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CHEMISTRY DEPARTMENT

**Removal of lead (II) ions from solution by adsorption using geopolymer derived from sugar
bagasse ash (SBA)**

by

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APPROVAL FORM

The undersigned certify that they have supervised, read and recommend to the Bindura University of Science Education for the acceptance of a research dissertation entitled: **Removal of lead (II) ions from solution by adsorption using geopolymer derived from sugar bagasse ash (SBA)**, submitted by **Xavier T Chidawanyika** in the partial fulfilment of the requirements for the Bachelor of Science Honors Degree in Chemical Technology.

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(Signature of Chairperson)  Date.....**02/10/2025**.....

DECLARATION

As Xavier Chidawanyika, I declare that this dissertation is entirely my own original research, and I am the sole author. I have properly cited all sources for any ideas or information I have used from others. This work has not been submitted or accepted anywhere else for any academic award or recognition.

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ABSTRACT

The escalating global challenge of heavy metal pollution, particularly lead (Pb(II)) ions in aqueous environments, necessitates efficient, sustainable, and cost-effective remediation. This study addresses this by valorizing sugar cane bagasse ash (SBA), an abundant agricultural waste, into a novel geopolymer adsorbent for Pb(II) removal.

Geopolymers were successfully synthesized from SBA via alkali activation. Characterization using Fourier Transform Infrared (FTIR) spectroscopy confirmed the aluminosilicate network (Si-O-T band around 1019 cm^{-1}) and the presence of crucial hydroxyl groups. X-ray Diffraction (XRD) patterns revealed a predominantly amorphous structure (broad hump around $2\theta=20\text{--}30$ degrees), indicative of successful geopolymerization and beneficial for adsorption. Adsorption experiments systematically evaluated Pb(II) removal under varying conditions. Optimal adsorption occurred at pH 4.5-5, attributed to increased negative surface charge. Kinetic studies showed the process followed the pseudo-second-order model ($R^2=0.9973$), suggesting chemisorption as the rate-limiting step. The calculated equilibrium adsorption capacity (q_e) was 4.73 mg/g . Thermodynamic investigations indicated a positive standard enthalpy change ($\Delta H^\circ=81.15\text{ kJ/mol}$), signifying an endothermic process favored by higher temperatures. A positive standard entropy change ($\Delta S^\circ=284.51\text{ J/molK}$) suggested increased randomness at the interface. Consistently negative standard Gibbs free energy change (ΔG°), ranging from -2.31 kJ/mol at 20°C to -13.69 kJ/mol at 60°C , confirmed the spontaneity and thermodynamic favorability of the adsorption.

In conclusion, this study successfully demonstrated that SBA-derived geopolymers are efficient and sustainable adsorbents for Pb(II) removal. This research offers a dual environmental benefit:

sustainable agricultural waste management and improved water quality, highlighting the significant potential of these cost-effective and eco-friendly materials for heavy metal remediation.

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DEDICATION

I dedicate this research project to my parents, who have been my unwavering pillars of support and encouragement throughout my entire academic journey. Their constant belief in my potential has been an endless source of motivation. I also dedicate this to my professors and mentors, whose guidance and knowledge have been truly invaluable. And finally, to my friends, who have been my companions through all the ups and downs of this journey.

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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
ATSDR	Agency for Toxic Substances and Diseases Registry
C_i	Boundary Layer Thickness
C_o	Initial Concentration
C_t	Concentration at Time t
FTIR	Fourier Transform Infrared Spectroscopy
IARC	International Agency for Research on Cancer
SBA	Sugar Bagasse Ash
XRD	X-ray Diffraction
WHO	World Health Organization
USEPA	United States Environmental Protection Agency
TGA	Thermogravimetric Analysis
T	Time
SEM	Scanning Electron Microscopy
R^2	Coefficient of Determination
Q_t	Adsorption Amount at Time t
Q_{max}	Maximum Adsorption Capacity
q_e	Equilibrium Adsorption Capacity
PZC	Point of Zero Charge
NPDRWs	National Primary Drinking Water Regulations
NPDES	National Pollutant Discharge Elimination Systems
MDPI	Multidisciplinary Digital Publishing Institute
MCLs	Maximum Contamination Levels
k_1	Rate Constant of the Pseudo First Order
k_2	Rate Constant of the Pseudo Second Order
K_{id}	Rate of Intraparticle Diffusion

K_L	Langumuir Constant
K_F	Freudlich Constant
ΔS°	Standard Entropy Change
ΔH°	Standard Enthalpy Change
ΔG°	Standard Gibbs Free Energy Change

CHAPTER 1: INTRODUCTION

1.1 INTRODUCTION

The escalating pace of industrialization and urbanization globally has inevitably led to a significant increase in environmental pollution, with heavy metals emerging as particularly persistent and hazardous contaminants. Among these lead (Pb^{2+}) stands out as a prevalent and highly toxic heavy metal (Dixit et al., 2015). Lead enters the environment through various anthropogenic activities, including mining, battery manufacturing, smelting operations, paint production and the discharge of industrial effluents (Bradl, 2005). Its non-biodegradable nature means it persists in the environment, accumulating in soil, water bodies and ultimately, the food chain. Exposure to lead even at low concentrations poses severe threats to human health, affecting neurological, renal, hematopoietic and reproductive system (ATSDR,2020). The insidious nature of lead toxicity coupled with its widespread presence in industrial wastewater necessitates the development of efficient and sustainable methods for its removal from aqueous environments before discharge.

Conventional methods employed for lead removal from wastewater include chemical precipitation, ion exchange, membrane filtration, coagulation-flocculation and adsorption using activated carbon (Fu and Wang, 2011; Kurniawan et al., 2006). While these techniques have demonstrated varying degrees of efficiency they often present challenges such as high operational costs, generation of secondary waste sludge, sensitivity to pH fluctuations and limited efficiency at low lead concentrations. These limitations underscore the urgent need for innovative, cost effective and environmentally benign technologies for lead remediation.

In line with global sustainability initiatives, the valorization of industrial and agricultural waste materials has emerged as a promising avenue for addressing environmental challenges (Mohanty et al., 2018). This approach not only provides solutions for waste management but also transforms discarded materials into valuable resources. In agricultural economies such as Zimbabwe, significant quantities of agro-industrial residues are generated annually. Among these sugar cane bagasse ash (SBA) a byproduct from the incineration of sugarcane bagasse for energy generation in sugar mills, is produced in large volumes. However, SBA is rich in pozzolanic components, primarily silicon oxide (SiO_2) and aluminum oxide (Al_2O_3) making it a viable alternative raw material for various applications, including the synthesis of geopolymers (Mendes et al., 2021).

Geopolymers are inorganic polymeric materials synthesized through the alkali activation of aluminosilicate precursors such as fly ash, metakaolin, blast furnace slag and increasingly agricultural waste ashes (Davidovits, 1991; Provis and van Deventer, 2009). The geopolymerization process involves the dissolution of the amorphous or semi crystalline aluminosilicate phases in a highly alkaline solution, followed by the polycondensation of dissolved species into a rigid, three-dimensional network. These materials exhibit a range of advantageous properties, including high mechanical strength, excellent thermal stability, low permeability and resistance to chemical attack (Duxson et al., 2007). Crucially, the unique microstructure and surface chemistry of geopolymers, characterized by the presence of negatively charged sites resulting from the isomorphic substitution of Si^{4+} by Al^{3+} in the tetrahedral framework, render them highly effective adsorbents for various pollutants, including cationic heavy metal ions like Pb(II) (Auta and Hameed, 2014; Zhang et al., 2019).

The utilization of SBA as a precursor for geopolymer synthesis offers a dual environmental benefit: it provides a sustainable pathway for managing an abundant agricultural waste product

and simultaneously facilitates the production of a low cost, high performance adsorbent for heavy metal removal, specifically lead. While the potential of geopolymers as adsorbents is recognized, a comprehensive investigation into the specific synthesis parameters, detailed characterization and systematic evaluation of Pb(II) adsorption performance of geopolymers exclusively derived from SBA particularly in the Zimbabwean context remains an area requiring in-depth study. This research aims to address this gap by exploring the full potential of SBA based geopolymers as sustainable and efficient adsorbents for Pb(II) ions.

1.2 RESEACH PROBLEM

Lead contamination in water bodies poses a persistent and severe threat to both ecological systems and human health, particularly in regions where industrial activities contribute to its discharge. Current remediation technologies for Pb(II) removal often face limitations concerning cost effectiveness, especially for low concentration effluents. Concurrently, the increasing accumulation of agricultural waste, such as SBA presents a significant disposal challenge. Despite the known adsorptive capabilities of geopolymers, the systematic development, optimization and characterization of geopolymers synthesized solely from SBA for Pb(II) adsorption remain underexplored. There is a critical need to understand how variations in geopolymer synthesis parameters influence the physicochemical properties of SBA based geopolymers and consequently their efficacy in removing Pb(II) ions. Furthermore, a comprehensive characterization of these novel materials is essential to elucidate the underlying adsorption mechanisms, thereby enabling the rational design of highly efficient, low cost and potentially reusable adsorbents derived from an abundant waste resource.

1.3 AIMS AND OBJECTIVES

1.3.1 Aim

The aim of this research is to synthesize, characterize and evaluate the Pb(II) adsorption performance of geopolymers derived from sugar cane bagasse ash.

1.3.2 Objectives

1. To synthesize geopolymer using sugar cane bagasse ash as the primary aluminosilicate source.
2. To characterize the synthesized geopolymer.
3. To evaluate its Pb(II) adsorption performance of from aqueous solutions under varying operational conditions.

1.4 JUSTIFICATION OF THE STUDY

The study directly addresses the challenge of agricultural waste management by transforming sugar cane bagasse ash into a valuable material. This aligns with circular economy principles, promoting resource efficiency and reducing landfill burden. Utilizing a readily available and low cost agricultural waste as a raw material for geopolymer synthesis offers a potentially more economical alternative to conventional and often expensive commercial adsorbents. Lead contamination is a pervasive environmental issue. This research provides a localized, sustainable solution for the removal of Pb(II) from industrial effluents and contaminated water sources, contributing directly to improving water quality and public health in affected communities. The comprehensive investigation into synthesis, characterization, kinetics and mechanisms will contribute to a deeper understanding of SBA-based geopolymers as adsorbents. This fundamental knowledge is a vital for the rational design and optimization of future generations of efficient and

specialized geopolymers. Successful development and scaling up of the technology could reduce reliance on imported water treatment solutions, potentially fostering local innovation, creating new industries and generating employment opportunities.

1.5 HYPOTHESIS

Geopolymers synthesized from SBA will exhibit well defined amorphous aluminosilicate structures with specific surface properties, enabling efficient and regenerable adsorption of Pb(II) ions from aqueous solutions via surface complexation and ion exchange mechanisms.

CHAPTER 2 LITERATURE REVIEW

2.1 INTRODUCTION

The pervasive contamination of water sources with heavy metals presents a significant global challenge with detrimental consequences for both human health and ecosystem integrity. Heavy metals, defined as metallic elements with a relatively high density that are toxic or poisonous even at low concentrations (Alloway, 2013), originate from diverse anthropogenic sources, including industrial discharges, mining activities, agricultural runoff, and improper waste disposal (Hudson-Edwards et al., 2019). These pollutants, such as cadmium (Cd), lead (Pb), chromium (Cr), arsenic (As), mercury (Hg), copper (Cu), nickel (Ni), and zinc (Zn), pose severe risks due to their persistence in the environment, bioaccumulation in food chains, and subsequent toxic effects on human health, including neurological disorders, kidney damage, various cancers, and developmental problems (Tchounwou et al., 2011). The World Health Organization (WHO) consistently emphasizes the urgent need for effective heavy metal removal from water sources, as millions worldwide lack access to safe drinking water due to contamination (WHO, 2021). While comprehensive global statistics on heavy metal water pollution are difficult to aggregate due to variations in monitoring capacities and reporting standards, regional studies frequently reveal alarming concentrations exceeding permissible limits, particularly in rapidly developing nations (Liu et al., 2015). This underscores the critical demand for effective and sustainable remediation strategies.

Conventional water treatment methods, such as chemical precipitation, ion exchange, and reverse osmosis, though widely employed, often exhibit limitations regarding cost-effectiveness, removal efficiency, and the generation of secondary waste streams (Fu & Wang, 2011). Chemical

precipitation, for instance, may be inefficient for removing certain heavy metal species and can produce substantial volumes of sludge requiring further management (USEPA, 2000). Ion exchange, while effective, can be expensive and necessitates periodic regeneration, leading to the production of concentrated waste solutions (Dabrowski et al., 2004). These limitations highlight the urgent need for alternative, environmentally benign, and economically viable solutions for heavy metal removal from contaminated water.

Adsorption has emerged as a promising technique for heavy metal remediation due to its high efficiency, relatively low cost, and ease of operation (Weber et al., 2019). This process involves the binding of heavy metal ions (adsorbate) onto the surface of a solid material (adsorbent). Several mechanisms can be involved in the adsorption process, including surface complexation, ion exchange, electrostatic interactions, and micropore filling (Sposito, 1989). The effectiveness of adsorption is strongly influenced by the properties of the adsorbent material.

Recently, geopolymers have garnered considerable attention as potential alternatives to conventional adsorbents for heavy metal removal (Davidovits, 2017). Geopolymers are aluminosilicate materials synthesized through the alkali activation of aluminosilicate precursors, such as fly ash, metakaolin, or slag (Provis & van Deventer, 2009). They possess a unique combination of desirable properties that make them attractive for adsorption applications, including high surface area, interconnected porosity, excellent chemical stability over a broad pH range, low cost due to the potential utilization of industrial waste materials as precursors, and the ability to tailor their surface properties through modifications (Pacheco-Torgal et al., 2017). This literature review will examine the current state of knowledge regarding the application of geopolymers as adsorbents for the removal of heavy metals from contaminated water. It will explore the synthesis and characterization of geopolymers, the mechanisms of heavy metal

adsorption, the factors influencing adsorption performance, and strategies for enhancing their adsorption capacity and selectivity. Furthermore, the review will address the regeneration and reusability of geopolymers, the potential for waste utilization in their production, and future research directions in this promising field.

2.2 Heavy Metals and Their Environmental Impact

2.2.1 Specific Heavy Metals of Concern

This review focuses on several key heavy metals commonly found in contaminated water: lead (Pb), cadmium (Cd), chromium (Cr), arsenic (As), mercury (Hg), copper (Cu), nickel (Ni), and zinc (Zn). Pb, primarily originating from industrial discharges, lead-acid batteries, and historically from leaded gasoline, can cause severe neurological damage, particularly in children (ATSDR, 2007). Cd, often associated with industrial wastewater, mining, and fertilizers, is known for its carcinogenic properties and its ability to cause kidney and bone damage (IARC, 1993). Cr exists in various oxidation states, with Cr(VI) being highly toxic and carcinogenic, primarily originating from industrial processes like electroplating (Laskin & Rivera, 2004). As, both naturally occurring and anthropogenic, can contaminate water through geological processes and industrial activities, causing various health problems, including skin lesions, cancers, and cardiovascular diseases (WHO, 2018). Hg, released from industrial activities and artisanal gold mining, is a potent neurotoxin, particularly in its methylated form, accumulating in the food chain and posing significant risks to pregnant women and developing fetuses (Grandjean et al., 2014). Cu, Ni, and Zn, while essential micronutrients at low concentrations, can become toxic at elevated levels. Cu, from industrial discharges and plumbing systems, can cause gastrointestinal problems (Gaetke, 2017). Ni, from industrial processes, can cause allergic reactions and dermatitis (IARC, 2012). Zn, from industrial effluents, can affect the immune system and impair neurological function (Plumlee

et al., 2006). These metals enter water bodies through various pathways, including direct industrial discharge, leaching from contaminated soils, atmospheric deposition, and runoff from mining and agricultural areas. Their presence in water poses significant threats to aquatic ecosystems, disrupting biological processes and impacting biodiversity.

2.2.2 Regulations and Standards

Numerous international and national organizations have established water quality guidelines and regulations to limit heavy metal concentrations in drinking water and wastewater discharge. The WHO, for instance, sets guidelines for heavy metal concentrations in drinking water (WHO, 2017). The United States Environmental Protection Agency (USEPA) establishes National Primary Drinking Water Regulations (NPDWRs) and National Pollutant Discharge Elimination System (NPDES) permits for various heavy metals (USEPA, 2022). Similar regulations exist in other countries and regions, often tailored to specific local conditions and environmental concerns. These regulations typically specify maximum contaminant levels (MCLs) for each heavy metal to protect human health and the environment.

2.3 Geopolymers

Geopolymer or alkali-activated aluminosilicate is a diverse group of ceramic-like materials produced by geosynthetic reaction of aluminosilicate minerals in the presence of an alkali solution at low temperature (less than 100 degrees Celsius) (Nikolic et al., 2015; Phair et al., 2004). It consists of a polymeric silicon-oxygen-aluminium framework with alternating silicon and aluminium tetrahedral joined together in three directions by sharing all the oxygen atoms (Ge et al., 2015). Fly ash, dolomite, expanded clay, natural zeolite, kaolinite, sugarcane bagasse and many are example of geopolymers. Geopolymers had interesting properties that are encouraged to investigated and developed. The major nature of geopolymers is tendency to drastically decrease

the mobility of most heavy metals ions contained within the geopolymeric structure, quick compressive development of strength, resistance to acid and fire, fat setting, low permeability and good resistance to freeze-thaw cycles (Al Zboon et al., 2011; Andrejkovicova et al., 2016). Theoretically, any alkali can be used in geopolymerization reactions; however, most of the studies have focused on the effect of sodium (Na^+) and potassium (K^+) ions. Both NaOH and KOH could be used in the activation process, but the extent of dissolution was higher when NaOH is used; this is due to the smaller size of Na^+ which can better stabilize the silicate monomers and dimers present in the solution, enhancing the minerals dissolution rate (Al Harahsheh et al., 2015). Studies have shown that geopolymer treated with NaOH is better than treated geopolymer by KOH (Williams et al., 2009).

2.3.1 Geopolymerization Process

Geopolymerization involves the reaction of an aluminosilicate source material (e.g., fly ash, metakaolin, slag or sugarcane bagasse ash) with an alkaline activating solution (e.g., sodium silicate, potassium hydroxide) (Davidovits, 2017). The process involves dissolution of the aluminosilicate source, followed by polycondensation and network formation, resulting in a three-dimensional aluminosilicate framework. The properties of the final geopolymer product are significantly influenced by the raw materials used, the composition and concentration of the activating solution, and the curing conditions (temperature and time) (Provis & van Deventer, 2009). Higher curing temperatures and longer curing times generally lead to increased strength and improved geopolymerization.

2.3.2 Geopolymer Classification

Geopolymers can be classified based on their source materials (e.g., fly ash-based, metakaolin-based, slag-based) and their structural characteristics (e.g., zeolitic-like, layered, three-dimensional framework) (Pacheco-Torgal et al., 2017).

2.3.3 Characterization Techniques

X-ray diffraction (XRD) analysis is crucial in characterizing geopolymers for Pb^{2+} adsorption, as it provides insights into the material's mineralogical composition, phase changes and crystallinity. Studies on geopolymers for heavy metal adsorption often reveal a predominantly amorphous structure, which is characteristic of the geopolymerization process where aluminosilicate precursors react to form an amorphous gel (Kenyatta University, 2022). While raw sugar bagasse ash may contain crystalline phases like quartz, the geopolymerization process typically leads to a reduction or disappearance of these sharp crystalline peaks, indicating the formation of a disordered, three-dimensional network (Dharani et al., 2020). The amorphous nature of the geopolymer is beneficial for adsorption as it often provides more active sites for metal ion interaction. The presence of broad hump rather than distinct peaks in the XRD pattern confirms the successful formation of an amorphous geopolymer structure, which plays a significant role in the adsorption mechanism of Pb^{2+} through ion exchange and surface complexation (Dharani et al., 2020; Kenyatta University, 2022).

Fourier Transform Infrared (FTIR) spectroscopy is an indispensable tool for investigating the functional groups and chemical bonding within the geopolymers derived from SBA, particularly in relation to Pb^{2+} adsorption. The FTIR spectra of geopolymers typically exhibit a broad adsorption band around $950\text{-}1000\text{ cm}^{-1}$, which is attributed to the asymmetric stretching vibrations of Si-O-T bonds (T represents Si or Al) within the aluminosilicate framework, confirming the formation of the geopolymeric gel (Kenyatta University, 2022). Other characteristic bands include

those at approximately 3400 cm^{-1} and 1650 cm^{-1} , corresponding to the stretching and bending vibrations of O-H groups from adsorbed water and silanol/aluminol groups, respectively (Kenyatta University, 2022; Madzivire et al., 2022). Upon adsorption of Pb^{2+} , shifts in the position or changes in the intensity of these bands, particularly the Si-O-T band, can indicate the involvement of these functional groups in the binding of Pb^{2+} to the geopolymer surface, suggesting mechanisms such as ion exchange, complexation or precipitation (Madzivire et al., 2022).

2.3.4 Properties of Geopolymers Relevant to Adsorption

The efficacy of geopolymer derived from SBA for Pb^{2+} adsorption is intrinsically linked to their unique physicochemical properties, which are significantly influenced by the geopolymerization process. Key properties include a high surface area, significant porosity and a negatively charged surface. The three dimensional amorphous network structure inherent to geopolymers provides a larger number of accessible adsorption sites (Encyclopedia.pub, n.d.). This mesoporous structure and high porosity, often ranging between $16.2\text{ m}^2/\text{g}$ and $216\text{ m}^2/\text{g}$, directly contribute to an increased contact area between the adsorbent and Pb^{2+} , thereby enhancing adsorption capacity (ResearchGate, 2025).

Furthermore, the presence of tetrahedral aluminum (Al^{3+}) units within the aluminosilicate framework imparts a net negative charge to the geopolymer surface. This negative charge is crucial for attracting and binding positively charged metal ions like Pb^{2+} through electrostatic interactions and ion exchange mechanisms, where alkali metal ions e.g., Na^+ or K^+ in the geopolymer structure are replaced by the heavy metal ions (MDPI, n.d.-a; Purbasari et al., 2023). The abundance of silanol ($-\text{Si}-\text{OH}$) and aluminol ($-\text{Al}-\text{OH}$) functional groups on the geopolymer surface also facilitates surface complexation with Pb^{2+} , further enhancing their immobilization (MDPI, n.d.-a).

2.3.5 Adsorption Mechanisms

The adsorption of Pb^{2+} onto SBA based geopolymers is a complex process involving a combination of several mechanisms, primarily driven by the unique surface chemistry and structural characteristics of the geopolymer. One of the primary mechanisms is ion exchange, where Pb^{2+} from the aqueous solution exchange with alkali metal cations such as Na^+ or K^+ that are loosely bound within the geopolymer framework to balance the negative charge imparted by the tetrahedral aluminum (AlO_4^-) units (Kenyatta University, 2022). This process is facilitated by the mobility of these alkali ions within the porous geopolymer structure.

Another significant mechanism is surface complexation, involving the formation of chemical bonds between Pb^{2+} and the active functional groups present on the geopolymer surface. These active sites primarily include silanol ($-\text{Si}-\text{OH}$) and aluminol ($-\text{Al}-\text{OH}$) groups, which can deprotonate to form negatively charged oxygen atoms that strongly coordinate with the positively charged Pb^{2+} ions (Madzivire et al., 2022). The high density of these surface hydroxyl groups, as evidenced by FTIR analysis, provides numerous binding sites for Pb^{2+} (Kenyatta University, 2022).

Furthermore, electrostatic attraction plays a crucial role. The overall negative surface charge of the geopolymer, resulting from the isomorphic substitution of silicon by aluminum in the framework, creates an attractive force for the positively charged Pb^{2+} ions, facilitating their migration towards and retention on the geopolymer surface (ResearchGate, 2025). In some cases, particularly at higher Pb^{2+} concentrations or under specific pH conditions, precipitation of lead compounds e.g., lead hydroxide on the geopolymer surface or within its pores can also contribute to the overall removal efficiency, acting as a co-precipitation or surface precipitation mechanism (MDPI, n.d.). The porous structure of the geopolymer also allows for physical adsorption and diffusion of Pb^{2+} into the internal pore network, increasing the overall adsorption capacity (ResearchGate, 2025).

2.3.6 Factors Affecting Adsorption

Some factors are influence in the process of adsorption of heavy metal into adsorbents such as pH, adsorbent dosage, initial concentration, contact time and temperature (Lim et al., 2014). Removal efficiency of heavy metals can be described as following equation (Luukonen et al., 2016):

$$\text{Removal efficiency (\%)} = \frac{(C_o - C_t)}{C_o} \times 100$$

Equation 2.1

Where C_o (mg/L) is the initial concentration and C_t (mg/L) is the concentration at time t (min). Adsorption amount at time t , q_t (mg/g) and at equilibrium, q_e (mg/g) are determined using following equations:

$$q_t = \frac{(C_o - C_t)V}{m}$$

Equation 2.2

$$q_e = \frac{(C_o - C_t)V}{m}$$

Equation 2.3

Effect of pH: pH significantly influences the speciation of heavy metals and the surface charge of the geopolymer, affecting adsorption efficiency. The surface charge of the solution, degree of ionization, and the adsorbate species influenced by the pH of the solution. Most of the metal adsorbed increase with the increasing of pH of the solution until certain point and followed by reduction if further increasing of pH. The equation of pH can be written as:

$$\text{pH} = \text{pka} - \log \frac{[AH]}{[A-]}$$

Equation 2.4

where, [A⁻] and [AH] represent the concentration of deprotonated and protonated surface groups and the equilibrium constants pK_a resembled the carbonyl groups. Study show that adsorption efficiency increase from 1% to 90.66% as pH of the solution increase from 1 to 5 and slightly decrease at pH 6 (Al Zboon et al., 2011). Therefore, pH 5 is indicated as zero-point charge. The pH at the point of zero charge (PZC) of the geopolymer, determined to be 5.0, provides crucial insight into its surface chemistry and its efficacy as an adsorbent for heavy metal ions like lead (Pb(II)). The PZC represents the pH value at which the net surface charge of the adsorbent is zero. Understanding the PZC is fundamental because it dictates the electrostatic interactions between the adsorbent surface and the adsorbate ions in solution (Ariffin et al., 2017).

Below the PZC (i.e., at pH values less than 5.0), the geopolymer surface tends to be positively charged due to the protonation of surface hydroxyl groups (e.g., $\text{-Si-OH} + \text{H}^+ \rightleftharpoons \text{-Si-OH}^{+2}$). In such acidic conditions, there is a high concentration of H⁺ ions in the solution, which compete with cationic heavy metal ions like Pb(II) for available adsorption sites. This competition, coupled with the repulsive electrostatic forces between the positively charged surface and the positively charged metal ions, typically leads to reduced adsorption efficiency.

Conversely, above the PZC (i.e., at pH values greater than 5.0), the geopolymer surface becomes increasingly negatively charged due to the deprotonation of surface functional groups (e.g., $\text{-Si-OH} \rightleftharpoons \text{-Si-O}^- + \text{H}^+$). This negatively charged surface strongly attracts positively charged metal ions such as Pb(II) through electrostatic interactions, thereby enhancing adsorption. As the pH increases further beyond the PZC, the availability of negatively charged sites on the geopolymer surface increases, leading to a more favorable environment for the adsorption of cationic species.

Effect of adsorbent dosage: Dose of adsorbent also is one of the main points to determine the capacity uptake of heavy metals by adsorbents. Usually, increase in the dose of adsorbents will increase in the adsorbed capacity until its reach a limit. If further increase the dose, the adsorption capacity will be constant. Wang study adsorbent loading from 0.5 g until 2.0 g, removal of copper increase with increase of adsorbent loading (Wang et al., 2007).

Effect of initial concentration: Adsorption dosage gain a strong effect by initial concentration of heavy metals. Generally, adsorption capacity increased with the increased initial concentration of heavy metals. Playing as important driving force, initial concentration influence in overcome all mass transfer resistance between solid and aqueous phases. Several studies have shown that removal efficiency of heavy metal is concentration dependent and there exist decreasing trend if further increase initial concentration (Jaarsveld et al., 2002).

Effect of contact time: Adsorption is a time-dependent process, and sufficient contact time is required to reach equilibrium. The interaction of functional group between the solution and the surface of adsorbent result in the adsorption capacity if adsorbate into adsorbent. Specific time needed to maintain equilibrium interaction therefore the adsorption process undergo completion. Cadmium removal using zeolite based geopolymer achieve equilibrium contact time at 7 hours (Javadian et al., 2015). However, fly ash-based geopolymer for lead removal gain equilibrium contact time at 120 min and thereafter, its remain constant (Al Zboon et al., 2011).

Effect of temperature: Temperature can affect the adsorption process, influencing the adsorption equilibrium and kinetics. The natures of the processes either exothermic or endothermic depends on the adsorption equilibrium that is affected by the temperature used. The uptake capacity of the adsorbates increases with the rises of the temperatures. This happens due to the enlargement of

pores and activation of the sorbent surface. Research shows removal of cadmium was increased with temperature range from 25°C to 45°C (Javadian et al., 2015).

2.3.7 Adsorption Isotherms

Adsorption isotherms describe the equilibrium relationship between the amount of heavy metal adsorbed and its concentration in solution. Common isotherm models include:

Langmuir isotherm: Assumes monolayer adsorption onto a homogeneous surface with a finite number of identical adsorption sites (Langmuir, 1916). Langmuir isotherm model is given by equation:

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max}K_L C_e}$$

Equation 2.5

Where q_e is the amount of heavy metal ions adsorbed at equilibrium, q_{max} is the maximum adsorption capacity (mg/g), K_L is the Langmuir constant and C_e is the heavy metal ion concentration.

Freundlich isotherm: Assumes multilayer adsorption onto a heterogeneous surface with varying adsorption energies (Freundlich, 1906). Freundlich isotherm model is given by equation:

$$\log q_e = \log K_F + \log C_e$$

Equation 2.6

Where q_e is the amount of heavy metal ions adsorbed at equilibrium, K_F is the Freundlich constant and C_e is the heavy metal ion concentration at equilibrium (mg/L).

Temkin isotherm: Considers the effect of indirect adsorbate-adsorbate interactions on adsorption (Temkin, 1941). The core idea behind the Temkin model is that the heat of adsorption or binding energy of all molecules in the adsorbed layer decreases linearly with increasing surface coverage (Ayawei et al., 2017; Chu, 2021). This contrasts with the Lungmuir model which assumes a constant heat of adsorption across all sites and the Freudlich model which implies a logarithmic decrease. It is given by the equation:

$$q_e = B_T \ln(A_T) + B_T \ln(C_e)$$

Equation 2.7

These models are used to determine the maximum adsorption capacity of the geopolymer for a specific heavy metal.

2.3.8 Adsorption Kinetics

Adsorption kinetics studies the rate of heavy metal adsorption onto geopolymers. Common kinetic models include:

Pseudo-first-order: Assumes that the rate of adsorption is proportional to the difference between the amount of metal adsorbed at equilibrium and the amount adsorbed at time t .

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303}$$

Equation 2.8

Pseudo-second-order: Assumes that the rate of adsorption is proportional to the square of the difference between the amount of metal adsorbed at equilibrium and the amount adsorbed at time

$t.$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

Equation 2.9

Where q_t is the metal uptake per unit weight of adsorbent (mg g^{-1}) at time t , while q_e is the metal uptake per unit weight of adsorbent (mg g^{-1}) at equilibrium. k_1 (min^{-1}) and k_2 ($\text{mg g}^{-1} \text{min}^{-1}$) are the rate constants of the first-order kinetics and second order kinetics respectively (min^{-1}). The slopes and intercepts are used to determine the values of k_1 and k_2 as well as the equilibrium capacity (q_e) (Chen, 2015). It is the plot that has the higher regression coefficient, R^2 , that gives the best fit.

2.3.9 Thermodynamic Studies

Thermodynamic parameters like Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) provide insights into the spontaneity and feasibility of the adsorption process (Atkins & de Paula, 2014). These parameters are essential in understanding how geopolymers perform and their mechanisms when used to remove heavy metals from water (Foo & Hameed, 2010). A focus on Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) offers valuable insights into the nature and feasibility of adsorption processes. A negative ΔG suggests that the adsorption occurs spontaneously and is thermodynamically favorable (Atkins & de Paula, 2014). In contrast, ΔH indicates if the process is endothermic or exothermic: a positive ΔH implies increased adsorption efficiency with rising temperature, typical of an endothermic reaction, while a negative ΔH indicates exothermic behavior, with optimal performance at lower temperatures (Foo & Hameed, 2010).

$$\ln K_C = -\frac{\Delta H}{RT} + \frac{\Delta S}{R}$$

Equation 2.10

Standard Gibbs Free energy is given by the equation:

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ}$$

Equation 2.11

Isotherm models, like Langmuir and Freundlich, are commonly used in thermodynamic studies to assess the adsorption capacity and surface interactions of geopolymers (Wang et al., 2020). These models enhance the understanding of how geopolymers interact with specific heavy metal ions, aiding in the optimization of their synthesis and functionalization (Mao et al., 2020; Wang et al., 2020).

The dependence of adsorption efficiency on temperature highlights the significance of thermodynamics in adapting geopolymers for practical uses. Analyzing thermodynamic parameters allows researchers to refine raw materials, synthesis methods, and functionalization approaches to improve the adsorption capabilities of geopolymers (Mao et al., 2020).

In summary, thermodynamic studies validate the effectiveness of geopolymers as adsorbents and facilitate their optimization and application in real-world water treatment. This understanding is crucial for promoting geopolymers as efficient, sustainable, and cost-effective materials for environmental remediation (Mokgosi et al., 2022; Wang et al., 2020).

2.4 Waste Utilization in Geopolymer Synthesis

2.4.1 Beneficial Use of Waste Materials

A significant advantage of geopolymer synthesis is the potential for utilizing industrial waste materials as precursors. This aligns with circular economy principles and addresses waste disposal problems (Lahoti et al., 2020). Various waste materials can be used as aluminosilicate sources, including fly ash from coal combustion, slag from metallurgical industries, red mud from alumina production, sugar cane bagasse ash (SBA) and mine tailings. Using these wastes reduces the cost

of geopolymer production and minimizes the environmental burden associated with waste disposal (Wang et al., 2021).

2.4.2 Impact on Geopolymer Properties

The type and characteristics of the waste material used influence the properties of the resulting geopolymers. For instance, fly ash-based geopolymers often exhibit high strength and durability, while red mud-based geopolymers may possess specific surface properties suitable for certain heavy metal adsorption (Al-Edani et al., 2021; Yeheyis et al., 2022). The chemical composition, mineralogy, and particle size of the waste material affect the geopolymerization process and the final product's properties, including its mechanical strength, porosity, and adsorption capacity. Careful characterization of the waste material is essential to optimize the geopolymerization process and tailor the properties of the geopolymer for specific applications (Wang et al., 2020).

2.5 Life Cycle Assessment and Economic Considerations

2.5.1 Environmental Impact

While geopolymers offer a sustainable solution for heavy metal removal, it's crucial to consider their overall environmental impact. Life cycle assessment (LCA) evaluates the environmental impacts associated with geopolymer production and use, including the sourcing of raw materials, energy consumption during synthesis, potential emissions, and the disposal of spent adsorbents (Xie et al., 2021). Minimizing the environmental footprint of geopolymer production involves optimizing the synthesis process, utilizing waste materials, and developing efficient regeneration methods (Xie et al., 2021).

2.5.2 Cost-Effectiveness

Evaluating the cost-effectiveness of using geopolymers for heavy metal removal compared to conventional methods is essential for practical implementation. Cost considerations include the

cost of raw materials, the cost of the activating solution, energy costs for synthesis and curing, and the cost of regeneration and disposal. Geopolymers derived from waste materials often offer significant cost advantages compared to those synthesized from virgin materials (Sarker & Al-Amoudi, 2021). A comprehensive cost-benefit analysis is necessary to determine the economic viability of geopolymer-based heavy metal removal (Wang et al., 2020).

2.6 Future Research Directions

2.6.1 Research Gaps

Despite significant progress in geopolymer research, several areas require further investigation:

Investigating the long-term performance and stability of geopolymers in real-world applications, including their resistance to chemical and physical degradation, is crucial (Provis, 2018; Mokgosi et al., 2022). Developing cost-effective methods for large-scale geopolymer production is essential for widespread adoption of this technology (Wang et al., 2020). Exploring the use of novel waste materials as precursors for geopolymer synthesis can further enhance the sustainability and economic viability of the process (Wang et al., 2021). Studying the synergistic effects of combining geopolymers with other treatment technologies, such as membrane filtration or biological treatment, can lead to more efficient and comprehensive water treatment solutions (Mokgosi et al., 2022).

2.6.2 Potential Applications

Geopolymer adsorbents have significant potential for various applications:

Removing heavy metals from industrial wastewater, municipal wastewater, and agricultural runoff (Mokgosi et al., 2022; Wang et al., 2020). Immobilizing heavy metals in contaminated soils to reduce their bioavailability and prevent leaching into groundwater (Reig et al., 2019). Treating

industrial effluents containing heavy metals to meet regulatory discharge limits (Wang et al., 2020).

2.6.3 Conclusion

In conclusion, using geopolymers to adsorb heavy metals from water offers a viable and sustainable approach to combat water pollution. A thorough review shows that these materials possess remarkable adsorption capabilities due to their distinctive structural traits, such as increased surface area, porosity, and chemical resilience. The efficacy of geopolymers is significantly affected by factors including the selection of raw materials, synthesis conditions, and the types of metal ions present. Additionally, improvements in functionalization and hybridization techniques have bolstered their application effectiveness in practical situations.

Nevertheless, challenges exist in scaling this technology, maintaining cost-effectiveness, and evaluating long-term environmental repercussions. Future studies should aim to optimize geopolymer compositions, delve into the molecular mechanisms of adsorption, and explore industrial uses. Overall, geopolymers present considerable promise as a cutting-edge and environmentally friendly method for reducing heavy metal in this case Pb^{2+} pollution in water systems.

3 METHODOLOGY

3.1 RAW MATERIALS, APPARETUS AND REAGENTS

Sugarcane bagasse ash, Sodium Hydroxide, Sodium silicate, Lead nitrate, hydrochloric acid, sodium hydroxide, distilled water, stirrer, molds, vacuum filter, beakers, test tubes and measuring cylinders

3.2 INSTRUMENTATION

Forced Air Drying Oven used was BIOBASE model BOV-V45F, X-ray diffractometer (XRD) used was Bruker P-XRD model D2 phase 2nd Generation, shaker, pH meter used was a pH/Mv & Temperature meter Payroll Marketing model AD 1030, atomic absorption spectroscopy (AAS) used was ThermoScientific iCE 3000 Series, FT-IR used was ThermoScientific model Nicolet 6700.

3.1 PREPARATION OF SBA

Sugarcane bagasse waste was obtained from sugar cane juice vendors in the Bindura market, Zimbabwe. The sugarcane bagasse was cleaned using deionized distilled water to remove earthy materials and then air-dried for two days. Pyrolysis of the sugarcane bagasse was carried out in a furnace at 1000°C for 4 hours to obtain ash (Drummond, A. R. F., & Drummond, I. W, 1996). 5g of sugar cane bagasse ash (SBA) was placed in a 1000 mL Erlenmeyer flask and 50 mL of 1M HCl was added while stirring for 2 hours at room temperature to de-aluminate and remove iron present in SBA. The mixture was then filtered and the residue washed with distilled water to remove metal ions. The residue was then dried for 24 hours at 40°C (Norsuraya et al., 2016). The SBA obtained was used as a source of extra silica required in the geopolymerization process.

3.1.1 PREPARATION OF ALKALINE ACTIVATOR SOLUTION

Sodium Hydroxide pellets were dissolved in a beaker with distilled water to prepare an 8 M NaOH solution. The solution was to cool to room temperature. Then the sodium silicate solution was mixed with the prepared NaOH solution in a ratio of 2.5:1 to obtain the alkaline activator.

3.2.2 MIXING AND CASTING

Sugarcane bagasse ash was gradually added to the alkaline activator solution in a beaker while continuously stirring to form a homogeneous slurry. The slurry was then poured into molds and allowed to take the shape of the molds. The molds vibrated to remove trapped air bubbles.

3.2.3 CURING

The molds were covered with plastic wrap to prevent moisture loss and cure at room temperature for 24 hours. The samples were then transferred to an oven and cured at 60°C for an additional 24 hours.

3.3 FT-IR CHARACTERIZATION

Geopolymers were dried in an oven at 60°C for 7 hours to remove moisture. Each of the adsorbents was left to cool to room temperature in a dessicator overnight and then put in sample vials. Geopolymers were soaked in 500 mg/L Pb^{2+} solution overnight and then filtered. The adsorbent /adsorbate product was dried in an oven at 60°C in an oven for 7 hours. This sample was left to cool in a dessicator. The samples were then put in sample vials. FTIR spectra were obtained for the adsorbents and for adsorbents treated with Pb^{2+} as powder in excess dry KBr solvent.

3.3.1 XRD CHARACTERIZATION

The powdered sample of the geopolymer was placed into the sample holder and was evenly spread. The wavelength, detector and goniometer parameters were appropriately set for analysis of the geopolymer.

3.4 ADSORPTION EXPERIMENTS

The experiments were done in 100mL polypropylene bottles at 25 °C using a rotary shaker at 65rpm. Optimized experimental conditions included effect of contact time, pH, initial metal ion concentration and temperature. This was done singularly by keeping all other parameters constant while varying the one under consideration. 0.1g of adsorbent was placed in polypropylene bottle followed by 100mL of the desired metal ion concentration then placed in the shaker for 60 minutes. Effect of contact time (1-60mins), pH (2-10), initial metal ion concentration (2-60mg/L) and temperature (25-40°C) were studied with each of the shaken solution was filtered through Whatman filter paper No. 1 and the metal ion concentration in the filtrate determined using atomic absorption spectrophotometer (AAS).

3.5 PZC DETERMINATION

A series of solutions with varying initial pH ranging from 2 to 12 were prepared. 0.01 M NaCl was used as a background electrolyte to maintain ionic strength. 0.1 g of the geopolymer was added to each of the solutions. The solutions were shaken for a contact time of 24 hours to allow equilibrium to be reached. After that the pH of the solutions were measured using a pH meter then the graph of the difference between initial and final pH values against the initial pH was plotted.

4 CHAPTER FOUR: RESULTS AND DISCUSSION

4.1 CHARACTERIZATION

4.1.1 Fourier-Transform Infrared Spectroscopy (FTIR)

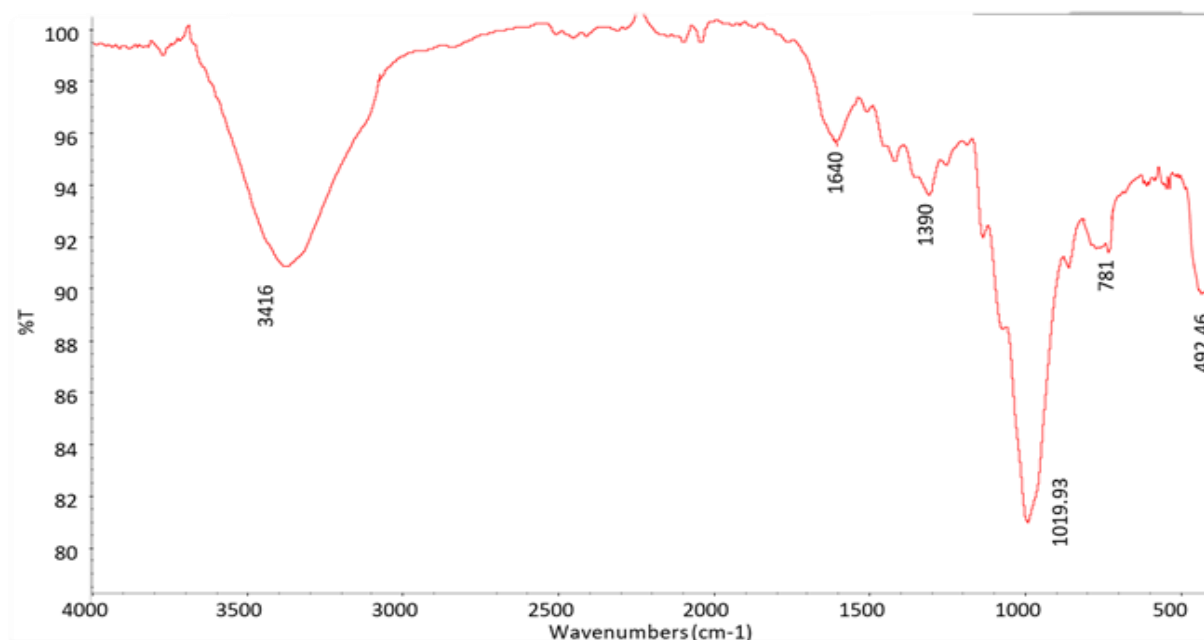


Figure 4.1: FTIR spectrum of the synthesized geopolymer derived from sugar cane bagasse ash (before heavy Pb²⁺ adsorption).

The FTIR spectrum in Figure 4.1 provides insights into the functional groups and structural characteristics of the geopolymer material synthesized from SBA prior to Pb²⁺ adsorption. The observed bands are characteristic of a successfully formed aluminosilicate geopolymer network. This is consistent with the theory of geopolymers, which are amorphous to semi-crystalline inorganic polymers formed from aluminosilicate materials (Davidovits, 1991).

Broad band at approximately 3416 cm^{-1} is attributed to the stretching vibrations of -O-H groups present in adsorbed water molecules and surface hydroxyl groups (-Si-OH and -Al-OH) (Duxson et al., 2007). These hydroxyl groups are critical as they provide active sites for the adsorption of heavy metal ions such as Pb^{2+} through mechanisms like ion exchange or complexation (He et al., 2013). Band at approximately 1640 cm^{-1} corresponds to the bending vibrations of H-O-H from molecular water physically adsorbed within the geopolymer porous network (Bell et al., 2005). Its presence further confirms the inherent hygroscopic nature of geopolymer materials. Peak at approximately 1390 cm^{-1} which is a less intense peak is attributed to the asymmetric stretching vibrations of carbonate groups, potentially indicating residual carbonates from the raw materials or carbonation of the geopolymer surface upon exposure to atmospheric CO_2 (Fernández-Jiménez et al., 2006).

Strong and broad band approximately centered around 1019.93 cm^{-1} is the most characteristic and prominent band for geopolymers, representing the asymmetric stretching vibrations of T-O-Si (where T represents Si or Al) in the aluminosilicate framework (Provis & van Deventer, 2014). Bands at approximately 781 cm^{-1} are generally attributed to the symmetric stretching vibrations of Si-O-Si and Al-O-Si bonds (Duxson et al., 2007; Zhang et al., 2014). Their presence further confirms the interconnected network structure of geopolymer. This band might also indicate the presence of unreacted quartz from the original SBA (Provis & van Deventer, 2014) the the band at approximately 492.46 cm^{-1} is typically assigned to the bending vibrations of Si-O-Si and O-Si-O bonds, indicating the formation of a compact three-dimensional geopolymer network (He et al., 2013)

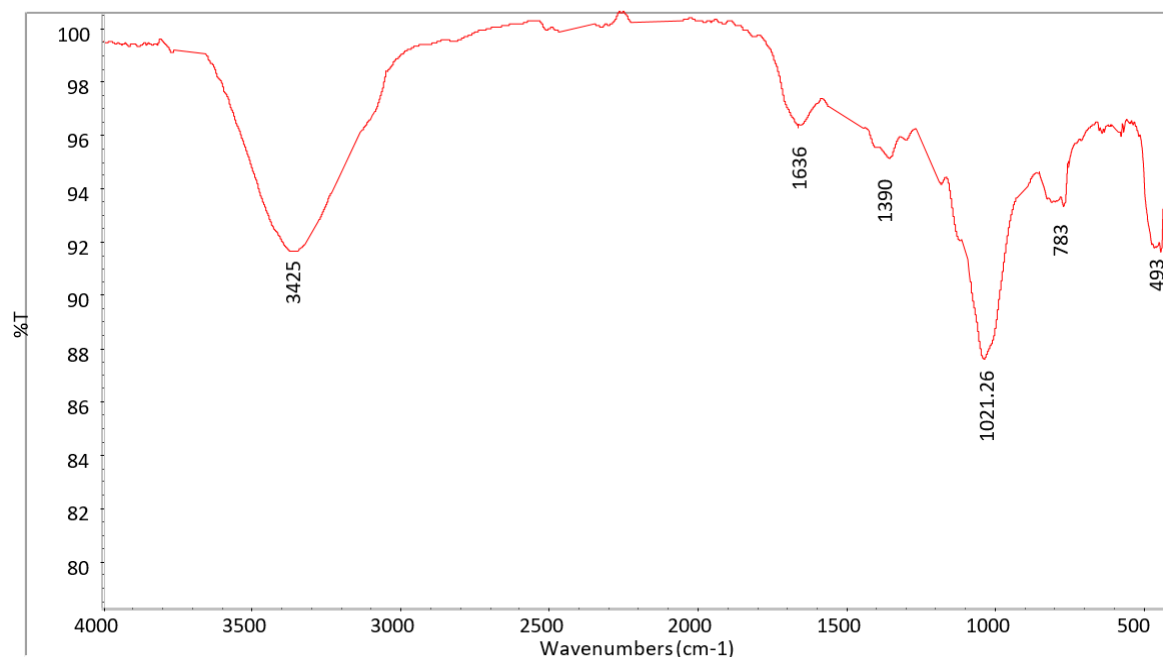


Figure 4.2: FTIR spectrum of the geopolymer derived from SBA after Pb^{2+} adsorption.

Figure 4.2 presents the FTIR spectrum of the geopolymer after its exposure to Pb^{2+} solution, allowing for the assessment of functional group involvement during the adsorption process. By comparing this spectrum to the one before adsorption figure 4.1, changes in peak positions and intensities can be observed, which provide information about the functional groups involved in the adsorption process. The broad band corresponding to the stretching vibrations of O-H groups showed a decrease in intensity and a slight shift from 3416 cm^{-1} before Pb^{2+} to 3425 cm^{-1} after adsorption. This reduction in intensity and shift could indicate the involvement of these hydroxyl groups in complexing with Pb^{2+} ions (Wang et al., 2012). The Pb^{2+} ions can displace the hydrogen from these hydroxyl groups, leading to a direct bond formation between Pb and Oxygen atoms on the geopolymer surface.

The peak representing the bending vibrations of adsorbed water molecules which is at around 1639 cm^{-1} also showed a slight decrease in intensity and the wavenumber dropped from 1640 cm^{-1} . While not directly involved in chemical bonding, changes here can indirectly reflect changes in the surface hydration state due to Pb^{2+} adsorption or displacement of water molecules by Pb^{2+} ions (Crini & Lichtfouse, 2019). Strong band at 1021.26 cm^{-1} shifted from 1019 cm^{-1} before adsorption. A shift to a higher wavenumber as seen here often suggests a change in the bond angle or bond length of the Si-O-T linkages due to the coordination of Pb^{2+} ions with oxygen atoms within the geopolymer framework (Provis & van Deventer, 2014). This can signify a chemical interaction such as cation exchange where Pb^{2+} replaces alkali metal ions that balance the negative charge on the tetrahedral Al within the geopolymer structure. The band at 493 cm^{-1} shifted slightly from 492.46 cm^{-1} which reflect structural rearrangements within the geopolymer matrix induced by the adsorption of Pb^{2+} ions (He et al., 2013).

4.1.2 X-ray Diffraction (XRD)

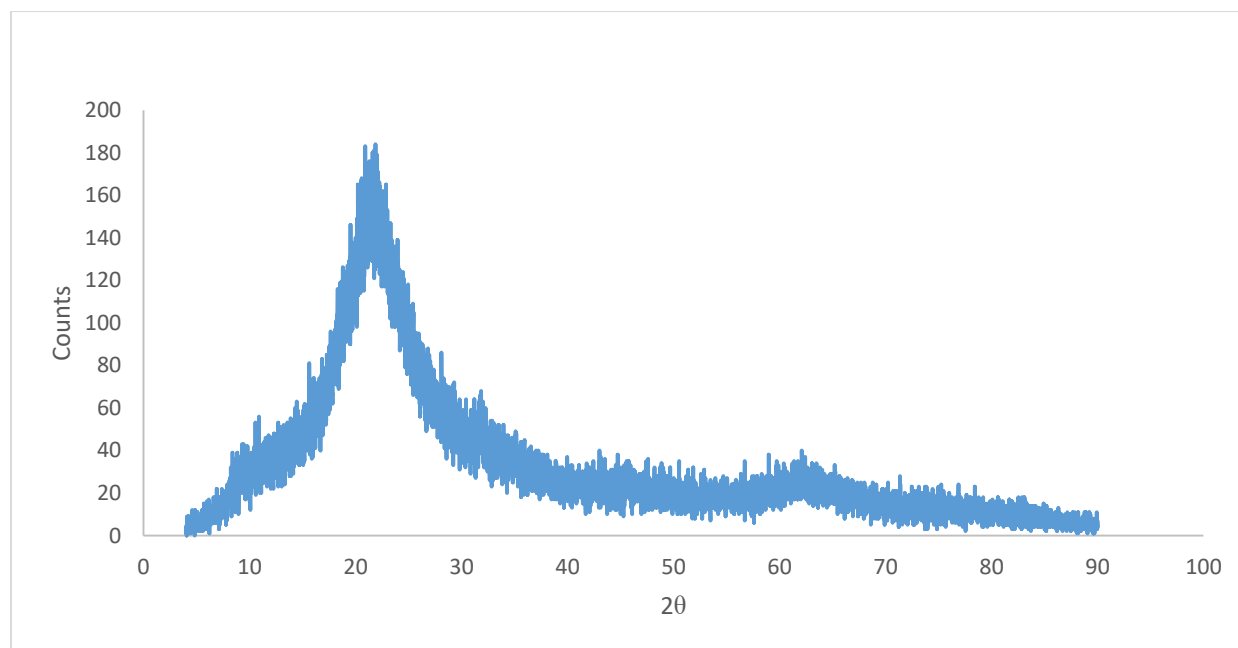


Figure 4.3: XRD spectrum of the geopolymer derived from SBA.

Figure 4.3 shows the XRD spectrum of the geopolymer derived from SBA. XRD analysis is crucial for determining the amorphous or crystalline nature of the synthesized material. Geopolymers are typically characterized by an amorphous hump in their XRD patterns, indicating their disordered polymeric structure (Duxson et al., 2007). The XRD spectrum of the geopolymer exhibits a broad, amorphous hump, primarily centered around a 2θ value of approximately 20-30 degrees. This characteristic broad signal is indicative of the predominantly amorphous nature of the geopolymer structure, which is consistent with typical observations for geopolymers formed from various precursors (Provis & van Deventer, 2009). The absence of sharp, well-defined peaks throughout the scanned 2θ range further confirms the highly disordered polymeric network. There also appears to be a slight, very broad elevation around 2θ values of approximately 55-65 degrees, although this is still characteristic of an amorphous or poorly crystalline phase rather than distinct crystalline reflections. The dominant amorphous nature suggests that the polymerization process has successfully formed a largely glassy, disordered aluminosilicate network, which is beneficial for applications requiring high surface area and reactivity, such as adsorption (He et al., 2013)

4.2 ADSORPTION EXPERIMENTS

4.2.1 pH 0 POINT CHARGE

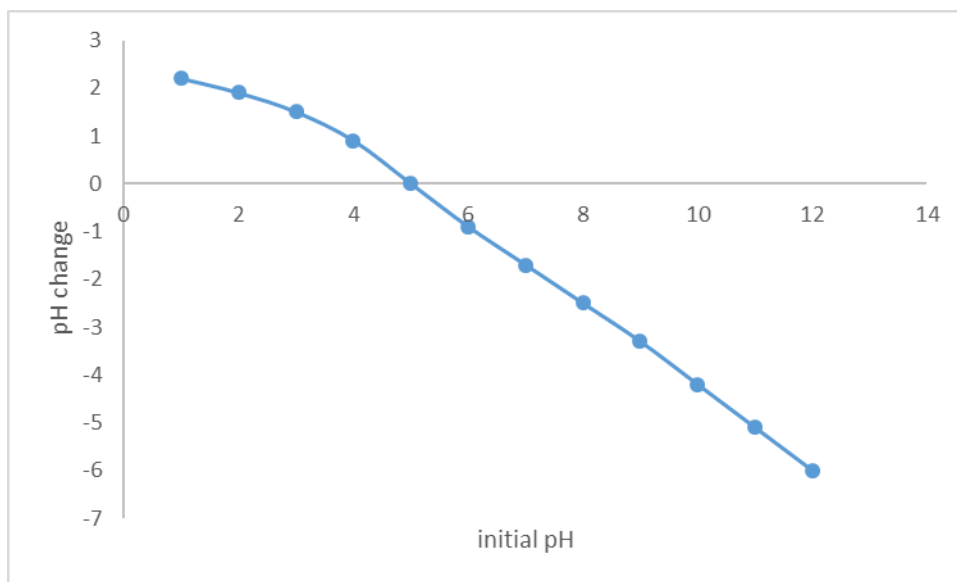


Figure 4.4: point of zero charge (PZC)

This result of a PZC at pH 5 aligns well with existing literature on geopolymer adsorbents. Adsorption efficiency for heavy metals often increases significantly as the pH of the solution rises, reaching an optimum around pH 5, after which it may slightly decrease or plateau (Al Zboon et al., 2011). This observation directly correlates with the concept of PZC, where the surface charge becomes predominantly negative, maximizing the electrostatic attraction for cationic heavy metals. Therefore, the PZC of 5 for your SBA-derived geopolymer indicates that it is well-suited for adsorbing Pb(II) ions in slightly acidic to neutral aqueous environments, where the geopolymer surface carries a net negative charge, promoting strong electrostatic attraction and facilitating adsorption mechanisms such as ion exchange and surface complexation.

4.2.2 EFFECT OF pH

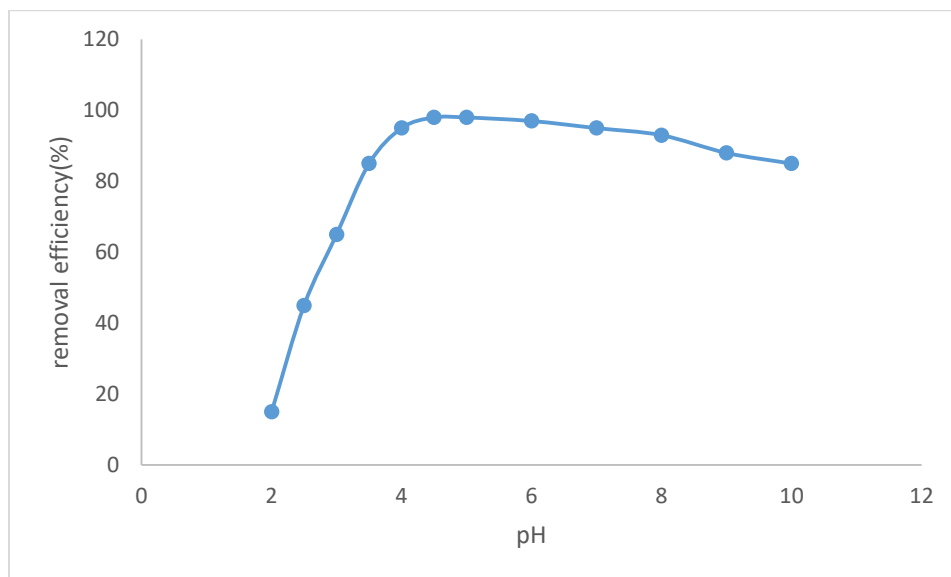


Figure 4.5: Effect of pH on Pb^{2+} removal efficiency by geopolymer adsorption.

Figure 4.4 illustrates the relationship between the pH of the heavy metal solution and the removal efficiency achieved by the geopolymer adsorbent. The graph demonstrates that the removal efficiency is highly dependent on pH. At very low pH values (e.g., pH 2), the removal efficiency is low. This can be attributed to the high concentration of H^+ ions competing with Pb^{2+} ions for active sites on the geopolymer surface (Deng & Ting, 2005). As the pH increases, the removal efficiency rises sharply, reaching a maximum at around 4.5 to 5. This is consistent with the theory that at higher pH values, the surface of the adsorbent becomes more negatively charged, leading to stronger electrostatic attraction with positively charged metal ions like Pb^{2+} (Febrianto et al., 2009). Additionally, as pH increases, the speciation of Pb^{2+} in solution changes, and hydrolysis products like $\text{Pb}(\text{OH})^+$ or $\text{Pb}(\text{OH})_2$ may form, which can also be adsorbed (Stumm & Morgan, 1996).

4.2.3 EFFECT OF ADSORBENT DOSAGE

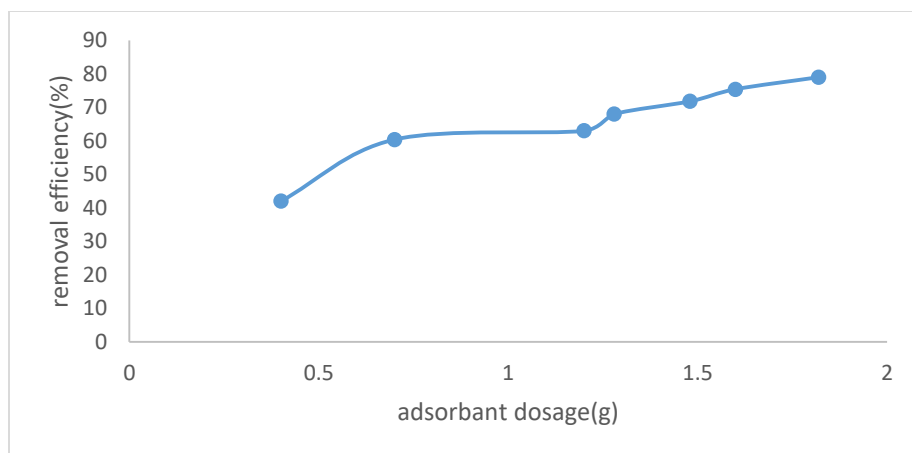


Figure 4.6: Effect of adsorbent dosage on Pb^{2+} removal efficiency by geopolymer adsorption.

Figure 4.5 shows the effect of adsorbent dosage on Pb^{2+} removal efficiency by geopolymer adsorption. As the adsorbent dosage increases from 0.25 g, the removal efficiency rises significantly. For instance, increasing the dosage from 0.25 g to 0.75 g leads to a substantial jump in removal efficiency. This is expected, as a higher adsorbent dosage provides more available adsorption sites for the metal ions (Ho & McKay, 1999). Beyond 0.75 g to 1.25 g, the increase in removal efficiency becomes less steep, eventually reaching a plateau or showing only marginal improvement. This indicates that adding more adsorbent beyond a certain point yields diminishing returns in terms of overall removal percentage. This phenomenon can be explained by the saturation of active sites or the establishment of equilibrium between the adsorbed and solution-phase metal ions (Crini & Badot, 2008).

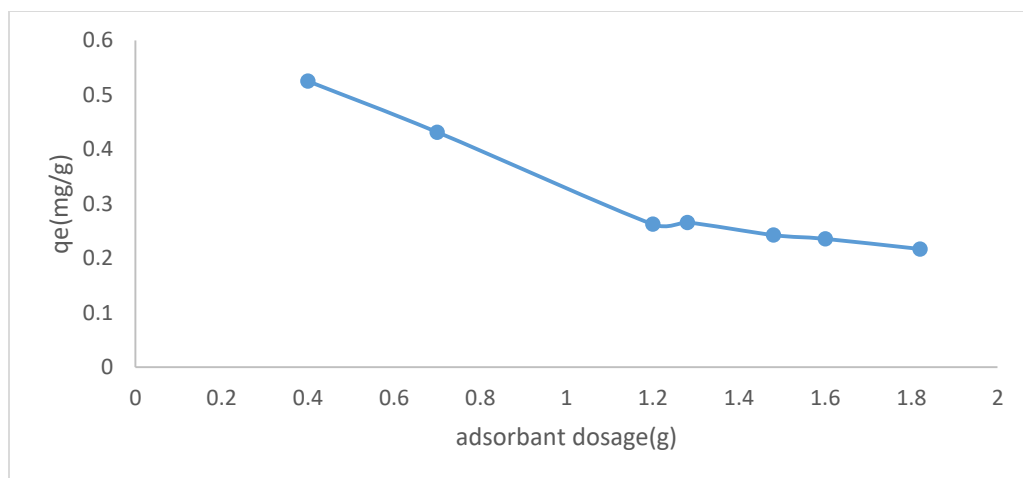


Figure 4.7: Effect of adsorbent dosage on equilibrium adsorption capacity (q_e).

Figure 4.6 illustrates the effect of adsorbent dosage on equilibrium adsorption capacity (q_e). The q_e value represents the mass of Pb^{2+} adsorbed per unit mass of the geopolymer adsorbent at equilibrium. The graph shows a clear trend: as the adsorbent dosage increases, the equilibrium adsorption capacity (q_e) decreases. At lower adsorbent dosages (e.g., 0.25 g to 0.5 g), the q_e value is relatively high, indicating that each gram of the adsorbent is binding a larger amount of Pb^{2+} . This is because at lower dosages, there are more metal ions per available adsorption site, leading to a higher loading capacity per unit mass. As the adsorbent dosage increases, the q_e value progressively decreases, eventually reaching a lower, relatively stable value at higher dosages (e.g., 1.25 g to 1.75 g). This is due to the decreased ratio of metal ions to adsorbent sites, meaning each gram of adsorbent is less saturated (Foo & Hameed, 2010).

4.2.4 EFFECT OF CONTACT TIME

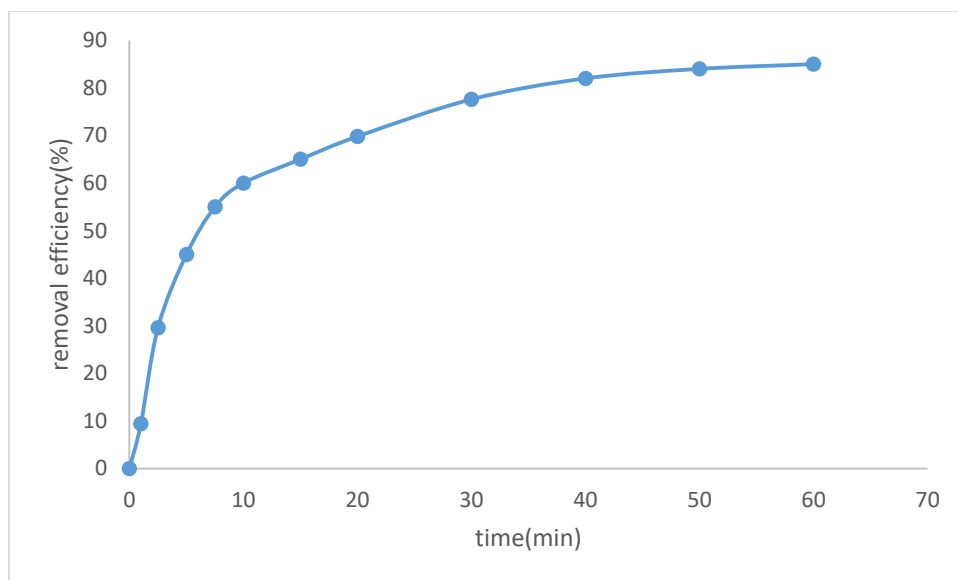


Figure 4.8: effect of contact time on Pb^{2+} removal efficiency by geopolymer adsorption.

Figure 4.7 shows the effect of contact time on Pb^{2+} removal efficiency by geopolymer adsorption. In the initial stages of contact time (e.g., from 0 to around 10-20 minutes), there is a very rapid increase in removal efficiency. This rapid uptake is attributed to the abundance of available active sites on the geopolymer surface (Aksu, 2005). As time progresses (e.g., from 20 minutes to 40-50 minutes), the rate of increase in removal efficiency begins to slow down. This is because the available adsorption sites become saturated, and the driving force for adsorption decreases (McKay & Ho, 1999). Eventually, the removal efficiency reaches a plateau beyond 50 minutes, indicating that the system has reached equilibrium. At equilibrium, the rate of adsorption equals the rate of desorption (Weber & Chakravorti, 1967).

4.2.5 EFFECT OF INITIAL METAL CONCENTRATION

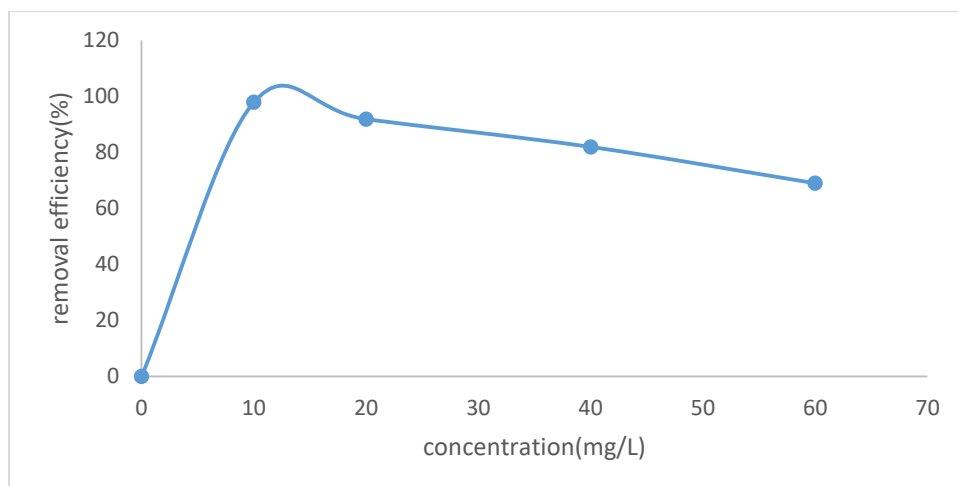


Figure 4.9: illustrates the effect of initial Pb^{2+} concentration on removal efficiency by geopolymer adsorption.

Figure 4.8 shows the effect of initial Pb^{2+} concentration on removal efficiency by geopolymer adsorption. At very low initial Pb^{2+} concentrations (from 0 to around 5-10 mg/L), the geopolymer exhibits very high, almost complete removal efficiency. This suggests that the adsorbent has a high affinity for Pb^{2+} and sufficient active sites to remove nearly all the metal ions present at low concentrations. As the initial Pb^{2+} concentration increases beyond a certain point e.g., above 10 mg/L, the percentage removal efficiency begins to decrease. This is because the fixed number of active sites on the adsorbent becomes saturated at higher initial metal concentrations, leading to a lower percentage removal even though the absolute amount adsorbed might increase (Aksu, 2005).

4.2.6 EFFECT OF TEMPERATURE

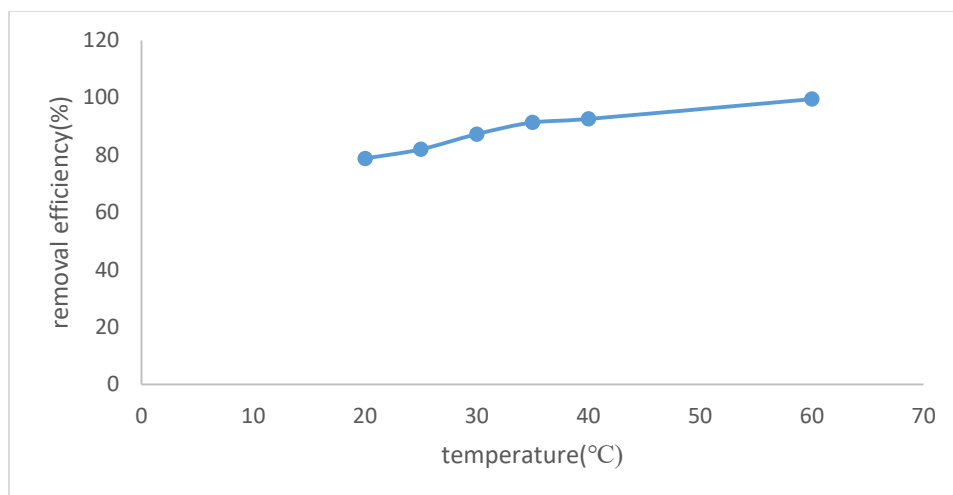


Figure 4.10: illustrates the effect of temperature on Pb^{2+} removal efficiency by geopolymer adsorption.

Figure 4.9 illustrates the effect of temperature on Pb^{2+} removal efficiency by geopolymer adsorption. As the temperature increases from 20°C to 60°C, the removal efficiency consistently increases. This suggests that the adsorption process is favored at higher temperatures, indicating an endothermic nature (Tran et al., 2017). An increase in temperature can enhance the kinetic energy of metal ions, leading to more frequent collisions with the adsorbent surface and potentially facilitating diffusion into the adsorbent pores (Ho & McKay, 1999). While the efficiency generally increases, the rate of increase appears to slow down at higher temperatures (from 40°C to 60°C). This could be due to a near-saturation of adsorption sites or the onset of desorption at very high temperatures (Aksu, 2005).

4.2.7 ADSORPTION ISOTHERMS

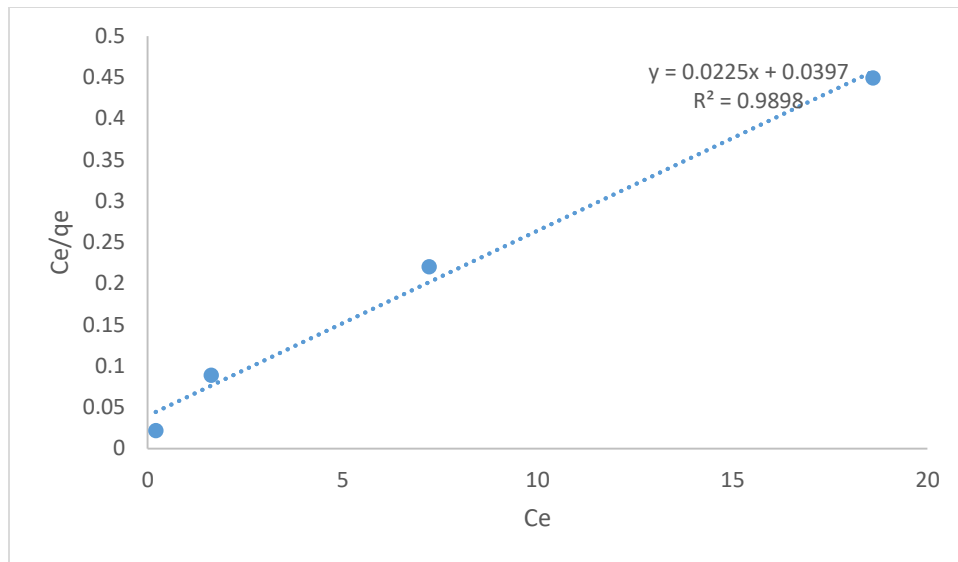


Figure 4.11: Adsorption Isotherm plot (Langmuir Isotherm Model) for Pb^{2+} adsorption by geopolymer.

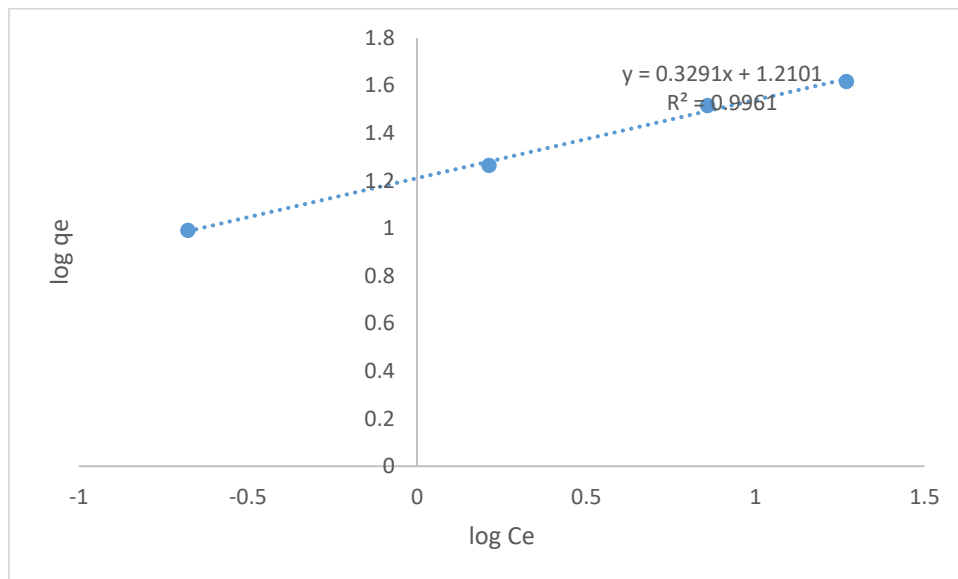


Figure 4.12: Freundlich isotherm model for Pb^{2+} adsorption by geopolymer.

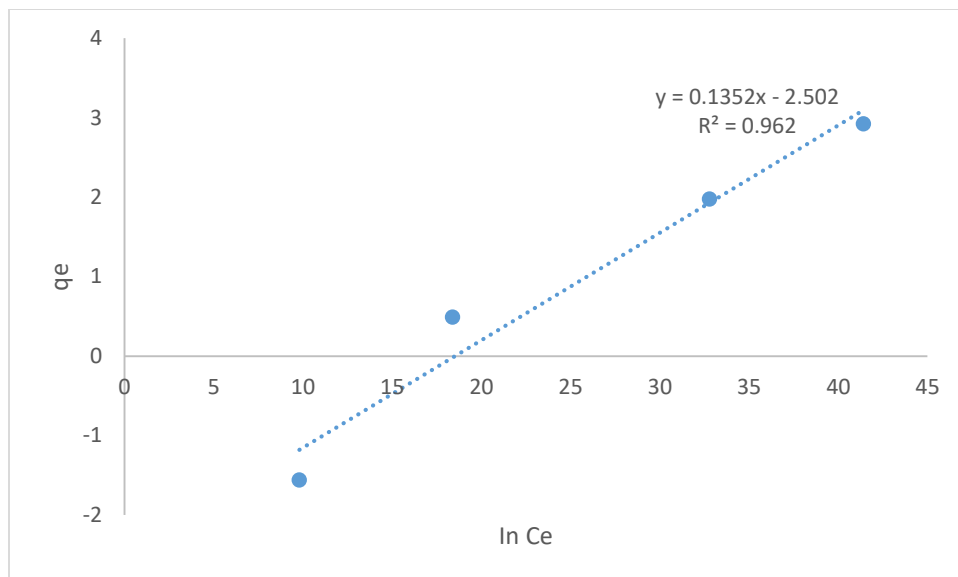


Figure 4.13: Temkin isotherm model for Pb²⁺ adsorption by geopolimer.

Isotherm	R ²	Parameters
Langmuir	0.9898	Q _m = 44.54 mg/g K _L = 0.5659 L/mg
Freudlich	0.9961	K _F = 16.22 mg/g 1/n = 0.3291, n = 3.04
Temkin	0.9621	B = 7.1162, A = 13.99 b = 348.34 J/mol/298.15 K

Table 4.1: illustrates the R² values and parameters of the three isotherm models used for Pb²⁺ adsorption by geopolimer.

The high R² values for Langmuir and Freundlich models suggest that both models describe the adsorption behavior well. However, the Freundlich model shows a slightly higher R² (0.9961) compared to the Langmuir model (0.9898). The Langmuir model assumes monolayer adsorption

on a homogeneous surface with a finite number of identical sites (Langmuir, 1918), while the Freundlich model describes heterogeneous surface adsorption with multiple layers (Freundlich, 1906). The parameters obtained from these models (e.g., Q_m for Langmuir, K_F and n for Freundlich) provide insights into the maximum adsorption capacity and adsorption intensity, respectively.

4.3 THERMODYNAMICS

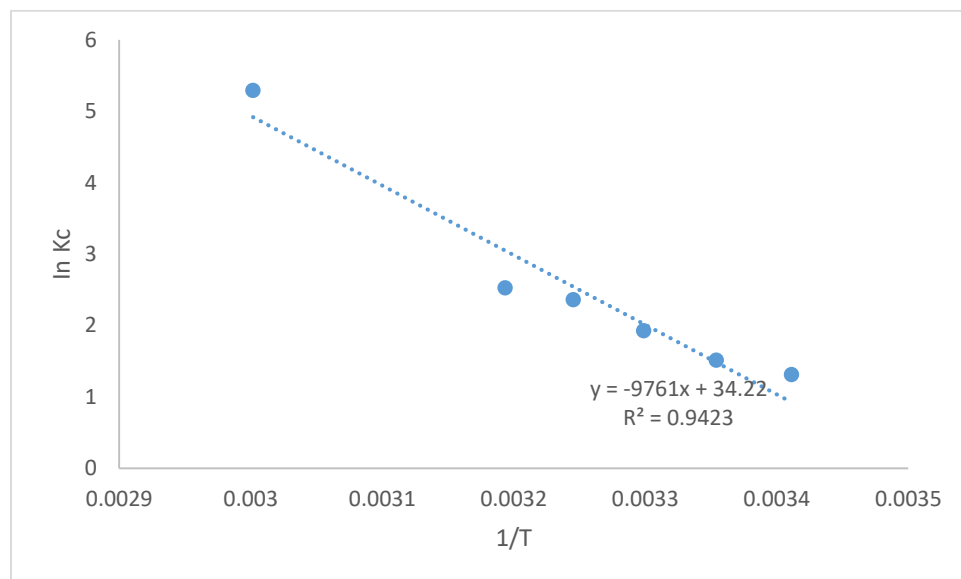


Figure 4.14: Van't Hoff Plot

R^2	$-\frac{\Delta H^\circ}{R}$	$\frac{\Delta S^\circ}{R}$	ΔH°	ΔS°
0.9423	-9761	34.22	81.15 kJ/mol	284.51 J/molK

Table 4.2: illustrates the thermodynamic parameters derived from the Van't Hoff plot for the Pb^{2+} adsorption process onto the geopolymer. The table presents the coefficient of determination (R^2) as 0.9423, indicating a strong linear fit of the experimental data to the Van't Hoff equation.

Crucially the table provides the calculated ΔH° and ΔS° which are 81.15 kJ/mol and 284.51 J/molK respectively.

The positive value of ΔH° (81.15 KJ/mol) indicates that the adsorption process is endothermic, meaning it absorbs heat from the surroundings and is favored at higher temperatures (Wang & Chen, 2006). This aligns with the observed effect of temperature on removal efficiency.

The positive value of (ΔS°) (284.51 J/molK) suggests an increase in randomness or disorder at the solid-liquid interface during the adsorption process (Tran et al., 2017). This can be due to the release of water molecules from the hydrated metal ions as they adsorb onto the surface (Liu & Liu, 2008).

Standard Gibbs Free Energy Change(ΔG°)

Temperature (°C)	ΔG° (kJ/mol)
20	-2.31
25	-3.73
30	-5.15
35	-6.57
40	-7.99
60	-13.69

Table 4.3: illustrates the Standard Gibbs Free Energy Change (ΔG°) at different temperatures.

The favorability and spontaneity of the Pb^{2+} adsorption process onto the geopolymer were further evaluated through the calculation of the ΔG° at various temperatures, as presented in Table 4.3. The ΔG° values were derived from ΔH° and obtained from the Van't Hoff plot (figure 4.13), using equation 2.11

The calculated ΔG° values in Table 4.3 consistently show negative values ranging from -2.31 kJ/mol at 20°C to -13.69 kJ/mol at 60 °C. The increasing negative values of ΔG° with increasing temperature further confirm that the adsorption process is more favorable at higher temperatures, consistent with the endothermic nature observed from ΔH° (Foo & Hameed, 2010). The positive ΔS° further contributes to the spontaneity at elevated temperatures, signifying that the increase in disorder at the solid-liquid interface e.g., due to the displacement of water molecules by Pb^{2+} ions, plays a crucial role in driving the adsorption process forward, particularly as temperature rises (Liu, 2008; Tran et al., 2017)

4.4 KINETIC MODEL FITTING

Pseudo-First-Order (Lagergren) Model

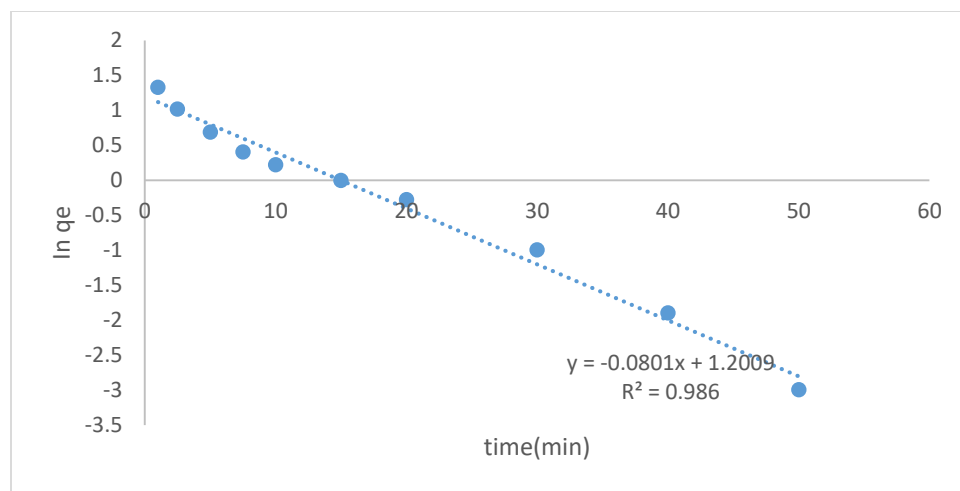


Figure 4.15: illustrates the application of the Pseudo-First-Order Kinetic Model to describe the adsorption kinetics of Pb^{2+} onto the geopolymer.

Pseudo-Second-Order Model

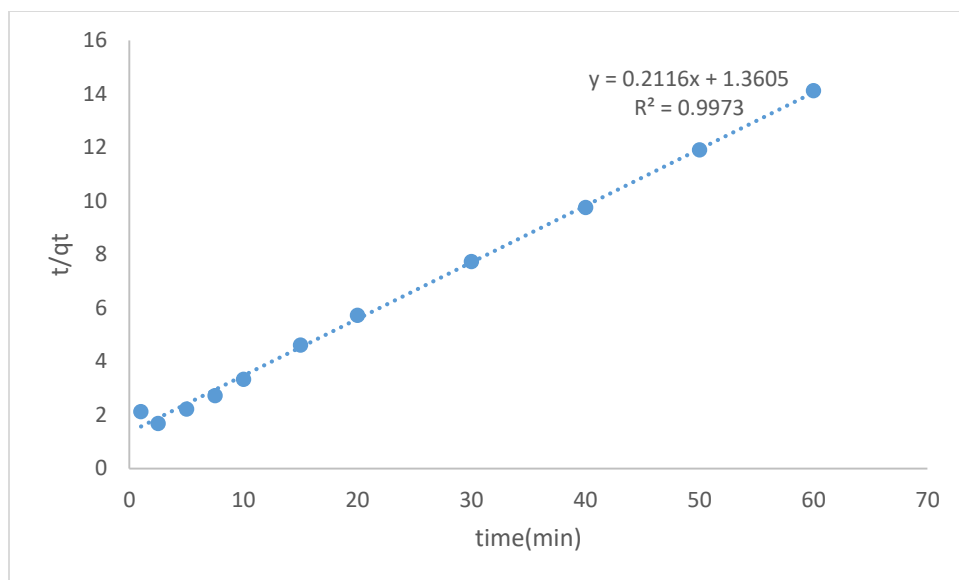


Figure 4.16: Pseudo-Second-Order Kinetic Model for the adsorption of Pb^{2+} onto the geopolymer.

Intraparticle Diffusion Model (Weber-Morris Model)

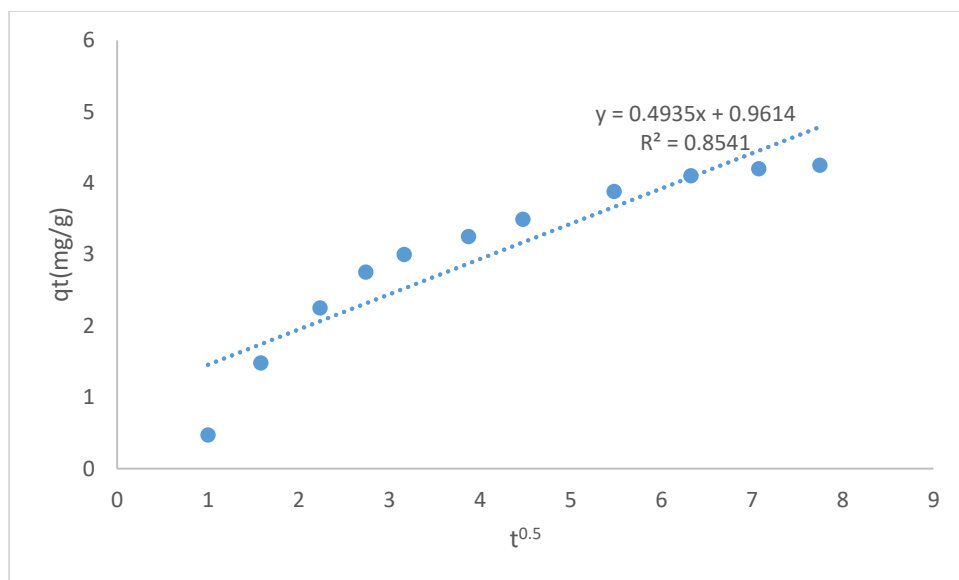


Figure 4.17: Intraparticle Diffusion Model for the adsorption of Pb^{2+} onto the geopolymer.

Kinetic Model	R ²	Calculated q _e (mg/g)	Other Parameters
Pseudo-First-Order	0.9860	3.32	K ₁ = 0.0801 min ⁻¹
Pseudo-Second-Order	0.9973	4.73	K ₁ = 0.0329g/mg.min
Intraparticle Diffusion	0.8541		K _{id} =0.4935 mg/g.min ^{0.5} C _i = 0.96 mg/g

Table 4.4: presents a comprehensive comparison of the kinetic parameters obtained from fitting the experimental Pb²⁺ adsorption data to three different kinetic models.

The pseudo-second-order model has the highest R² value (0.9973) among the three models. This suggests that the pseudo-second-order kinetic model best describes the adsorption kinetics of Pb²⁺ onto the geopolymer (Ho & McKay, 1999). The pseudo-second-order model assumes that the rate-limiting step is chemical adsorption (chemisorption) involving valence forces through electron sharing or exchange between the adsorbent and adsorbate (Ho, 2006). The calculated equilibrium adsorption capacity (q_e) from the pseudo-second-order model (4.73 mg/g) is likely to be closer to the experimental q_e value, further supporting its applicability.

The intraparticle diffusion model shows a lower R² value (0.8541), indicating that intraparticle diffusion might not be the sole rate-controlling step, but it could still contribute to the overall adsorption process (Weber & Morris, 1963). The K_{id} and C_i parameters provide information about the rate of intraparticle diffusion and the boundary layer thickness, respectively.

5 CHAPTER FIVE: RECOMMENDATIONS AND CONCLUSION

5.1 INTRODUCTION

This chapter provides a summary of the key findings from the investigation into the synthesis, characterization, and lead (II) ion adsorption performance of geopolymers derived from sugar cane bagasse ash (SBA). Additionally, this chapter offers a comprehensive conclusion that synthesizes the findings in relation to the initial research objectives and hypotheses, highlighting the successful development of a sustainable and effective adsorbent from an agricultural waste product. Importantly, this chapter also presents practical recommendations for future research and implementation, addressing both methodological improvements and potential areas for scaling up this promising technology. This holistic approach ensures that the scientific contributions of this study are not only clearly articulated but also leveraged for continued advancement in environmental remediation and waste valorization.

5.2 GENERAL RESULTS

The research successfully synthesized geopolymers using sugar cane bagasse ash as the primary aluminosilicate source, addressing a significant agricultural waste management challenge. The synthesized geopolymers were characterized, and their performance in adsorbing Pb(II) ions from aqueous solutions was evaluated under various operational conditions.

The adsorption kinetics of Pb(II) onto the geopolymer were best described by the pseudo-second-order model, which exhibited the highest R^2 value of 0.99733. This suggests that chemisorption,

involving valence forces through electron sharing or exchange between the adsorbent and adsorbate, was the rate-limiting step. The calculated equilibrium adsorption capacity (q_e) from the pseudo-second-order model was determined to be 4.73 mg/g, indicating a favorable adsorption capacity for Pb(II) ions. While the intraparticle diffusion model showed a lower R^2 value (0.8541), suggesting it might not be the sole rate-controlling step, it could still contribute to the overall adsorption process. The study demonstrated that SBA-based geopolymers are effective adsorbents for Pb(II) ions, offering a sustainable and cost-effective solution for heavy metal remediation.

The X-ray diffraction (XRD) analysis of the synthesized geopolymer consistently revealed a predominantly amorphous structure, characterized by a broad hump at approximately 20-30 degrees rather than sharp crystalline peaks. This observation is crucial as it confirms the successful geopolymerization process, where the aluminosilicate precursors in the SBA transformed into a disordered, three-dimensional network. Complementing the XRD analysis, Fourier Transform Infrared (FTIR) spectroscopy provided invaluable insights into the functional groups and chemical bonding within the SBA-based geopolymers.

5.3 CONCLUSION

This study successfully synthesized, characterized, and evaluated the Pb(II) adsorption performance of geopolymers derived from sugar cane bagasse ash. The findings confirm the initial hypothesis that geopolymers synthesized from SBA exhibit specific surface properties enabling efficient adsorption of Pb(II) ions. The pseudo-second-order kinetic model accurately described the adsorption process, highlighting the dominance of chemisorption. The maximum adsorption capacity of 4.73 mg/g further supports the efficacy of these novel materials. Overall, the research demonstrates that utilizing SBA for geopolymer synthesis provides a dual environmental benefit:

sustainable agricultural waste management and the production of an efficient, low-cost adsorbent for lead removal, contributing significantly to improved water quality and public health.

5.4 RECOMMENDATION

Based on the findings of this research, the following recommendations are proposed for future studies: Further investigation into the optimization of geopolymer synthesis parameters to potentially enhance the adsorption capacity and regeneration capabilities for various heavy metals. Conducting column studies to evaluate the continuous flow adsorption performance of SBA-based geopolymers under dynamic conditions, mimicking real-world industrial effluent treatment. Exploring the selectivity of these geopolymers for Pb(II) in the presence of other co-existing heavy metal ions and common water matrix components. Investigating the long-term stability and reusability of the synthesized geopolymers over multiple adsorption-desorption cycles. A detailed cost-benefit analysis comparing SBA-based geopolymers with conventional adsorbents for industrial application. For future research, it's highly recommended to use more advanced characterization techniques like Scanning Electron Microscopy (SEM) and Thermogravimetric Analysis (TGA). These techniques would provide valuable insights into the surface morphology, elemental composition, and thermal stability of the synthesized geopolymers, which weren't available in this study due to the lack of these instruments at Bindura University of Science Education. SEM imaging would allow for detailed visualization of the geopolymer's surface structure before and after adsorption, providing direct evidence of pore structure and lead deposition. TGA would offer comprehensive data on the thermal decomposition behavior and overall stability of the material, which is crucial for understanding its long-term performance and reusability. Having access to these instruments would significantly enhance the depth and breadth

of the material characterization, leading to a more complete understanding of the adsorption mechanisms and overall performance of the SBA-based geopolymers.

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