KAOLIN-BASED GEOPOLYMER AS A HEAVY METAL REMOVAL: SHORT REVIEW

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Abstract

Adsorption procedure have been studies and found to be an effective, easy, and low-cost approach for removing heavy metals from aqueous solution when compared to other methods and technologies. Porous geopolymer will be made by combining aluminosilicate mineral with an alkaline activation solution to be utilized as an adsorbent. This review focuses on the current development in heavy metal removal material. This work also summarize the crucial factors (such as solid-to-liquid ratio, foaming agent ratio, sintering temperatures, and adsorbent dosage) influenced the properties (pore formation, microstructure, chemical bonding, and phase analysis) of kaolin-based geopolymer were highlighted. This review manifests the remarkable potential of kaolin-based geopolymer in high-added value applications.

Keywords*: kaolin, geopolymer, adsorbent material, heavy metal removal.*

Introduction

Heavy metal refers to a metallic element with a high density. Heavy metals with atomic weight ranging from 63.54 to 200.59 have a specific gravity greater than 4 [1]. Heavy metal ion content, such as lead, zinc, cobalt, nickel, and copper, has been affected by rapid industrialization and economic growth. Heavy metal is typically derived from industries such as mining, electroplating, and battery manufacturing. Even if heavy metal ion concentrations are minimal, it still has a harmful influence on human health if they are not appropriately managed. For living things, these heavy metals are divided into two categories; essential and non-essential trace elements. Table 1 illustrates the several excessive and high concentration heavy metal amounts affected on the human body.

Table 1. Parameters for the production of magnetic composites

Heavy Metal	Health Effect	Reference
Lead	Inhibition of harmoglobin. Damage of kidney and memory loss	[2]
Zinc	Loss of appetite and stiffness of muscular and nausea	[3]
Chromium (III)	Rspiratory problem and immunological system problem	[4]
Nickel	Skin irritation and lung and bone cancer	[5]

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Copper (Cu)

Copper which is produced by man-made activities such as smelting, metal cleaning, and electroplating baths, is one of the most prevalent heavy metal contaminants in the environment. It penetrated and flowed into the water sources in the forms of ions. Copper is an influential heavy metal band that belongs to the group of vital elements for living things. Excessive amounts, on the other hand, are damaging to the environment and cause health problem. The allowed amount of copper ions value for raw water quality standard is at most 1.0 mg/L. according to the National Standard for Drinking Water Quality, Engineering Services Division, Ministry of Health Malaysia [6] (Table 2). Abdominal cramp and diarrhea, liver and renal damage, and nausea were some of the side effect of too much copper, especially in newborns [7]. As a result, removing copper ions from wastewater is critical to preventing copper ion diffusion in the environment and reducing the negative impact on humans and other living things.

Table 2. Parameter limits for sewage and industrial effluents in mg/L

Heavy Metal	Standard	
	A	B
Mercury	0.005	0.05
Lead	0.1	0.5
Copper	0.2	1
Zinc	1	1
Iron (Fe)	1	5

Method of Heavy Metal Removal

To ensure removal efficiency, heavy metal pollutant and contaminant in waste water must be eliminated using the proper technique. Adsorption, ion exchange, chemical precipitation, membrane filtration, and electrochemical processes are some of the most prevalent methods for removing heavy metals. Despite the fact that several removal methods have been developed, the process is still hampered by limitations such as high costs. Previous research has found that adsorption is an excellent method for removing heavy metals and has a a number of advantages over other methods.

Adsorption

Adsorption has been chosen as one of the strategies for heavy metal removal because it has high removal capacity, is straightforward to treat, and produces less toxic secondary products [8]. Adsorbent and adsorbate are two commonly used terminologies in adsorption. A material or substance with a surface that can adsorb another substance is referred to as an adsorbent. Then there's the adsorbate, which is a substance that will be adsorbed on the adsorbent's surface and is normally in the form of an ion. In general, heavy metal ion adsorption occurs when ions in an aqueous solution adhere to a solid adsorbent surface as the adsorbate.

The right adsorbent must be chosen to ensure that heavy metals are removed from wastewater efficiently. Soil and mineral materials, agricultural products, marine sources, and many other materials can be used as adsorbents [9]. An excellent adsorbent, on the other hand must have a wide surface area, high adsorption capacity, and aqueous medium stability. Chemical adsorption and physical adsorption, sometimes known as chemisorption and physisorption, are two types adsorption that can occur. Due to its surface area, surface functions, and porosity, several studies and research have recently proven a good outcome in the use of geopolymer material as an adsorbent in adsorption methods [10].

Chemisorption

Chemisorption [11] is an irreversible process that refers to the formation of chemical bonds between the adsorbate and the adsorbent's surface. Chemisorption is difficult to reverse due to the strong connection, and the chemically adsorbed molecule is unable to desorb [12]. If the adsorbate can form a direct bond with the adsorbent surface, chemisorption can occur. Chemical bonds in chemisorption can have energies of up to 800 kJ/mole, making it an extremely strong bond [13].

Physisorption

The adsorbate ions adhere to the adsorbent surface due to the weak Van Der Waals force attraction in physical adsorption [11]. In contrast to chemisorption, the physisorption process is reversible, as well as being weakly bound. The molecule that adsorbed the ions only diffuse over the surface of the adsorbent and is not attached to a specific binding site in physisorption. The total surface area and pore volume, as well as the distribution of pore volume and pore area, will be revealed by physisorption analysis Due to the fluid passing through into the solid phase, absorption and adsorption are occasionally combined, Due to the weak Van Der Walls force, the physisorption energy is frequently quite high, exceeding 80 kJ/mole.
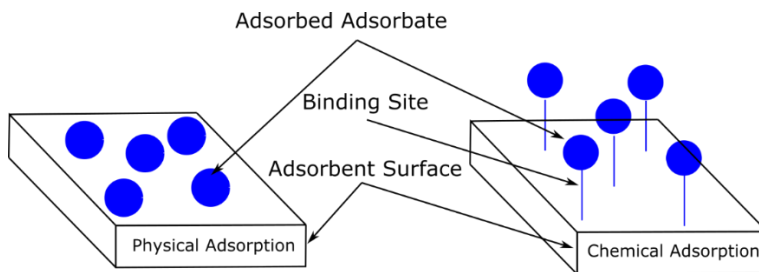

Fig. 1. Concept illustration of (a) physisorption and (b) chemisorption. Redrawn from [14]

Geopolymer

Joseph Davidovits invented aluminosilicates inorganic polymers, often known as geopolymers, in the 1970s. It has tetrahedral structure that is made up of Si-O-Al links. It was linked to an oxygen atom with a negative charge sites, which attracts positive charge ions in the solution [15]. According to previous studies, the structure is almost identical to a polymeric Si-O-Al framework observed in zeolites [16, 17]. Zeolites are an aluminosilicate clay mineral with a porous structure that allows metal ions to adsorb. However, zeolites require a higher temperature for synthesis, which contributes to the high operation cost, whereas geopolymer may be generated at a lower temperature, lowering the operation cost.

Geopolymerization is a reaction that occurs when the raw material (aluminosilicate) is mixed with an alkaline activator solution, Dissolution of aluminosilicate, gelation, polymerization, and hardening are the essential processes in the forming process [18]. Metakaolin, fly ash, slag and kaolin [19], as well as a mixture of sodium silicate (Na_2SiO_3) and sodium hydroxide, were used as common sources of aluminosilicates material in prior studies (NaOH). The sodium was chosen because to its widespread availability and lower cost than potassium. The alkaline activator, which mixes soluble silicate and alkaline hydroxide, produces a high-rate reaction.

Jaya et al. [20] produced metakaolin geopolymer with alkaline activator ratio, S/L ratio, and NaOH concentration of 0.20-1.20, 0.6-1.0, and 6M -14M, respectively, in a prior work. With 10M NaOH, 1.0 alkaline activator ratio, and 0.8 S/L ratios, the compressive strength was optimized. The compressive strength was reduced by increasing the mixing parameter beyond the ideal value. The compressive strength of geopolymer can be affected by curing, which aids in

rapid hardening. According to Zarina et al. [21], the maximum compressive strength of geopolymer was obtained by curing at 80°C for 24 hours, while the lowest strength was obtained by curing at ambient temperature due to inadequate geopolymerization. However, using a high temperature for curing is not necessary, as it can cause moisture loss.

Porous Geopolymer

Currently, porous geopolymer is generally recognized as a high-efficiency adsorbent in the adsorption method for removing heavy metal pollutants from wastewater. Because it has a high porosity, it has better removal ability than a traditional geopolymer [22]. The porous geopolymer is derived from a modification of a traditional geopolymer with numerous pores. The binding sites on the adsorbent are more available with porous geopolymer. Direct foaming, additive manufacturing, sacrificial filler method, replica method, and other way are several of fabrication methods for porous geopolymer fabrication. Owing to it is a simple and flexible process, the direct foaming method has been the sole extensively utilized way to synthesis porous geopolymer so far. In the manufacture of porous geopolymers, hydrogen peroxide (H_2O_2) was extensively used as a solution-based pore foaming agent. H_2O_2 was added to the geopolymer slurry in an alkaline environment and cured at temperatures ranging from 40 to 80°C.

The porosity created by the blowing agent through the gas bubble, on the other hand, was unstable, resulting in a porous structure with changing sizes and distribution. As a result, stabilizing agents such as surfactant can be added to prevent the structure from altering [23]. In general, gas is produced in a homogenous manner, and the trapped bubble gas in the geopolymer paste expands, causing macropores to void. As a result, the final characteristic, pore size, apparent density and microstructure are affected by variations in H_2O_2 content. As the foaming agent is increased, the material density decreases and more pores appear, resulting in a porous geopolymer [24].

Factor Affecting of Heavy Metal Removal

Solid-to-Liquid Ratio

The ratio of mixing between the raw material and the Na_2SiO_3/NaOH ratio is known as the solid-to-liquid (S/L) ratio. The physical parameters of geopolymer, such as slurry viscosity and geopolymer strength, will be affected by this ratio. Furthermore, a high S/L ratio does not imply great strength because a low amount binder formation is present by Heah et al. [25]. Aside from the physical feature, the S/L ratio has an impact on the adsorption efficiency. The geopolymer with raying S/L ratios has been utilized as the adsorbent in heavy metal removal by adsorption method, resulting in a range of the capacity that it can ingest. The high S/L ratio for kaolin based geopolymer adsorbent resulted in a reduction in heavy metal removal capability. The largest ratio employed in that experiment was 0.8 S/L, and while utilizing a 0.5 S/L ratio, nearly 80% of copper ions may be eliminated. Due to high aluminosilicate raw materials have a lower porous surface area and fewer adsorption sites.

Foaming Agent Ratio

The amount of foaming agent affects the number and volumes or f pores, which affected the adsorption performance of geopolymer adsorbent. According to Luukkonen et al. [26], the usage of a porous metakolin-based geopolymer as an adsorbent was successful in removing Ca^{2+}.Their described material, on the other hand, requires a longer contact time to attain equilibrium, resulting in a reduced adsorption capacity. Furthermore, the amount of H_2O_2 in the adsorbent affected the sorption capacity of the adsorbent in Pb removal. Owing to higher porosity, there is a larger adsorption site, which has increased the capacity of Pb adsorption to 6.34 mg/g. However, when compared to the capacity, the value is still insignificant.

Sintering Temperature

Sample sintering at different temperatures will have variable compressive strength, density and water absorption characteristics. The sample with the foaming agent was shown to have low compressive strength, low density, and a high degree of water absorption when exposed to sintering temperature. Also, the existence of pore size grew larger and more widely dispersed. As a result, the porous structure will trap heavy metal ions in aqueous solution.

Adsorbent Dosage

Adsorbent dose is another factor that can have a significant impact on adsorption capacity and performance. The highest removal effectiveness was seen while applying a high absorbent dosage, which provided more sorption sites and increase the adsorption of metal ions in the solution onto the adsorbent. However, as the concentration of metal ions increase, the removal effectiveness decreases, although the adsorbent dosage remains constant. This will result in a lack of binding sites for metal ions to adhere. According to Tan et al. [5], the removal effectiveness of Ni (II) decrease as the starting concentration of Ni (II) rises, but the dosage of porous geopolymer spheres remains constant. As a result, the binding sites are insufficient to make the adsorbate adhere, the ratio of adsorbent dosage to adsorbate amount be balanced in general, and this resulted in effective metal ion removal from aqueous solution.

Pore structure

Brunauer-Emmet-Teller (BET) analysis of the kaolin-geopolymer sample revealed a 1.0 S/L ratio decrease in the porous surface area of the sample. The porous surface area of the sample will decrease as the raw material is increased. As a result, the 0.5 S/L ratio revealed more adsorption sites than the 1.0 S/L ratio. Furtehrmore, Luukkonen et al. [5] found that porous geopolymer spheres with 1.2 wt% H_2O_2 addition have a large surface area of 54.76 m^2/g using BET analysis (Table 3). Due to the dense structure, the BET specific surface area of H_2O_2-containing samples was larger than non-formed samples in another studies by Masdiyana el al. [27]. As a result of the use of the foaming agent, the porosity of the surface area was increased. Nadiah et al. [28] described the pore structure of sintered and unsintered kaolin-based geopolymers, and the BET result revealed that the unsintered sample has the lowest surface area when compared to the fly ash and slag samples. However, when sintered at 900°C, kaolin geopolymer has the largest surface area and pore volume compared to unsintered samples.

Table 3.. BET surface area analysis of geopolymer

Sample Material	Surface Area (m^2/g)	Foaming agent (wt.%)	Ref
Porous Kaolin Geopolymer	54.81	Al Powder	[27]
Porous Metakaolin Geopolymer Spheres	53.95	H_2O_2	[28]
Porous Caclcine Kaolin Sphere Geopolymer	54.76	H_2O_2	[27]

Microstructure evaluation

As described by Nadiah et al. [28], the microstructure of sintered and unsintered kaolin-based geopolymer, with SEM result revealing the presence of well-defined clay platelets. The glassy and smooth surface was exmined at 900°C sintering temperature due to moisture hydration. Another study on fly ash lightweight geopolymer [29] showed the influence of different exposure temperature on the sample microstructure at 200°C, 400°C, 600°C, and 800°C. The sample already contains a high number of pores due to the addition of foam, and exposure at temperatures of 200°C and 400°C revealed a fine pore and no crack, however exposure at temperatures of 600°C and 800°C revealed a huge crack and pores. The addition of a foaming

agent to the geopolymer paste, on the other hand, had a significant impact on the void and porosity in the porous geopolymer.

Chemical bonding

The porous geopolymer's functional group and bonding were determined and described using Fourier Transform Infrared (FTIR) testing. The major bands of geopolymer are usually found between 1300 and 900 cm^{-1}, corresponding to asymmetric stretching vibrations of Si-O-T (T is tetrahedral Al or Si) [30]. Study by Ariffin et al. [31] looked at the FTIR spectra of kaolin-based geopolymer with scan ranges of 450-4000 cm^{-1} and found that the chain structure of kaolin changed from Si-O bond to Al-O-Si bond (1000 cm^{-1}) after reaching the curing temperature. Al-O-Si asymmetric stretching vibrations and Si-O-Si bending vibrations have a frequency range of 800 cm^{-1} to 400 cm^{-1}. The peak was estimated to be between 3500 and 3450 cm-1 and 1670 to 1680 cm^{-1}, according to the research.

Al-O-Si asymmetric stretching vibrations and Si-O-Si bending vibration have a frequency range of 800 cm-1 to 400 cm^{-1}. Due to the water present in geopolymer, the peak at roughly 3500 to 3450 cm^{-1} and 1670 to 1600 cm^{-1} is for stretching (OH) and bending vibration (H-O-H) of hydroxyl. Also visible is a CO_3^{2-} peak. Carbonates ion vibration is more common in samples with a high Na^+ ratio.

Phase analysis

In the research by Ariffin et al. [31], the XRD data of raw kaolin revealed that the predominant phase is crystalline, with kaolinite at 2θ of 12.1°, 25.2° quartz at 26.7°, and illite as an impurity at 17.9°. After synthesis, the XRD data revealed that the kaolinite peaks had vanished due to heat treatment and water dihydroxylation. In the alkaline solution, the crystalline phases were dissolved. Peak in sodalite were found at 17.8°, 21.7°, and 33.4°, indicating an interaction between Na, SiO_2, and Al_2O_3. According to Aziz et al. [32], the amorphous structure was revealed by the small intensity in the XRD pattern between 18° and 25°. This result was similarly obtained in the metakaolin sample by Jaya et al. [20]. The amorphous structure is indicated by the large diffraction hump between 18° and 25.2°. The XRD pattern was unaffected by the addition of H_2O_2 and the surfactant Tween 80.

Remarkable future

In environmental remediation methods, geopolymers have a lot of potential. Its high flexural strength, low carbon footprint, quick curing and solidification times, and toxic resistance provide new research opportunities. When properly developed, it has the potential to outperform activated carbon in the adsorption of dangerous compounds in polluted air and water. Because of its great porosity, this is the case. Geopolymer adsorption performance is influenced by the raw materials employed in its production. Waste water can be easily transformed to clean water by using geopolymer as a purifier with the right precursor and synthetic methods.

Geopolymer can also be used as photocatalyst supports because of the unique composition, which producesa variety of metal oxides. However, more research is needed in order to develop scalable geopolymer adsorbent and photocatalyst support technologies. In the near future, surface nanostructuring, coating, and composite synthesis are projected to be key activities. In all of these scenarios, synthesis and process controls will be important to optimize.

Conclusions

Geopolymeric materials have the potential to provide useful solutions to environmental remediation issues. These solutions are expected to be low-maintenance and relevant to the scalability requirement of today's industrial waste waste treatment plants. Geopolymer has become a material relevant for cleaner production and green technologies due to its ability to (i) synthesize from readily available raw materials and (ii) fabricate employing room temperature techniques with low or zero greenhouse gas emissions. This opens up 'green technology' potential in related areas such as building, surface engineering, and healthcare.

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