H_2O (with a $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of 3.20). The mixture was carefully stirred or agitated to ensure the complete integration of the components, resulting in a homogeneous alkali activator solution. This step was crucial to achieve a consistent composition and concentration of the activator, which played a vital role in the subsequent synthesis of metakaolin-based porous alkali-activated materials (AAM). To enhance the porous structure of the adsorbents, a solution of hydrogen peroxide was prepared and used as a foaming agent. The 3 wt.% hydrogen peroxide was diluted by a 30 wt.% H_2O_2 solution from Sigma Aldrich in Malaysia. A surfactant known as Tween 80 or polysorbate 80 was incorporated into the mixture. This surfactant, obtained from Sigma Aldrich in Malaysia, consists of approximately 70% oleic acid, with the remaining content primarily composed of linoleic, palmitic, and stearic acids. $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ were used to prepare Cu(II) ions for the adsorption process.

2.2. Preparation of Metakaolin based Alkali Activated Materials as an Adsorbent

A predetermined amount of metakaolin is mixed with an alkaline activator (a combination of NaOH and sodium silicate (Na_2SiO_3)) at 0.50 alkaline activator (AA), $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio and 0.8 MK/AA ratio. It was selected following a previous study [13]. The metakaolin and alkaline activator mixture is thoroughly mixed using a mechanical stirrer and homogenized to ensure uniform distribution of the activator within the metakaolin matrix. This step promotes the activation and formation of the desired AAM structure. To enhance the porous structure of the adsorbents, a hydrogen peroxide, H_2O_2 at 1.25 wt.% by mass of solid, was added as a foaming agent and a surfactant known as Tween 80 or polysorbate 80 was incorporated into the mixture. Including the surfactant as stabilizing agent in the mixture serves an important purpose. It reduces the surface tension and drainage of the alkali-activated materials, a common function of surfactants in such applications. In this study, the surfactant content was varied at different weight percentages: 1 wt.%-5 wt.% relative to the mass of metakaolin. The metakaolin based alkali activated materials foams were shaped into 1-2 cm sphere-shaped and subjected to a curing process at 60°C for 24 hours. Curing allows for the hardening and developing the desired properties in the AAM. After the curing period, the AAM samples are dried to remove any remaining moisture. This step helps enhance the mechanical properties and stability of the AAM. The experimental technique for producing an ideal metakaolin based AAM in a simplified form in Fig. 1.

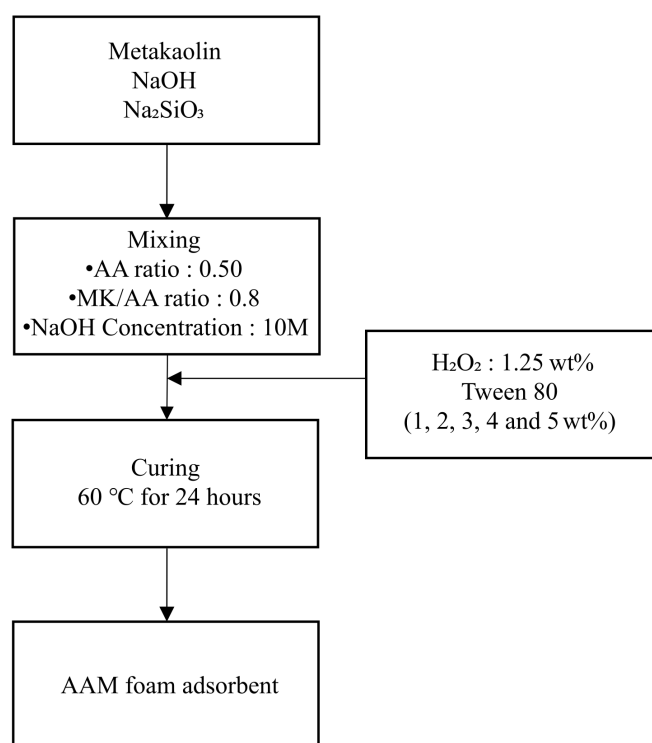


Fig. 1. Schematic experiment to optimize metakaolin based AAM

2.3. Test and Analysis Method

To achieve this objective, a series of experiments will synthesize metakaolin-based AAM samples with varying surfactant concentrations. The physical and mechanical properties, including density, water absorption and porosity, were evaluated to assess the structural changes induced by the surfactant. The porosity was determined using Eq. (1) and following ASTM C642 [14].

$$\begin{aligned} \text{Porosity (\%)} &= \\ &= \frac{\text{Wet weight (g)} - \text{Dry weight (g)}}{\text{Dry weight (g)} - \text{Suspended weight (g)}} \times 100 \quad (1) \end{aligned}$$

The electronic densitometer MD-3005 was used to calculate the metakaolin based AAM density following ASTM D792 [15]. While the water absorption tests conducted in this study followed the guidelines outlined in the ASTM D570 standard. Before the water absorption tests, the samples were typically subjected to a pre-conditioning step. This involved drying the samples to a constant weight (dry weight) in an oven or at a specific temperature and humidity condition to remove any existing moisture and achieve a consistent starting condition. The pre-conditioned samples were immersed or placed in distilled water for 24 hours. After the immersion period, the samples were removed from the water and any excess surface water was gently removed using a damp cloth or paper towel. The samples were then weighed using a precise balance to determine their weight after water absorption (wet weight). The water absorption percentage was calculated using the Eq. (2).

$$\begin{aligned} \text{Water absorption (\%)} &= \\ &= \frac{\text{Wet weight (g)} - \text{Dry weight (g)}}{\text{Dry weight (g)}} \times 100 \quad (2) \end{aligned}$$

The compressive strength also provided insights into the stability and durability of the AAMs. The compressive strength of metakaolin-based alkali-activated materials (AAM) was assessed using a Universal Testing Machine (UTM) manufactured by Shimadzu Japan, model UH-1000 kN. The loading rate applied during the testing was set at 0.5 MPa/s. The compressive strength measurements were conducted on cubic specimens with dimensions of 50×50×50 mm³, following the guidelines specified in ASTM C109 [16]. The compressive strength tests were performed after 7 days of curing, by which time the AAMs had already developed their early strength. To ensure reliable results, three specimens were subjected to compressive strength testing, and the average value of the three samples was reported as the representative compressive strength of the metakaolin-based AAM.

Additionally, microstructural analysis using advanced techniques like scanning electron microscopy (SEM) using TESCAN VEGA's 4th generation Scanning Electron Microscope (SEM) (Brno, Czech Republic) with a tungsten filament electron source combines SEM imaging and energy dispersive X-ray

(SEM-EDX) were employed to examine the morphology and elemental composition of the AAM adsorbents.

2.4. Preparation of Copper Ions Solution

Cu(NO₃)₂·3H₂O was used to simulate the adsorption process to prepare Cu(II) ions. Cu(NO₃)₂·3H₂O, also known as copper(II) nitrate trihydrate, was selected as the source of Cu(II) ions due to its solubility in water and ability to provide a consistent and controllable concentration of Cu(II) ions. To prepare a solution with a concentration of 1000 ppm (parts per million) of copper (II) nitrate, 3.929 grams of copper (II) nitrate powder was accurately weighed using a balance. The weighed copper (II) nitrate powder was added to a volumetric flask containing 1 litre of distilled water. The solution was thoroughly mixed or stirred until the copper (II) nitrate powder was completely dissolved in the water. Once the copper (II) nitrate powder was fully dissolved, the volumetric flask was filled to the top with additional distilled water up to the 100 ml mark. This resulted in a 1000 ppm copper nitrate solution in the volumetric flask. To prepare a 100 ppm copper nitrate solution, distilled water was added to the volumetric flask until the upper line or mark on the flask was reached. The addition of distilled water further diluted the concentration of copper nitrate, resulting in a solution with a concentration of 100 ppm.

2.5. Preparation of Metakaolin based AAM Adsorbent

At first, the metakaolin-based alkali-activated materials (AAM) were crushed into a powder particle size and sieved to a particle size of 150 μm to be used as an adsorbent. By reducing the AAM to a fine powder and sieving it to a specific particle size, the surface area of the adsorbent is increased. Finer particles have a larger surface area per unit mass, which enhances the adsorption capacity of the material. This increased surface area provides more active sites for the adsorption of target ions or contaminants [17,18]. In addition, reducing particle size allows for better contact and interaction between the adsorbent particles and the target ions in the solution [19]. This promotes efficient adsorption kinetics and enhances the overall performance of the adsorbent material.

2.6. Copper Ion Adsorption Test

The metakaolin-based alkali-activated materials (AAM) adsorbent was washed with distilled water for 1 hour to avoid precipitation and high sodium hydroxide concentration. The adsorbent was dried under a vacuum at 105°C for 24 hours. The 0.15-gram dried samples were examined with a 100 mg/L copper nitrate solution at pH 5. This adsorption test was based on [20,21], which showed excellent copper adsorption capacity and

removal efficiency utilizing metakaolin geopolymer adsorbents. The dried samples were placed in Erlenmeyer flasks and shaken at 250 shakes/min for 1 hour in an orbital shaker at room temperature ($\sim 25^\circ\text{C}$) to start adsorption. After the specified time, flask samples were collected and analyzed for Cu^{2+} content using atomic absorption spectroscopy (AAS) using a Perkin Elmer device in Llantrisant, UK. The following Eq. (3) and (4) was used to compute the copper's adsorption capacity (q_e , mg/g), as well as its removal effectiveness ($R\%$), respectively.

Adsorption capacity,

$$q_e = \frac{(C_o - C_e)V}{W} \quad (3)$$

Removal efficiency,

$$R\% = \frac{(C_o - C_e)}{C_o} \times 100 \quad (4)$$

Where:

- C_o – Initial concentration of the copper solution (mg/L),
- C_e – Final concentration of the copper solution after adsorption (mg/L),
- W – The weight of the adsorbent usage (g),
- V – The volume of the Cu(II) solution (L).

3. Results and discussion

3.1. Porosity

Fig. 2 shows the porosity value of the alkali activated materials sample with different surfactants amount. The porosity of the AAM sample with 3 wt.% of surfactant was the highest, 83.23%, compared to the 40.03% with 1 wt.% of surfactant. This demonstrates a substantial increase in porosity compared to the samples with lower surfactant amounts, indicating that a higher surfactant content greatly enhanced the porosity of the AAM [12]. The findings suggest that adding a surfactant to metakaolin-based AAM increased porosity. As the surfactant content

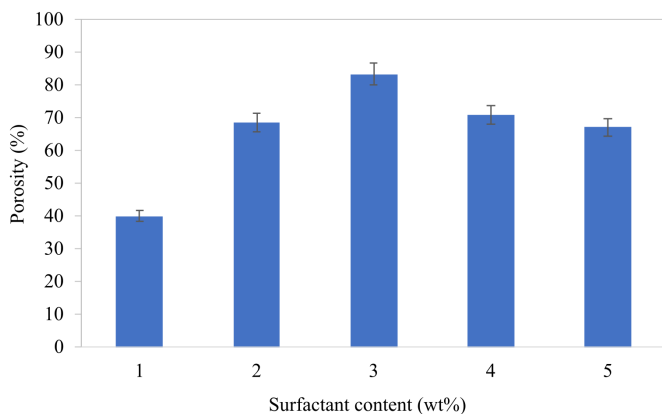


Fig. 2. The porosity value metakaolin based alkali activated materials sample with different surfactants amount

increased from 1 to 3 wt.%, porosity was substantially enhanced. However, beyond 3 wt.% of surfactants, the effect on porosity became less pronounced or slightly decreased. These findings highlight the influence of surfactant content on the porosity of the AAM, providing valuable insights for optimizing the material's porosity for specific adsorption applications.

3.2. Water Absorption

Fig. 3 shows the water absorption value of the metakaolin based alkali activated materials sample with different surfactant amounts. The AAM sample with 1 wt.% surfactant exhibited the lowest water absorption value of 10.50%. This indicates the amount of water the material absorbs relative to its initial weight. A lower water absorption value suggests that the material has relatively lower porosity and low capacity to absorb water. The AAM sample with 3 wt.% surfactants demonstrated a significant increase (highest) in water absorption, with a value of 19.87%. From the observation, higher surfactant up to 3 wt.% content leads to higher water absorption. When the surfactants are introduced into the AAM, they can interact with the material's surface, reducing surface tension and promoting better wetting by water [22]. This facilitates the penetration and absorption of water into the material. In addition, surfactants can act as foaming agents, promoting the formation of bubbles and increasing the pore volume and pore size distribution in the AAM structure [23]. As the surfactant content increases, more bubbles are formed, resulting in a higher porosity. This increased porosity provides more space for water to be absorbed and retained within the material.

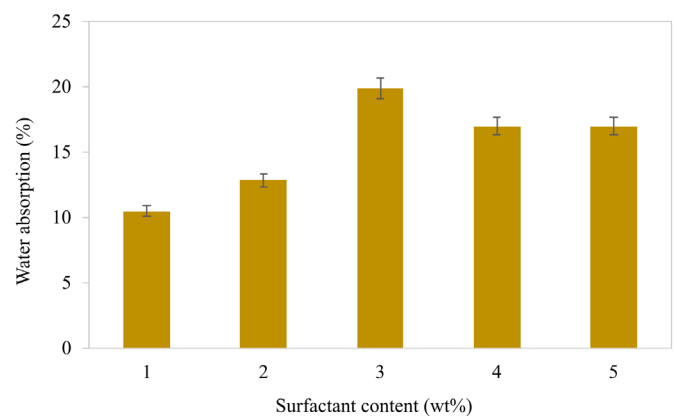


Fig. 3. The water absorption of metakaolin based alkali activated materials with different surfactant content

The results suggest that introducing surfactant to metakaolin-based AAM can impact its water absorption characteristics. As the amount of surfactant increased from 1 wt.% to 3 wt.%, a substantial enhancement in water absorption capacity was observed. However, beyond 3 wt.% surfactants, the impact on water absorption became less prominent, with only minor fluctuations noticed.

3.3. Density

Fig. 4 illustrates the density of the alkali activated material samples with varying surfactant amounts. The sample containing 1 wt.% of surfactant exhibited the highest density at 1.885 g/cm³ compared to the sample with 3 wt.% of surfactants, with a density of 1.153 g/cm³. This difference can be attributed to more pores in the sample with 3 wt.% of surfactants. The density continued to increase beyond 3 wt.% of surfactants, specifically at 4 wt.% to 5 wt.%, reaching values of 1.656 g/cm³ and 1.679 g/cm³, respectively.

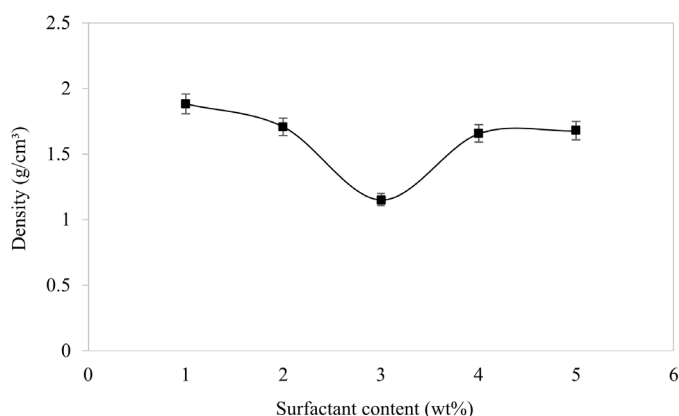


Fig. 4. The density of alkali activated materials at different amount of surfactant

Particularly, adding surfactant to the AAM samples resulted in forming a porous structure within the AAM, leading to a reduction in density. The lower density in the alkali activated material samples indicates a higher number of pores, which promotes a favorable environment for the adsorption mechanism. The alkali activation process contributes to forming an amorphous and porous structure, further reducing the material density [24,25]. However, the alkali activated material samples containing 4 wt.% and 5 wt.% of surfactant displayed increased density. The strong affinity of the surfactants for liquid-air interfaces within the alkali-activated material increased viscosity and facilitated pore formation. This stronger adsorption of surfactants on the metakaolin's surface led to improved dispersion of metakaolin particles and a denser microstructure, as determined by the molecular configurations of the surfactants.

3.4. Compressive Strength

Fig. 5 shows that the AAM samples' compressive strength exhibits a decreasing trend as the surfactant content increases from 1 wt.% to 3 wt.%. The sample with 1 wt.% of surfactant demonstrates the highest compressive strength of 22.045 MPa, while the sample with 3 wt.% of surfactant shows the lowest compressive strength of 16.218 MPa. Whereas the compressive strength slightly improves when the surfactant content is increased from 3 wt.% to 5 wt.%. The samples with 4 wt.% and

5 wt.% of surfactant exhibit 18.713 MPa and 18.9 MPa compressive strengths, respectively. These findings suggest that an increase in surfactant content beyond a certain point can reduce the compressive strength of the metakaolin-based AAM. However, a further increase in surfactant content does not significantly affect the compressive strength.

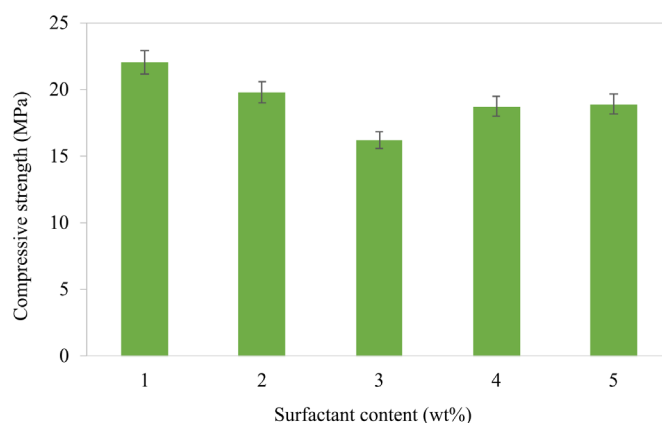


Fig. 5. The compressive strength of alkali activated materials at different amounts of surfactant

Therefore, a compressive strength of 16.218 MPa at 3 wt.% surfactant content may be considered acceptable as long as the material exhibits satisfactory adsorption performance and meets the required criteria for removing the target contaminants, such as high adsorption capacity and efficient removal of Cu(II) ions. The acceptable compressive strength at 3 wt.% surfactant content allows for a balance between adsorption properties and mechanical strength when using the metakaolin-based AAM as an adsorbent material.

3.5. Microstructural Analysis

The selected mixing design of the samples of metakaolin-based alkali activated materials appeared on the microstructural of the sample structure in Fig. 6. The microstructure of the alkali activated material at 1 wt.% surfactant is shown to be dense and coarse. On the other hand, at 3 wt.%, surfactant displays a porous microstructure, which is attributed to the increasing addition of Tween 80 surfactant. The presence of the surfactant promotes the formation of a high pore structure. However, at higher surfactant levels (>3.0 wt.%), some of the pores collapse or coalesce, leading to the formation of large interconnected pores. This observation suggests that excessive surfactant content can have a detrimental effect on the pore structure, resulting in the appearance of irregular and non-uniform pores [26].

As the surfactant content increases, the stability of the microstructure initially decreases, leading to a decrease in total porosity and average pore size. This can be attributed to the increase in viscosity of the slurry and the decrease in foamability as the surfactant amount increases [27,28]. These factors contribute to a more compact and less porous structure. Furthermore, higher

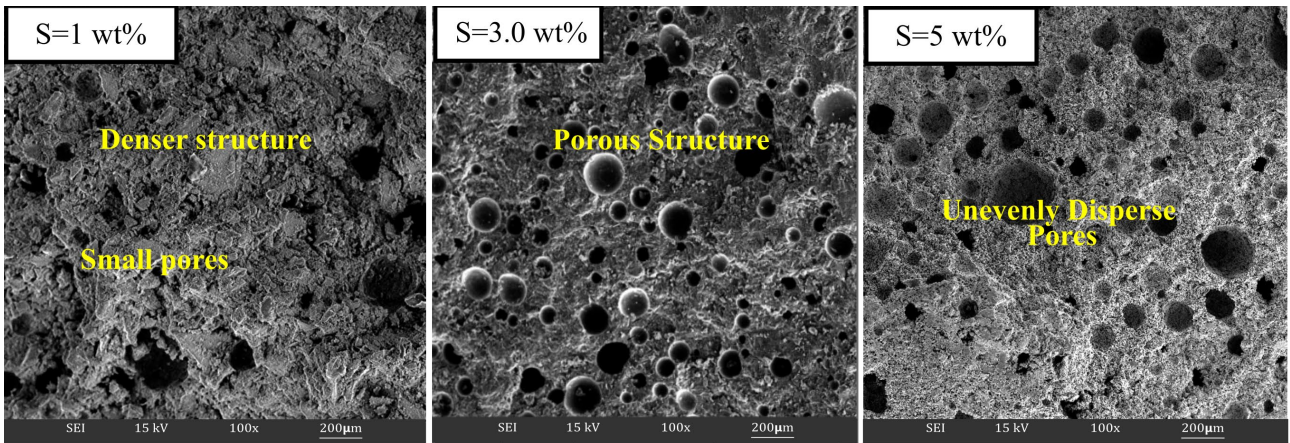


Fig. 6. Morphology analysis of the SEM on metakaolin based AAM

(at 5 wt.%) indicates that the pores are mostly closed and the larger cells are unevenly distributed. This observation aligns with the findings reported by Bai et al. [29]

3.6. Effect of Surfactant Content on Copper Ions, Cu(II) Adsorption

The adsorption capacity and removal efficiency of metakaolin-based alkali activated materials (AAM) were analyzed at different surfactant amounts, demonstrated in Fig. 7. At 1 wt.% surfactant, the adsorption capacity was measured to be 51.75 mg/g with 77.63% removal efficiency, indicating the ability of the AAM to adsorb a significant amount of Cu(II) ions from the solution. As the surfactant content increased to 2 wt.%, there was a noticeable improvement in both adsorption capacity and removal efficiency. The adsorption capacity increased to 62.51 mg/g, indicating an enhanced capability of the AAM to adsorb more Cu(II) ions. The removal efficiency also significantly increased to 93.76%, indicating a higher percentage of Cu(II) ions removed from the solution. Following 3 wt.% surfactants, the AAM exhibited the highest adsorption capacity of 64.78 mg/g, indicating its ability to adsorb a large amount of Cu(II)

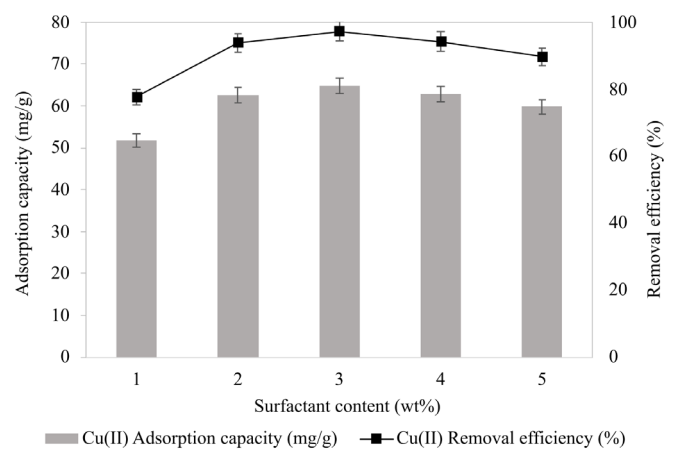


Fig. 7. The adsorption capacity and removal efficiency of metakaolin based AAM at different surfactant content

ions. The removal efficiency reached an impressive value of 97.17%, showing the AAM’s highest effectiveness in removing Cu(II) ions from the solution.

As can be seen in Fig. 8, it is clearly showing the presence of copper attached to the surface of the metakaolin-based alkali activated materials (AAM) adsorbent. This observation is further

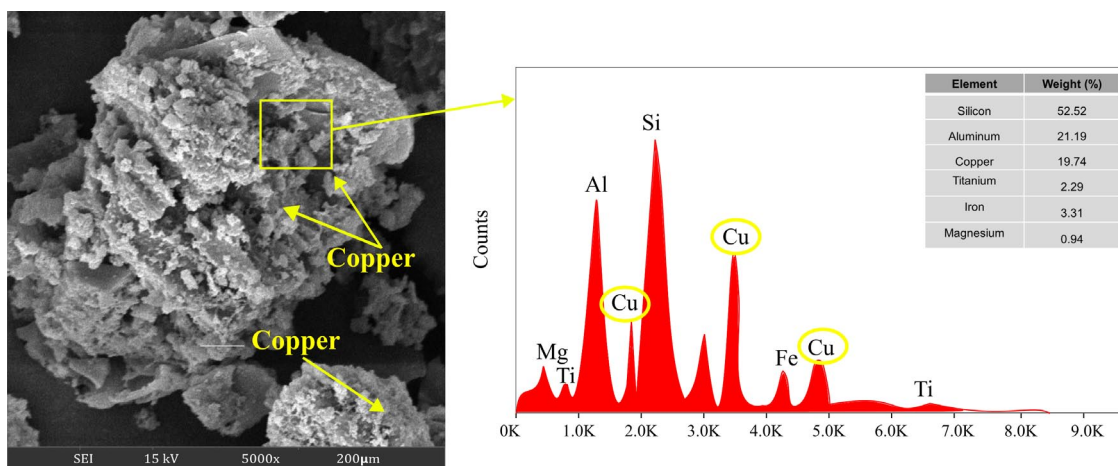


Fig. 8. Microstructure and elemental analysis of metakaolin AAM after copper ions adsorption

supported by the elemental analysis presented. The study reveals the presence of a strong copper peak with 19.74 weight % and the absence of significant peaks for silica and alumina in the elemental analysis provides strong evidence for the successful attachment of copper onto the metakaolin-based AAM adsorbent. This confirms the efficacy of the AAM adsorbent in selectively capturing and retaining copper ions from the solution.

With a further increase in surfactant content to 4 wt.% and 5 wt.%, the adsorption capacity and removal efficiency slightly decreased. However, the values remained relatively high, with adsorption capacities of 62.79 mg/g and 59.73 mg/g, and removal efficiencies of 94.18% and 89.60%, respectively. Overall, the findings demonstrate that the surfactant content influences the adsorption capacity and removal efficiency of Cu(II) ions by metakaolin-based AAM. Higher surfactant amounts generally improved adsorption capacity and removal efficiency up to a certain point. The optimal surfactant content for achieving the highest adsorption capacity and removal efficiency in this study was found to be at 3 wt.%.

4. Conclusions

This work examined the physical, mechanical, and microstructure characteristics of metakaolin-based porous alkali activated materials (AAM) at varied surfactant concentrations for Cu(II) ion removal. The experimental findings showed that surfactant content significantly affects AAM characteristics and adsorption. The addition of surfactants up to 3 wt.%, particularly Tween 80, led to forming a porous microstructure in the AAM, promoting the greatest pore structure (83.23% total porosity) and optimal water absorption capacity (19.87%). However, the effect on water absorption became less pronounced beyond a certain surfactant content (>3 wt.%). The compressive strength analysis revealed a decreasing trend in the strength of the AAM with increasing surfactant content. This can be attributed to the higher porosity and reduced density to 1.153 g/cm³, resulting from surfactants. However, it should be noted that the compressive strength values of the AAM (~16 MPa) remained within an acceptable range for their application as adsorbent materials. Copper ions, often originating from industrial processes, can have detrimental effects on aquatic ecosystems and human health. Overall, the findings highlight the importance of surfactant content in tailoring the physical, mechanical, and microstructural properties of metakaolin-based AAM for Cu(II) ion removal. This research contributes to the development of supplementary cementitious material, alternative to traditional cementitious materials aligns with the growing demand for sustainable and efficient methods for mitigating metal ion pollution in water bodies. By exploring its use in porous alkali-activated materials, this research aligns with the growing demand for sustainable construction materials, reducing the carbon footprint of the construction industry and promoting eco-friendly practices. Ultimately, the optimal surfactant content should be carefully selected to balance the desired properties and adsorption

performance of the AAM. Further recommendations could be conducting comparative studies to evaluate the performance of the porous metakaolin-based AAM adsorbents regarding adsorption capacity and removal efficiency under various adsorption conditions. Explore factors such as the initial concentration of Cu(II) ions, solution pH, contact time, and temperature. This will provide a comprehensive understanding of the adsorption behaviors and efficiency of the porous AAM adsorbents under different operating conditions, enabling the identification of optimal conditions for maximum adsorption capacity and removal efficiency. It is also exciting to investigate the regeneration and reusability of AAM after Cu(II) ion adsorption to develop environmentally friendly and sustainable adsorption techniques.

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