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Article *in* American Journal of Chemistry · April 2022

DOI: 10.5923/j.chemistry.20221202.02

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Geopolymers Filters from Calcined Clay and Sugarcane Bagasse Blended with Activated Carbon for Removal of Selected Heavy Metals from Wastewater

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Abstract The growth of industries and the exploitation of natural resources have led to increase of heavy metals concentrations in the earth's surface, atmosphere and water bodies. Heavy metals pose serious environmental and health effects in the society since they are toxic and non-biodegradable. Removing heavy metals from aqueous solutions especially wastewater has become an area of study in the recent past. Various methods like ion exchange, reverse osmosis, coagulation and adsorption among others have been put forth in the removal of heavy metal ions. Geopolymers are zeolite analogues and there is little information on their use as water filters in literature. Incorporation of geopolymers and activated carbon to form wastewater filters have not been studied. This study focused on fabricating geopolymer filters derived from calcined clay and sugarcane bagasse ash incorporated with activated carbon for removal of heavy metals from aqueous media. Characterization of the Geopolymer, and activated carbon were done using XRF, XRD and FT-IR. Column adsorption using geopolymer/activated carbon filters was employed with Pb (II) and Cu (II) as the test heavy metal ions while varying concentration, filter thickness, and pH. Halo broad peaks were observed in the diffraction patterns at angle 2-theta between 20°- 34° in both geopolymer and activated carbon an indication that they were amorphous. Geopolymer: activated carbon ratio of 2:1, filter thickness of 4cm and pH range of 4-6.5 gave removal percentage of above 85% for both heavy metal ions. The experimental data fitted best in Freundlich adsorption isotherm with correlation coefficient (R^2) of 0.854, 0.978 for Pb (II) ions, and Cu (II) respectively. Adsorption capacities of 0.028 mg/g and 0.015mg/g for Pb (II) ions, and Cu (II) ions were obtained respectively. The study shows that geopolymers/activated carbon filters have a potential of removing heavy metal ions from aqueous solution and hence can be applied for remediation of wastewater from industrial effluents.

Keywords Activated carbon, Characterization, Geopolymers Filter, Hheavy metals, Wastewater

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1. Introduction

Sources of heavy metal pollution are mining waste and industrial wastewater. When these waters are not taken care of, they lead to heavy metal pollution [1]. According to Ndeda and Manohar [2], the Nairobi dam is highly polluted with Pb(II), Cd(II), Cu(II), and Ni(II) ions hence the water is unsuitable for domestic and agricultural use. Muiruri, et al. [3], also concluded that there is bio-accumulation of heavy metals in fish at Athi-Galana, Sabaki tributaries in Kenya. Kaluli *et al.* [4] confirmed that irrigation of food crops with wastewater in Nairobi-Kenya increased the concentration of heavy metals such as lead and cadmium in food production to unsafe levels. Ravindra, et al. [5] clearly stated that the presence of heavy metals and their toxicity in the environment and human beings is a problem of global concern, and various ways of removing them should be studied. Adsorption of ingested lead may bring about chronic effects of lead poisoning hence causing constipation, colic, and anemia [6]. Extreme amounts of Cu (II) ions in the human body have been known to cause mucosal irritation, aquatic fauna, and central nervous system irritation followed by depression [5]. Geopolymers have been proven to be good adsorbents of heavy metal ions from solutions [7], [8]. Most researchers have used commercial raw materials in their geopolymerization process while Maingi et al. [9] used clay and rice husks ash but there is little information in the literature on the use of sugarcane bagasse ash. Maingi et al. [9] experienced clogging in his column adsorption and hence the need to remove clogging if the geopolymers have to be used for industrial wastewater management. This study aimed at developing a safe and easy way of removing heavy metals from an aqueous medium with minimal or no clogging. Therefore, the focus of this study was to fabricate

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Received: Mar. 1, 2022; Accepted: Mar. 30, 2022; Published: Apr. 15, 2022

geopolymer filters using calcined clay, sugarcane bagasse ash, and incorporating activated carbon to increase porosity and reduce clogging during the adsorption of heavy metal ions from an aqueous solution.

2. Material and Methods

2.1. Chemicals and Reagents

The chemicals used were of analytical grade and obtained from Kobian Kenya. They included $Pb(NO_3)_2$ and $Cu(NO_3)_2$ for preparing synthetic wastewater. HCl was used to de-aluminate and remove iron from the sugarcane bagasse (SBA), and adjust the pH during the adsorption process. NaOH was used as an alkali-activator during geopolymerization process. KBr was used in FT-IR for characterization. $ZnCl_2$ was used as an activator in the production of activated carbon.

2.2. Raw Materials

2.2.1. Clay as a Raw Material

Clay was obtained locally at a clay working site in Kikuyu, Kenya. The clay was washed with distilled water and dried for ten days. Calcination was carried out in a furnace (Eisklo 120) at 670℃ and then grounded to fine particles [10]. The powdered calcined clay obtained was used as a source of alumina and silica which are key ingredients for geopolymerization.

2.2.2. Sugarcane Bagasse Ash

Sugarcane bagasse waste was obtained from sugar cane juice vendors in the Githurai market, Kenya. The sugarcane bagasse was cleaned using deionized distilled water to remove earthy materials and then air-dried for one week. Pyrolysis of the sugarcane bagasse was carried out in a furnace at 1000℃ for 4 hours to obtain ash [11]. 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℃ [12]. The SBA obtained was used as a source of extra silica required in the geopolymerization process.

2.3. Synthesis of Geopolymer

8 M NaOH solutions were added to SBA in in the ratio of 5L: 1Kg and the mixture was stirred for 15 minutes. 50 g of the powdered calcined clay was then added to the mixture. The mixture was stirred for 5 hours using a magnetic stirrer [9]. The paste obtained was poured into ceramic crucibles and cured in an oven at 100℃ for 10 hours for polycondensation to occur [13]. The cured geopolymer paste was then removed from the mold, washed with distilled water to remove excess alkali, and placed in an oven to dry at 150℃ for 12 hours.

2.4. Preparation of Activated Carbon

1200 g of sawdust was properly washed to remove earthy materials and then air-dried for 5 days in the open to remove loosely bound water. The sawdust was then, placed in an oven and dried at 110℃ for 10 hours before crushing and grinding [14]. The impregnating ratio of sawdust and zinc chloride of 1:1 was used at a temperature of 600–800℃ for 3 hours to yield activated carbon with good porosity and surface area [15]. The activated char was leached with 1M HCl solution for 24 hours to remove excess activator, then washed thoroughly with distilled water, and dried at 80℃ for 6 hours [16].

2.5. Characterization of Geopolymer and Activated Carbon

The characterization was carried out using FT-IR (IR tracer) and XRD (D8 Advanced, BRUKER AXS). FT-IR was used for functional group analysis. A fine powdered sample was ground using a mortar and pestle. KBr was mixed with the powder at a ratio of 1: 20. The mixture was then compressed in a sample holder of the infrared machine and analyzed in varying wavelengths. XRD was used to identify the mineralogical composition of the geopolymer, phase compositions, and crystallinity hence the structural properties of the geopolymer using the model (Bruker AXS D8 Advance). Continuous 2-theta scans in the locked coupled mode were used for measurement with a tube of Cu-Ka radiation (lKa=1.5406Å) and a Lynx Eye position-sensitive detector.

2.6. Adsorption Experiments

2.6.1. Effects of the Ratio of Geopolymer to Activated Carbon on Heavy Metal Ions Removal

Geopolymer was mixed thoroughly with activated carbon in a ratio ranging from 2:1 to 1:2. The different ratios were used in fabricating column filters that were employed for column adsorption. 50 mL of 100 mg/L of Cu (II) and Pb (II) were added onto each column filter separately and allowed to seep through the column with aid of gravity. The adsorption experiments were carried out in triplicates. The concentration of residues metal ions in emerging solutions was determined using FASS.

2.6.2. Effects of Filter Thickness on Heavy Metal Ions Adsorption

The filter thickness was varied from 6 cm to 15 cm for the adsorption of the heavy metal ions. The study was done in triplicates. 50 mL of 100 mg/L of Cu (II) and Pb (II) were added onto each column filter and allowed to seep through the column with aid of gravity. The residual metal ions concentration in the filtrate was analyzed using FASS.

2.6.3. Effects of pH on Heavy Metal Ions Adsorption

Optimization of pH was done by varying the pH of the working solution of 100.mg/L of Pb (II) and Cu (II) from 2 to 6. The filtered solutions were analyzed for residual metal ions concentrations using FAAS at a wavelength of 283.3nm for Pb (II) ions, and 324.8nm for Cu (II) ions.

2.6.4. Effects of Initial Metal Ion Concentration on Adsorption

The effect of initial metal ion concentration on the efficiency of the geopolymer filters was studied using 50mL of each metal ion with concentrations varied from (15, 30, 75, 100, and 150) mg/L. The mixtures were passed through the geopolymer filters with aid of gravitational force. The filtered solution was analyzed for residual metal ions using FASS.

2.7. Adsorption Isotherms

Langmuir and Freundlich isotherms were used to determine the adsorption capacities of the geopolymer filters. Equation 1 shows the linearized Langmuir adsorption isotherm used [17].

$$\frac{Ce}{Qe} = \frac{1}{Qm\ Kl} + \frac{Ce}{Qm} \tag{1}$$

Where Q_e is the number of metal ions adsorbed by the geopolymer, C_e =Metal ion equilibrium concentration, K_L= Langmuir constant, Q_m= Maximum adsorption capacity of the adsorbent. A plot of vs C_e was used to plot the experimental data and determine the adsorption capacities of the geopolymer filters. The linearized Freundlich adsorption isotherm equation 2 was used to plot the experimental data.

$$\log a = \log k + \frac{1}{n} \log c \tag{2}$$

Where ‘a’ is the mass of the solute adsorbed per gram of adsorbent, ‘k’ is the Freundlich constant, 1/n is the heterogeneity index and ‘c’ is the concentration of the solute in solution. A plot of log ‘a’ versus log ‘c’ will give a straight line where the slope gives the adsorption capacity.

3. Results and Discussions

3.1. Chemical Composition of the Geopolymer Raw Materials

Table 1. Mean percentage of chemical composition of calcined clay, and sugar bagasse ash

Oxides (%)	Calcined clay	Sugar bagasse ash
	Mean ± SD	Mean ± SD
SiO_2	30.511 ± 0.453	64.386 ± 0.666
Al_2O_3	20.790 ± 0.080	13.716 ± 0.578
K_2O	0.6515 ± 0.406	2.817 ± 0.037
CaO	4.282 ± 0.840	2.272 ± 0.031
P_2O_5	6.523 ± 0.057	0.623 ± 0.084

SD-Standard deviation

X-ray fluorescence spectrometer was used in the determination of the chemical composition for the raw materials used. The main components of the calcined clays and sugar bagasse ash were oxides. Table 1 shows the mean% oxides obtained when calcined clay and SBA was analyzed using XRF.

The main oxides present in both calcined clay and SBA was silica and alumina. The percentage of SiO_2 was 30.511 and 64.386%, Al_2O_3 was 20.790 and 13.716%, K_2O was 0.6515, and 2.817%, Cao was 4.282, and 2.272% and P_2O_5 was 6.523, and 0.623% for calcined clay, and sugar bagasse ash respectively. The results obtained for sugarcane bagasse were similar to those obtained by Danbala et al. [18]. For calcined clay, since it was obtained from the natural environment and not processed, the results were incoherent to those obtained by Salim et al. [19]. The high% of silica and alumina obtained in the calcined clay and SBA provided their potential as raw material for synthesis of geopolymers.

3.2. Chemical Composition of the Geopolymer and Activated Carbon

The chemical composition of geopolymer and activated carbon used in fabrication of geopolymer filters is presented in table 2.

Table 2. Mean percentage of chemical composition of geopolymer, and activated carbon

Oxides (%)	Geopolymer	Activated carbon
	Mean ± SD	Mean ± SD
SiO_2	66.391 ± 0.600	0.459 ± 0.162
Al_2O_3	11.744 ± 0.449	11.104 ± 0.373
Fe	9.70 ± 0.053	0.696 ± 0.011
K_2O	4.507 ± 0.039	0.048 ± 0.015
CaO	1.773 ± 0.028	0.000 ± 0.017
P_2O_5	0.512 ± 0.600	2.310 ± 0.077
Zn	1.812± 0.012	21.283± 0.042
Cl	1.888 ± 0.017	63.482 ± 0.046
Ti	1.104 ± 0.015	0.000± 0.010

SD- Standard deviation

The major components of the geopolymer were SiO_2 at 66.391% and Al_2O_3 at 11.744%. These results were in agreement with those obtained by Maingi et al. [9]. The other elements and oxides identified were Fe at 9.70 ± 0.053%, K_2O at 4.507 ± 0.039%, Cl at 1.888 ± 0.017%, Zn at 1.812± 0.012%, CaO at 1.773 ± 0.028%, and Ti at 1.104 ± 0.015%. Activated carbon was analyzed to identify if the activator was embedded in its structure. XRF fluorescence could not identify carbon as an element since it was beneath its spectrum. The major elements identified were Zn and Cl. The result from activated carbon was similar to those obtained by Shamsuddin, et al. [20]. The high% of Zn and Cl obtained in activated carbon could be attributed to the $ZnCl_2$ used as an activator.

3.3. Characterization of Geopolymer and Activated Carbon Using FT-IR

Fourier Transform Infra-Red analysis was carried out for spectroscopic analysis. The identification of functional groups in both geopolymer and activated carbon is critical for the adsorption of heavy metals. FT-IR absorption spectra were recorded in the wavelength range of 4600-150 cm^{-1}, using FT-IR (IR tracer 100 Shimadzu). Potassium chloride was used to make the nujol mull to be analyzed.

In the geopolymer, the spectra band at 989.50 cm^{-1} could be attributed to Si-O-Al and Si-O-Si asymmetrical vibrations. The band at 1451.46 cm^{-1} could be a result of O-C-O, formed from remaining unreacted activator and carbon (IV) oxide [21]. The peak at 1641.45 cm^{-1} corresponds to H-O-H bending vibrations, and at 3415.03 is related to –OH, H-O-H bending vibrations [21]. For activated carbon, the spectra band at 3415.03 is attributed to the stretching O-H bond in the phenolic, carboxylic functional group, and adsorbed water [22]. These functional groups obtained in both geopolymer and activated carbon is vital in the adsorption of positively charged metal ions through electrostatic attraction.

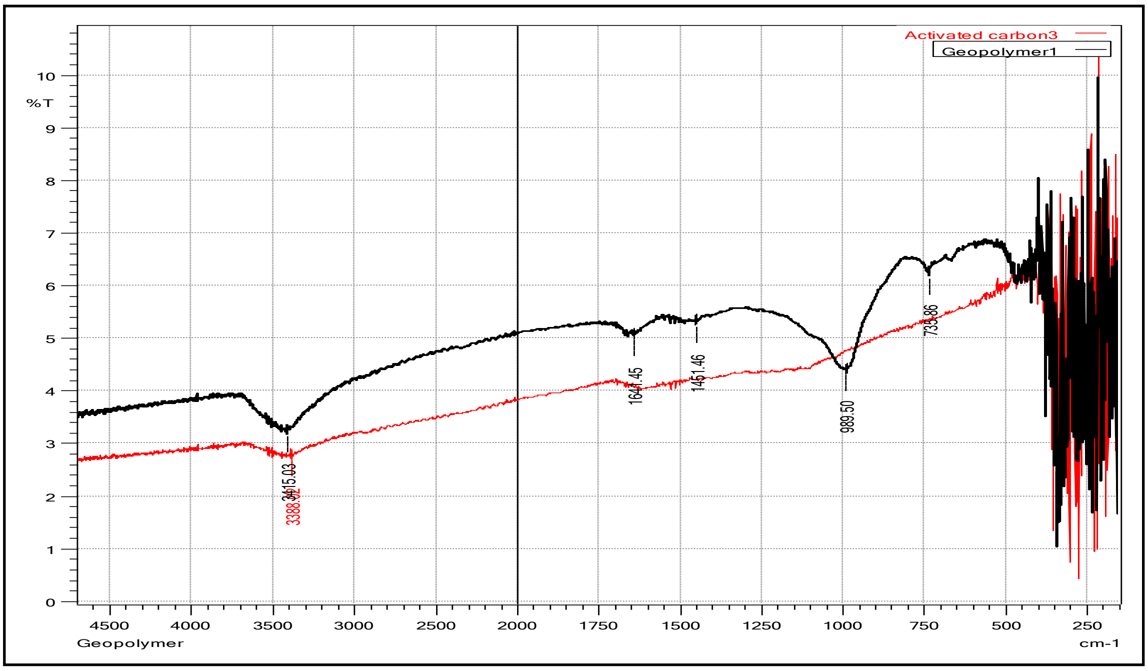

Figure 1. FT-IR spectra for geopolymer, and activated carbon

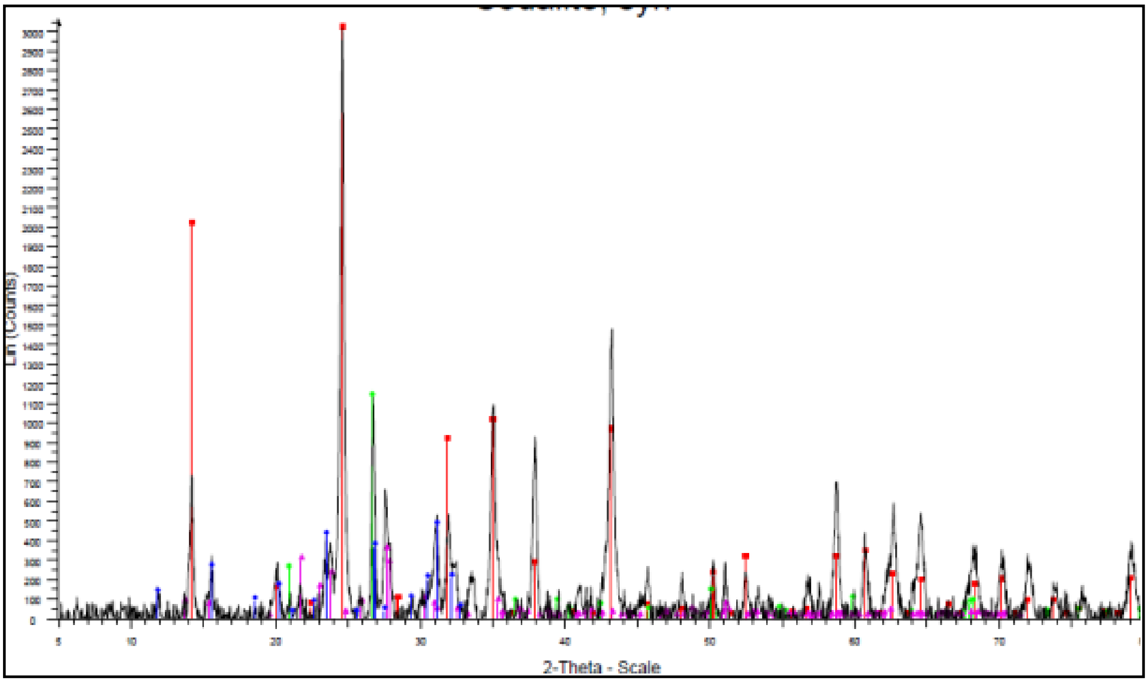

Figure 2. Powder diffraction of the Geopolymer

3.4. Characterization of Geopolymer, and Activated Carbon Using XRD

X-ray diffraction was required to give details on whether the geopolymer and activated carbon were amorphous or crystalline. If the high percentage was crystalline it would indicate that the adsorption sites were fewer than expected but if it was amorphous it would illustrate that the adsorption site were more exposed [23]. Figure 2 shows the diffraction pattern of geopolymer.

The XRD allows us to identify different phases between the activator and the reference material. The main phases were Quartz (SiO_2), Albite ($NaAlSi_3O_8$), Sodalite ($Na_8Al_6Si_60_{24}(OH)_2(H_2O)_2$), and Sodium Aluminum Silicate Hydrate Furfural ($C_5H_4O_2 \cdot Na_2O \cdot Al_2O_3 \cdot 3.3SiO_2 \cdot 7H_2O$) in the geopolymer. Sodalite yielded the most at 59.16%, Quartz yielded 22.13%, and Albite yielded 6.77%. The presence of quartz and albite was an indication of crystallinity in the geopolymer [23]. The appearance of the sodalite phase which is specific for zeolites identifies the formation of a material that is mesoporous. Sodalite content is usually proportional to the cation exchange capacity between the activating solution and raw materials [24]. The geopolymer obtained was semi-crystalline and therefore its use as an adsorbent especially in column adsorption could result in clogging hence the need to reduce crystallinity. Figure 3 shows the diffraction pattern of the activated carbon.

The activated carbon sample had the signature of graphite, despite having very broad peaks characteristic for activated carbon. The absence of sharp peaks and the appearance of broad diffractions background is an indication that the structure was amorphous. Two broad peaks were witnessed at between 10 and 34°C and between 40 and 50°C in 2theta scales. The diffraction pattern shows the activated carbon was mainly amorphous. These results are in tandem with those of Shamsuddin, et al. [20]. The amorphous nature of activated carbon provides a good surface for reducing crystallinity in geopolymer and hence aiding the adsorption process when columns are used.

3.5. Adsorption of Pb (II) and Cu (II) Ions Using the Geopolymer/ Activated Carbon Filters

3.5.1. Effects of Geopolymer: Activated Carbon Ratios on the Adsorption of Pb (II) Ions, and Cu (II) Ions

Geopolymer: activated carbon ratio was studied using three filters, A, B, and C. Filters A, B, and C had a ratio of 1:2, 1:1, and 2:1 respectively. Table 3 shows the effect of varying the ratio of geopolymer: activated carbon on the adsorption of Pb (II), and Cu (II) ions from aqueous solutions.

Table 3. Effect of geopolymer: activated carbon ratio on mean percentage adsorption of Pb(II) and Cu(II) ions

	Pb(II)	Cu(II)
Geopolymer filters	Mean±SD	Mean±SD
A	76.19 ± 0.19^c	64.52 ± 1.13^c
B	91.69 ± 0.66^b	76.73 ± 0.53^b
C	95.90 ± 0.69^a	97.85 ± 0.87^a

SD-Standard deviation

There was significant difference in mean percentage removal with change in geopolymer: carbon ratio. Filter C had higher adsorption efficiency compared to filters A and B. This could be attributed to the high amount of geopolymer in the filter as compared to filters B, and A. The passage of synthetic wasterwater in filter A was faster compared to filters B, and C. The high amount of activated carbon in it increased the porosity of the filter hence the high flow rate. Even though increasing porosity is important in this study, the removal of heavy metal ions was of more importance. Therefore, Filter C with a geopolymer: activated carbon ratio of 2: 1 was selected for other optimization procedures due to its high adsorption efficiency.

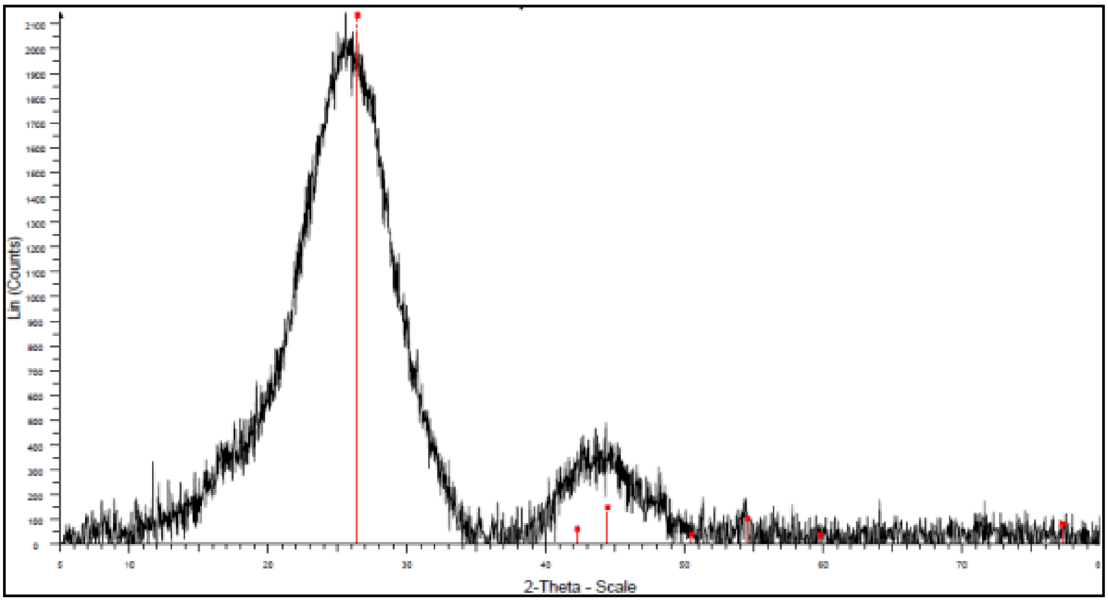

Figure 3. Powder diffraction of the activated carbon

3.5.2. Effects of Filter Thickness on the Adsorption of Pb (II) and Cu (II) Ions

Three different filter thicknesses of 2cm, 4cm, and 8cm were used. Table 4 shows the results of effect of varying filter thickness on adsorption of Pb (II) and Cu (II) ions from aqueous solutions.

Table 4. Effect of filter thickness on mean percentage adsorption of Pb (II) and Cu (II) ions

	Pb(II)	Cu(II)
Filter thickness(cm)	Mean±SD	Mean±SD
2	85.49 ± 1.28^{c}	80.42 ± 0.75^{c}
4	92.53 ± 0.29^{b}	90.29 ± 1.13^{b}
8	98.64 ± 0.97^{a}	97.61 ± 1.42^{a}

SD-Standard deviation

There was significant difference in the mean percentage adsorption of the two heavy metal ions studied when the filter thickness was varied from 2 to 8 cm. As the filter thickness increased, the percentage removal of the heavy metal ions also increased. The removal increased steadily from a thickness of 2cm to 8cm. The increase in the filter thickness resulted in an increase in adsorption sites [25] and hence increased rate of removal of the heavy metal ions.

3.5.3. Effects of Initial pH on the Adsorption of Pb (II) and Cu (II) Ions

Table 5 shows the effect of varying pH on the adsorption of Pb (II) and Cu (II) ions from synthetic wastewater when geopolymer: activated carbon ratio of 2:1 and filter thickness of 4 cm was used.

Table 5. Effect of pH on mean percentage adsorption of Pb(II) and Cu(II) ions

	Pb(II)	Cu(II)
pH of sorbate	Mean±SD	Mean±SD
2.1	66.54 ± 2.24^{c}	84.59 ± 2.16^{c}
4.4	86.38 ± 0.57^{b}	88.90 ± 0.82^{b}
6.1	92.64 ± 0.27^{a}	95.78 ± 0.26^{a}

SD-Standard deviation

From table 5, an increase in the pH of the solutions increased the percentage removal of the metal ions. At low pH, the H^+ ions and the metal ions compete in the solution hindering adsorption but as the pH increases, H^+ ions concentration decreases hence reducing the competition, and as a result metal ions are adsorbed more [26].

3.5.4. Effects of Initial Metal Ion Concentration on the Adsorption of Pb (II) and Cu (II) Ions

The initial metal ion concentration of any solution is important since it indicate the concentration in which the adsorbent is most effective. It also indicates how much of the adsorbent is to be used or how much of the solution should be passed through the filter. Figure 4 shows the effect of initial concentration on the adsorption of Pb.(II) and Cu.(II) ions from aqueous solutions.

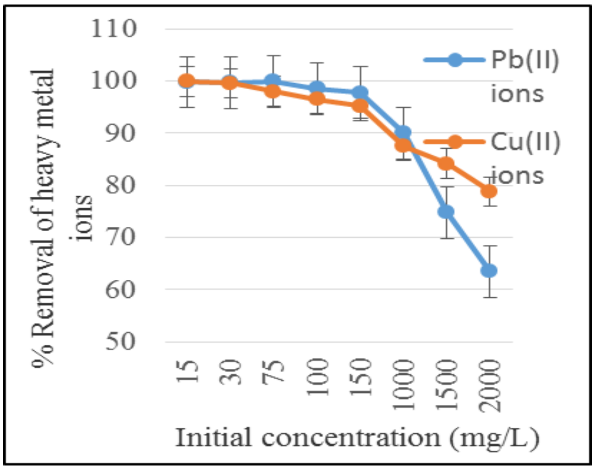

Figure 4. Effect of initial concentration of heavy metal ions on Pb (II) and Cu (II) ions adsorption on 50 mL solutions, Geopolymer: activated carbon ratio 2:1, filter thickness of 4 cm, Pb (II) ions pH of 5.76, and Cu (II) ions pH of 5.57

At low concentrations of heavy metal ions, the highest percentage removal of the metal ions was observed. As the concentrations of the metal ions increased, the removal rate reduced. At low concentrations, the adsorption sites were more than enough to adsorb the metal ions, and as the concentration of heavy metal ions increased the adsorption sites remained constant. This shows low initial metal ion concentration favors adsorption due to a high number of sorbent sites as compared to higher initial metal ion concentration [27]. Initial metal ion concentration is also key in determining the adsorption capacities of sorbent and therefore providing an economic view of the sorbent.

3.6. Equilibrium Studies Using Adsorption Isotherms

The adsorption of Pb (II) and Cu (II) ions was studied using Langmuir and Freundlich adsorption isotherms. Table 6 shows the isotherm constants for adsorption of Pb (II) and Cu (II) respectively.

Table 6. Isotherm model constants and correlation coefficients for adsorption of Pb (II) and Cu (II) ions using the geopolymer filter

	Langmuir			Freundlich		
Metal ions	K_L	Q_{max} (mg/g)	R^2	1/n	K_F	R^2
Pb(II)	0.033	8.403	0.917	0.760	0.015	0.978
Cu(II)	0.002	0.599	0.801	0.382	0.028	0.854

K_L = Langmuir constant, Q_{max} = Maximum adsorption, R^2 = correlation coefficient, 1/n = adsorption intensity, K_F = Freundlich constant

From table 6, the experimental data of both adsorption of Pb (II) and Cu (II) ions fitted best in Freundlich isotherm due to the high correlation coefficients obtained. The heterogeneity index (1/n) was less than one for both heavy metal ions an indication that the adsorption process was feasible on heterogenous surface. The Freundlich isotherm was therefore used to determine the adsorption capacitiy of the geopolymer filter used. The K_F value of 0.015 and 0.028 were obtained for Pb(II) and Cu(II) ions respectively. The heterogeneity indices obtained on the two heavy metal ions shows that geopolymer filter favored multilayer adsorption as opposed to monolayer adsorption.

4. Conclusions

FT-IR analysis showed the presence of functional groups in the geopolymer filter vital for the adsorption of heavy metals. Phase composition analysis using XRD showed that the geopolymer was crystalline and activated carbon was amorphous therefore mixing the two reduced the crystallinity level and hence increasing the adsorption process. The geopolymer: activated carbon ratio of 2:1 and filter thickness of 4cm gave the highest removal rate of the Pb (II) and Cu (II) ions. Experimental data fitted best in linearized Freundlich isotherm an indication that the adsorption occurred on a heterogonous surface. This study has shown that geopolymer filters derived from calcined clay, sugar bagasse, and activated carbon has high potential of being used as filters for the removal of heavy metals from an aqueous solution.

ACKNOWLEDGEMENTS

The authors would like to express their sincere gratitude to Department of Chemistry Kenyatta University and Department of Science Technology and Engineering, Kibabii University for assistance offered during this study.

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