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# HYDROGEN PEROXIDE MODIFICATION ENHANCES THE ABILITY OF METAKAOLIN BASED ALKALI ACTIVATED MATERIALS ADSORBENT TO REMOVE THE $Ni^{2+}$ IONS

The presence of nickel ions in wastewater is a significant environmental concern due to its toxicity, which can cause severe health problems. Metakaolin is a pozzolanic material that can be activated by alkali to produce a highly porous and reactive material that can be utilised as a heavy metal ion adsorbent. However, the adsorption capacity of metakaolin-based adsorbents is limited by their surface chemistry and porosity. Metakaolin-based alkali-activated materials adsorbent modified with hydrogen peroxide can effectively remove nickel ions from wastewater. The modification process increases the surface area and porosity of the adsorbent, enhancing its adsorption capacity. The modified adsorbent (1.00 wt.% $H_2O_2$) showed a higher sorption capacity of 26.57 mg/g and efficiency of 85.22% compared to the non-modified adsorbent (10.55 mg/g) sorption capacity and 45.63% nickel removal efficiency, indicating the potential of hydrogen peroxide-modified adsorbents as an economical and ecologically sustainable solution for environmental applications, particularly for metal immobilization.

*Keywords:* Metakaolin; Alkali activated materials; Adsorbent; $Ni^{2+}$ removal; Heavy metals

## 1. Introduction

A significant and ongoing environmental issue is the occurrence of heavy metals in industrial wastewater because of their non-biodegradability and toxicity [1,2]. One of the most prevalent heavy metals in industrial wastewater is nickel, which harms people and plants. Nickel is widely used in various industrial operations, such as electroplating, paint, textile production, paper and battery manufacture. In addition, mining and metallurgical operations are also significant sources of nickel contamination due to their extensive utilization. Since this type of heavy metal is poisonous and carcinogenic, its removal from wastewater is thus of utmost significance [3,4]. For the removal of heavy metals, several techniques have been used, including chemical precipitation, membrane filtration, coagulation, ion exchange, and adsorption [5,6]. Adsorption is one of the approaches for removing heavy metals from water effluents among the many treatment procedures because of its ease of use, efficiency, and affordability [7-10]. The adsorbents employed for the adsorption of heavy metals might be of mineral origins, such as natural zeolite, calcium silicate powders, and natural clay [11-14]. Common adsorbents, including activated carbon, alumina and silica, have high levels of efficiency, but their high manufacturing costs limit their use [15-17]. It is increasingly important to create eco-friendly and affordable technology to protect the environment and promote sustainability. As a result, efforts are being made to create affordable, alternative metakaolin-based alkali activated materials adsorbents.

Alternative low-$CO_2$ binders to Portland cement, such as alkali-activation (or geopolymer) technology, are a subset of this technology and have received a great deal of interest in the recent decade for their potential in water and wastewater treatment [18-21]. Many inorganic wastes may be converted into useful products using alkali-activation technology. This is essential for furthering the circular economy [22-24]. By treating aluminosilicate precursors with concentrated alkali hydroxide and silicate solutions at (near) ambient conditions, alkali-activated materials (AAMs) may be made using a comparatively easy and less energy-intensive preparation approach [18,25]. The negatively charged aluminosilicate framework that makes up the chemical structure of AAMs allows charge-balancing cations to exchange with cations in the solution, which raises the prospect of employing them as adsorbents [26,27]

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Adding hydrogen peroxide, which is well known to break down quickly in alkaline solution, is one of the preliminary requirements for synthesizing alkali activated materials adsorbents [28,29]. The AAMs structure might capture the oxygen produced, enhancing the material's porosity characteristics and, therefore, its adsorption performance while maintaining its surface chemistry [30-33]. The final products often feature a porous structure, great physical and chemical stability, an amorphous structure, and ion exchange capacities [34-36]. Some of the precursors are industrial waste products or other easily accessible, low-cost resources, which further improve the practicality from an economic and environmental standpoint.

The role of hydrogen peroxide in declaring alkali-activated materials better at absorbing or worse at absorbing cannot be measured or inferred. In particular, for alkali activated materials based on pozzolan, it has not yet been described how hydrogen peroxide affects the textural and adsorption properties of these materials. This research aimed to develop a hydrogen peroxide-modified pozzolan-based alkali activated material as an eco-adsorbent to improve the adsorption properties of metakaolin-based alkali activated materials. The impacts of hydrogen peroxide incorporation on the textural, structural, and adsorptive performance for nickel ion removal from aqueous solution were investigated and reported herein.

## 2. Experimental method

### 2.1. Materials

In this study, kaolin from Kaolin Sdn. Bhd. Tapah, Perak, Malaysia, served as aluminosilicate materials that were first calcined to create metakaolin. Metakaolin was used in this experiment, which was confirmed by X-ray fluorescence (XRF) elemental analysis, with the main silica oxide ($SiO_2$) content of 56.84% and aluminium oxide ($Al_2O_3$) composition of 35.60%. Technical-grade sodium hydroxide (NaOH) and sodium silicate solution ($Na_2SiO_3$) were utilized as an alkaline activator. Hydrogen peroxide, $H_2O_2$, and Tween 80 surfactant were introduced as colourless foaming agents. Nickel ions stock was dissolved with the required weight of $Ni(NO_3)2.6H_2O$ in distilled water to create solutions containing nickel ions.

### 2.2. Geopolymer synthesis and analysis

The metakaolin as resource material was mixed with 8M sodium hydroxide solution and sodium silicate solution to create paste specimens at metakaolin/alkaline activator ratio 0.8 by mass of solid. The mass ratio of $Na_2SiO_3$/NaOH was kept constant at 0.5 in this experiment. With a constant of 3 wt.% of Tween 80, the hydrogen peroxide, $H_2O_2$, was changed from 0.50 wt.%, 0.75 wt.%, and 1.00 wt.% by mass of metakaolin. For comparison, a control sample was made without using a foaming agent. Alkali activated materials paste began to take shape after adding all the ingredients. The resultant slurry was then shaped into the appropriate shape, which measured around 1 cm (formed into a small round shape). The sample was put in a steel tray hermetically sealed to prevent moisture from evaporating and placed in an oven to cure at 60°C for 24 hours. The samples of metakaolin-based alkali-activated materials were taken out of the oven after 24 hours and left to rest for 7 days before testing. The alkali-activated materials were processed into a powder form suitable for sorption operations by crushing and sieving through a 150 μm particle size sieve. The resulting powder was washed with acetone at least three times to eliminate any excess alkaline activator solution. Using the Micrometrics Tristar II 3020 volumetric adsorption/desorption device, the Brunauer, Emmet, and Teller (BET) techniques were used to calculate the specific surface area and pore volume. SEM was used to characterize the structural characteristics of a sample of metakaolin alkali activated materials with varying hydrogen peroxide contents (SEM).

### 2.3. Adsorption test

For the adsorption test, 0.1 L of metal solution and 0.15 g of adsorbent were agitated to study the sorption of nickel ions on metakaolin-based alkali activated materials. The adsorption test was carried out at room temperature with the initial concentration of 100 mg/L fixed at pH 7. The pH values of the metal ion solutions were fixed by adjusting them with negligible quantities of 0.01M HCl and 0.01M NaOH. The orbital shaker was used with the flasks for 50 minutes at 150 rpm. Atomic absorption spectroscopy, or AAS, was used to analyze samples for the presence of $Ni^{2+}$ and to determine the concentration of nickel ions in the filtered sample. The experiment was carried out three times for each condition to get the mean results. Eqs. (1) and (2) were used to determine the nickel removal efficiency, $R$, and the quantity of nickel adsorption on the metakaolin-based alkali activated materials sample, $q_e$.

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{1}$$

$$R = \frac{(C_0 - C_e)}{C_0} \times 100\% \tag{2}$$

Where $C_0$ and $C_e$ are the initial and equilibrium concentrations (mg/L), respectively, of nickel ions in solution, $V$ is the volume (L) and $m$ is the mass (g) of the adsorbent.

## 3. Results and discussions

### 3.1. Surface area and total pore volume analysis

TABLE 1 shows the BET surface areas and pore volumes of metakaolin-based AAMs adsorbents at various hydrogen peroxide concentrations determined from Nitrogen, $N_2$ adsorption.

Unfoamed metakaolin-based alkali activated materials have the lowest surface area and pore volume, measuring 7.1216 $m^2/g$ and 0.028246 $cm^3/g$, respectively. The surface area and pore volume of the hydrogen peroxide added to metakaolin-based alkali activated materials are larger than those of the control sample, maximum of 1.0 wt.% $H_2O_2$ concentration with 28.6057 $m^2/g$ of surface area and 0.194348 $cm^3/g$ of the pore volume. According to Mondal et al., 2021 [37] and Suresh Kumar et al., 2019 [38], increasing pore volume and surface area increased the number of active sorption sites. Thus, more active functional groups are exposed, which may increase the ability of nickel ions to bind to surfaces and remove them more effectively.

TABLE 1

Textural properties of metakaolin based alkali activated materials adsorbent

| Samples | Specific Surface Area, $S_{BET}$ ($m^2/g$) | Pore Volume, V ($cm^3/g$) |
|---|---|---|
| Control sample | 7.1216 | 0.028246 |
| 0.50 wt.% | 10.8483 | 0.100423 |
| 0.75 wt.% | 24.6076 | 0.171588 |
| 1.00 wt.% | 28.6057 | 0.194348 |

### 3.2. Microstructural analysis

Using a scanning electron microscope, (SEM) the surface of the inner sample structure was examined. AAMs samples of the control sample, 0.5 wt.%, and 1.0 wt.% hydrogen peroxide contents were chosen for analysis and study.

The quantity of hydrogen peroxide impacts the microstructure of geopolymers made from metakaolin (Fig. 1). Surfactant is added to the fixed volume as a stabilizing agent, which helps govern the pore-dispersion of metakaolin-based alkali activated materials [39]. The surfactant also stopped pore coalescence from becoming too extreme. This is shown by the little variations in the average pore diameter (Fig 1). The metakaolin-based AAMs control sample had the densest AAMs matrix since it lacked a foaming component. Adding hydrogen peroxide to samples of AAMs may modify the reaction kinetics and microstructure of the material [40]. This can occur when hydrogen peroxide breaks into water and oxygen gas, creating porosity in the AAMs sample. Thus, hydrogen peroxide can enhance the sample's surface area and porosity, boosting its ability to adsorb or remove heavy metals such as nickel ions.

The metakaolin-based AAMs displayed a loose and porous structure that diffused evenly throughout the matrix by adding hydrogen peroxide. The addition of $H_2O_2$ increased pore volume and average pore size. Hajimohammadi et al., 2017 [41] and Korat & Ducman, 2017 [42] supported the idea that the geometric form of pores changes when $H_2O_2$ concentration rises. All small diameter pores were spherical and homogeneous in structure at low $H_2O_2$ (0.5 wt.%). High $H_2O_2$ concentrations up to 1.00 wt.% enhanced the likelihood of larger holes forming. Pore walls in these samples were very thin due to the substantial amount of foaming, which allowed certain pores to merge and create these enormous voids.

### 3.3. Adsorption capacity and removal efficiency analysis

Figs. 2 and 3 depict the adsorption capacity and the nickel ions removal efficiency from the outcomes of an adsorption test using foaming agents in various concentrations on samples of metakaolin-based alkali activated materials, respectively. The pattern demonstrated that the metakaolin-based AAMs samples adsorption capacity and removal effectiveness increase linearly with an increase in hydrogen peroxide concentration.

At 1.0 wt.% addition of hydrogen peroxide, the maximum removal efficiency of 85.22% was attained, leading to the adsorption of 26.57 mg/g of nickel ions. By contrast, nickel uptake and elimination efficiency were the lowest in the control sample (at 20.55 mg/g and 65.92%, respectively) due to the lack of a porous structure. Increasing the concentration of hydrogen peroxide led to higher $Ni^{2+}$ adsorption and removal efficiency,

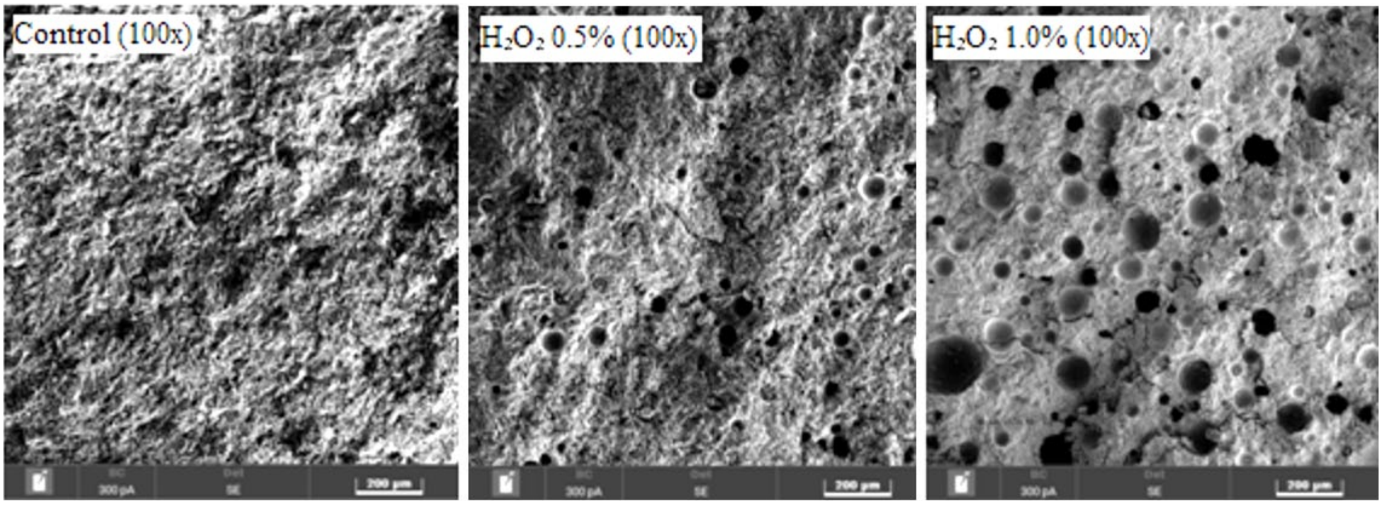

Fig. 1. SEM micrograph of metakaolin based alkali activated materials with different hydrogen peroxide content

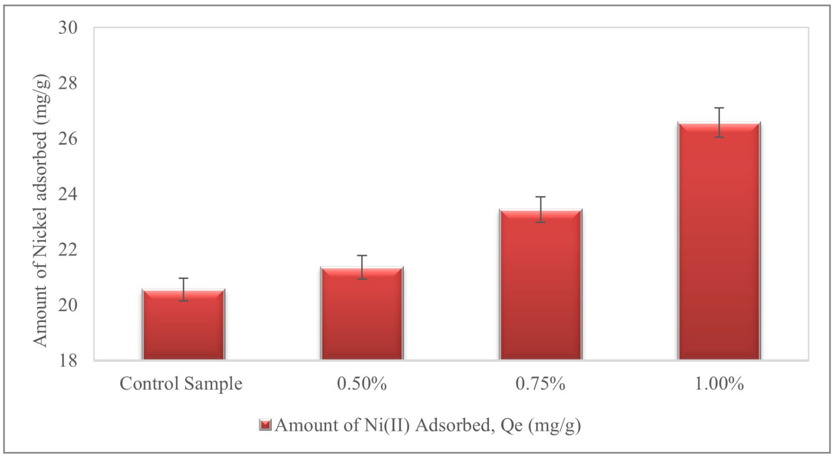

Fig. 2. Effect on hydrogen peroxide content on Ni(II) Adsorption quantity

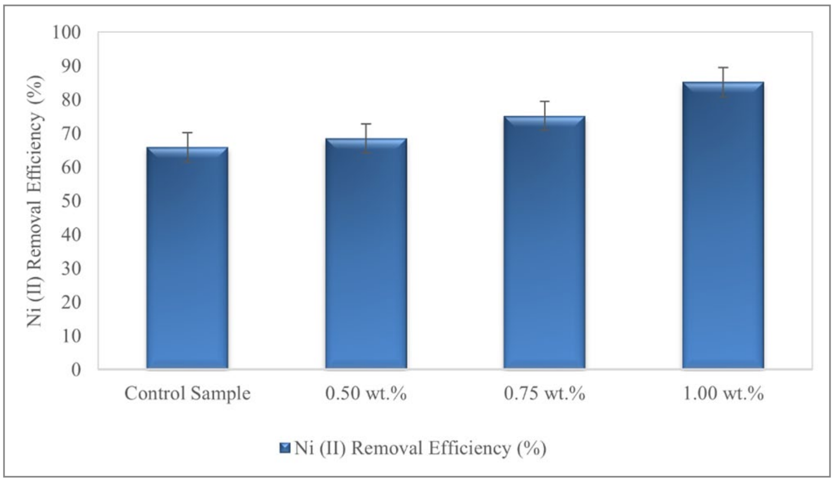

Fig. 3. Effect on hydrogen peroxide content on Ni(II) Removal Efficiency

suggesting that the alkali-activated materials' adsorption capacity was improved by adding hydrogen peroxide. Adsorbent materials with a greater number of pores or higher porosity offer a larger surface area for the adsorption of heavy metal ions, resulting in increased adsorption capacity due to more active adsorption sites [43,44]. However, excessive use of foaming agents (>1.0 wt.%) can create too large, uneven, or irregular pores, which can impair mechanical strength and reduce its durability. This is because the viscosity of the mixture rises when the foaming agent is added, resulting in fewer bubbles bursting, which does not encourage the pore growth of alkali-activated materials that function as adsorbents [45-47].

## 4. Conclusions

The study investigated the effect of hydrogen peroxide modification on removing nickel ions using metakaolin-based alkali-activated materials as adsorbents. The results showed that adding hydrogen peroxide enhanced the adsorption capacity and efficiency of the adsorbent, leading to improved nickel ion removal. At a temperature of 60°C, which is lower than the sintering temperature of typical zeolite or membranes used for heavy metals removal, the metakaolin-based AAMs adsorbent was created utilizing a simple method. The metakaolin-based AAMs adsorbent has a highly developed porous structure and larger surface area, facilitating nickel ion adsorption. Adding $H_2O_2$ is anticipated to enhance the efficiency of metakaolin-based AAMs adsorbent in removing nickel ions, as the control sample's surface chemistry and porosity may limit its effectiveness. The best conditions for $Ni^{2+}$ elimination (85.22%) and adsorption capacity values of 26.57 mg/g were achieved with 1.0 wt.% hydrogen peroxide added at pH 7, 25°C adsorption condition, and a 50-minute contacting period. The study concluded that modifying metakaolin-based alkali-activated materials adsorbent with hydrogen peroxide can enhance its ability to remove nickel ions.

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