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EXPLORING THE SELECTIVE REMOVAL OF CATIONIC POLLUTANTS FROM SYNTHETIC MINE WASTEWATER USING ZEOLITIC MATERIALS SYNTHESIZED FROM GHANAIAN BAUXITE AND KAOLIN

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Abstract

Zeolites A, X and Y were successfully synthesized using hydrothermal methods from local deposits of bauxite (Awaso) and kaolin (Anfoega) with unique pore sizes and frameworks. The zeolites were characterized by XRD and SEM/EDX techniques. A ratio of 2 g of each zeolite to 100 ml of laboratory prepared mine wastewater with predetermined concentrations of Pb^{2+}, Cu^{2+}, Fe^{2+}, Zn^{2+} and CrO_4^{2-} was considered to investigate the removal of the heavy metals. Pb^{2+} removal was highest in both single and mixed metal solutions with a removal efficiency or uptake of about 99% by all zeolites in single metal solutions and 90, 94 and 96 % removal using zeolites A, X and Y respectively in the mixed systems. CrO_4^{2-} recorded the least removal efficiency of 5% for Zeolite A and around 20 % for zeolite X and Y. In the mixed metal solution, CrO_4^{2-} removal efficiency reached an equilibrium after 24 hours run for the types of zeolite materials below 20 % of the initial concentration indicating selective removal of cations by the different types of zeolites compared with anionic species.

Keywords
heavy metals, removal efficiency, selectivity sequence, zeolites, characterization

Introduction

Mining is considered an important economic endeavor which contributes to the development and livelihood of the region where it is located. It plays an important role in the economic development of the country or community bestowed with the resource, by generating revenue usually through employment opportunities and foreign exchange. It has been estimated that small-scale mining contributes to 12% of the world's total gold production (Besseah, 2011) and 40% of gold production in Ghana.

According to (Asklund and Eldvall, 2005) over 150,000 persons in Ghana have been estimated to be operating illegally on concessions belonging to large-scale operators, or in restricted areas. Washing of the gold ore to remove gangue is a major process in small-scale mining activities which requires volumes of fresh water. Ghana's large alluvial gold deposits also encourage operations of these small-scale miners around streams and rivers which causes siltation thereby increasing the amounts of heavy metals in water bodies which serve as sources of drinking water for populations downstream (Oduro et al., 2012) or seep into underground water polluting it.

Heavy metals occur naturally and play a vital role in the metabolism of many organisms but may have inimical effect on the ecosystem and human health (Whitehead, 2000). Heavy metals are considered very toxic. They are known to bioaccumulate in organisms, causing morbidity and mortality (Inglezakis et al., 2004). Remediation methods that seek to remove dissolved heavy metals from polluted waters can be challenging but necessary, regarding the correct management of wastewaters for a sustainable and cleaner production as well as the effect of competing cations on the efficiency of the exchange medium. This paper investigates synthesized pure phase Zeolites A, X and Y from bauxite and kaolin in Ghana, with unique pore sizes and frameworks in the removal of dissolved heavy metals (Pb, Cu, Fe, Zn, Cr) as constituents of mine wastewater prepared as both a single and mixed metal solution in the laboratory. The experiments were conducted to comprehend the processes involved in the removal of the metals in synthetic mine wastewater both as single and multiple metal solutions and to subsequently achieve maximum efficiency of the system. This was considered to investigate the effect of competing cation against the other in solution. Findings from the study would provide a remediative solution to water pollution caused by anthropogenic activities such as agriculture and mining.

Materials and Methods

Zeolites were synthesized from Awaso bauxite and Anfoega kaolin in the Western and Volta Regions of Ghana, using the hydrothermal protocol as outlined by (Kwakye-Awuah et al., 2014) but with varying aging times of 5, 24 and 48 hours to obtain different zeolites using a 2 M NaOH solution and heated at 110°C. No pH adjustments were made. A constant crystallization time of 6 hours was considered for all the synthesis. The zeolites were characterized by XRD and SEM/EDX analytical techniques.

Synthetic mine water was produced in the laboratory to simulate the mine wastewater that was sampled from a mining area. Preliminary studies showed the presence of Pb, Fe, Cu and Zn in the mine waste water. Standard stock solutions were prepared from Lead (II) nitrate (ACS Reagent $\geq$ 99.0%), Copper (II) chloride (ACS Reagent $\geq$ 99.0%), Iron (II) chloride tetrahydrate (Reagent Plus 98%), Zinc chloride (Reagent grade $\geq$ 98%) and Potassium dichromate (ACS Reagent $\geq$ 99.0%) all sourced from Sigma Aldrich, UK and used without further purification or treatment to prepare the wastewater matrix. The stock solutions were appropriately labelled and stored in 1000 ml conical flasks. For single metal solutions, concentrations of Zn^{2+} (5 mg l^{-1}), CrO_4^{2-} (0.5 mg l^{-1}), Cu^{2+} (5 mg l^{-1}), Pb^{2+} (2 mg l^{-1}) and Fe^{2+} (35 mg l^{-1}) were used. However, for the mixed metal solutions, Zn^{2+} (5.1 mg l^{-1}), CrO_4^{2-} (2.21 mg l^{-1}), Cu^{2+} (4.6 mg l^{-1}), Pb^{2+} (2.32 mg l^{-1}) and Fe^{2+} (29.8 mg l^{-1}) were considered. With the exception of Fe, all the stock solutions were prepared with concentrations higher than that of the mine wastewater.

Removal of dissolved heavy metals by Batch Technique

To investigate the removal of dissolved heavy metals by the synthesized zeolites, the batch technique was used. The parameters considered for the investigation were: the type of zeolite (adsorbent), adsorbent to wastewater dosage, contact time, and heavy metal concentration. A kinetic study of the adsorption process was conducted using a solid to liquid ratio of 1:100, with time intervals of 15, 30, 45, 60, 120, 180, 240 and 1440 mins and continuous agitation using a rotary shaker at an average speed of 200 revolutions per minute (rpm). Increasing the amount of zeolite increases the pH of the system above the stipulated value thus the 1:100 solid-liquid ratio was used to maintain the pH of the batch system (acidic) to simulate the treatment of contaminated waters in an acidic medium. The zeolite was filtered from the liquid phase immediately after the required time using a Buchner funnel and Whatman No. 1 filter paper under vacuum to reduce contact time after the reaction duration. All experiments were conducted at 27°C. Three replicates of the experiment were conducted. The concentrations of the individual heavy metals in aqueous solution were determined with an TNTplus Test methods using a Hach DR3900 UV-Vis spectrophotometer (Hach Company, 2007). Cr^{6+} was determined using the 1,5-Diphenylcarbohydrazide Method 8023 at λmax 540 nm. Cu^{2+} was determined using the Bicinchoninate Method 8026 at λmax 560 nm. Fe^{2+} was determined using the 1,10-Phenanthroline Method 8146 at λmax 510 nm. Zn^{2+} was determined using the Zincon Method 8009 at λmax 620 nm. Pb^{2+} was determined using the Dithizone Method 8033 at λmax 515 nm.

Results and Discussion

Zeolite characterization

From the XRD of the synthesized zeolites in this study (Figure 1), zeolite Linde Type A (LTA) was obtained when an aging time of 5 hrs was considered whilst Faujasite (FAU) type zeolites were obtained with 24 and 48 hrs aging. Their characteristic peaks appeared at $2\vartheta = 7.18^o$, 10.17^o and $2\vartheta = 6.12^o$, 10.00^o for LTA and FAU respectively, in agreement with previous studies in literature (Treacy and Higgins, 2007). Nucleation of zeolite crystals, according to the literature (Rivas Cardona, 2011) is believed to occur during the aging. It was observed in an earlier study (Akolekar et al., 1997) that the dissolution of silica causing the release of silicate ions occurs during aging of the reaction mixture according to thus increasing the yield and crystallinity of the synthesized product. According to a recent work (Xu et al., 2007) the formation of FAU is observed with long aging and LTA with short aging time. Also, it has been reported (Ogura et al., 2003) that the aging process is necessary for the formation of FAU and also proposed that 6R and double membered rings (D6R) are the precursors of FAU and are formed in the gel phase during the aging process.

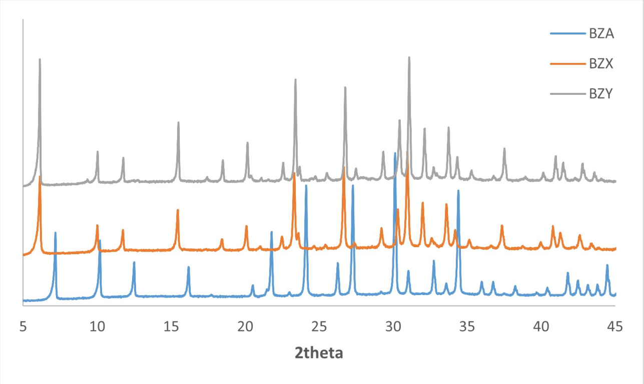

Figure 1. XRD plots as-synthesized zeolites with different aging times of 5 hrs (BZA), 24 hrs (BZX) and 48 hrs (BZY).

Zeolites A, X and Y were successfully synthesized from bauxite and kaolin with Si/Al ratios of 1.1, 1.3 and 1.5 with varied aging times of 5 (Figure 2a), 24 (Figure 2b) and 48 (Figure 2c) hours. SEM images obtained for the zeolites corroborate the XRD results for the as-synthesized zeolites with cubic crystals for zeolite LTA and hexagonal shaped crystals for FAU zeolites.

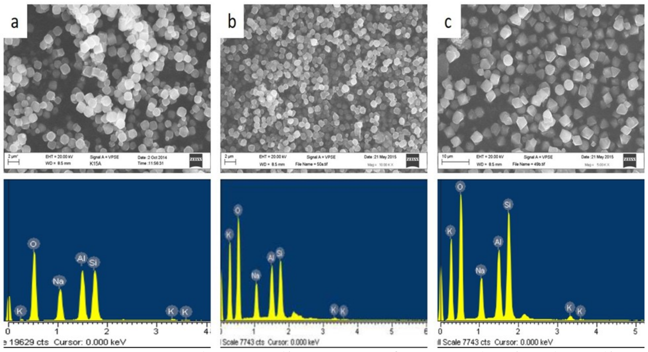

Figure 2. SEM and EDX diagrams of as-synthesized zeolites from bauxite and kaolin (a) 5hrs (b) 24 hrs (c) 48 hrs.

Kinetic Studies

Single metal solutions

The pH of mine wastewater is generally acidic, hence the pH range examined in this experiment was within the 1 – 7 range.

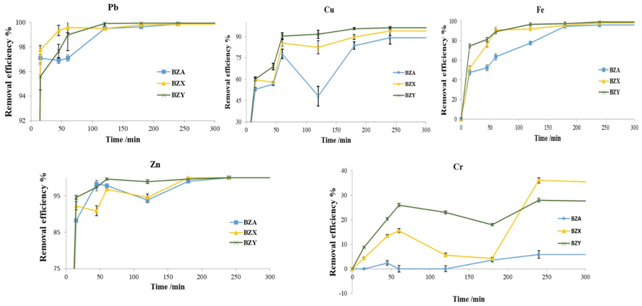

Figure 3. SEM and EDX diagrams of as-synthesized zeolites from bauxite and kaolin (a) 5hrs (b) 24 hrs (c) 48 hrs.

The performance of each synthesized zeolite in the removal of metal ions from single metal solutions was assessed and is presented in Figure 3. For all the types of as-synthesized zeolites, the rate of removal in single metal solutions were of the order of decreasing removal efficiency $Pb^{2+} > Zn^{2+} > Fe^{2+} > Cu^{2+} > Cr^{6+}$. From Figure 3, it can be inferred that in the exchange of Pb^{2+} equilibrium (>95 % ion exchange) is attained after 15 mins with no minimal changes in the cationic exchange on further exposure to the zeolitic material beyond the equilibrium point (i.e. after 60 mins). Zeolite X (BZX) had the highest rate of removal of Pb^{2+} followed by Zeolite Y (BZY) and Zeolite A (BZA). The rate of ion exchange of Zn^{2+} with the zeolitic materials (Figure 3) was however, BZY > BZX > BZA. Similar trends were observed for Fe^{2+}, Cu^{2+} and Cr^{6+} (Figure 3).

Cr^{6+} had the least removal efficiency of the heavy metals using the as-synthesized zeolites with ca 20 % equilibrium removal efficiency for BZY and BZX. Zeolite A reached equilibrium removal of Cr^{6+} *ca* 5 % (see Figure 3). Cr^{6+} removal was very poor because the Cr^{6+} exists in solution as an anion (CrO_4^{2-}) with weak ionic exchange with the zeolite framework.

Ion exchange capacity has been reported to be higher for zeolites with high Al in their zeolite framework as more charge compensation cations will be present to preserve neutrality (Pitcher, 2002). Low Si/Al ratios are an indication of high terminal Al-OH group concentration at the mineral/ solution interface and are also hydrophilic. Also, zeolites which possess low Si/Al ratios are known to hydrolyze easily in solutions (acidic conditions). Similarly, a larger ligand exchange and improved overall performance is observed during treatment experiments of metal removal from wastewater (Alvarez-Ayuso et al., 2003; Misak, 2000; Rios et al., 2008; Semosa et al., 2002).

Effect of competing metal cations (mixed metal solutions)

The effect of competing metal ions was investigated using zeolites A, X and Y. Figure 4 depicts the rate of removal of all the heavy metal ions by the individual zeolites as a function of time. As observed in the single metal solutions, the initial 15 mins was characterized by a sharp decrease of all the metals in solution but with varied rates of removal. All cations in solution were removed at efficiencies greater than 80% after 240 mins, for Zeolite X and Y whilst greater than 50% removal efficiency of all the cations were achieved with Zeolite A. The sudden removal of heavy metal ions during the early 15 mins of reaction can be attributed to the availability of free exchange sites on the zeolite surface which permit very rapid diffusion and exchange of the metals in the solution into the zeolites. However, the extent of metal removal depends on inherent properties of the species involved (Rios et al., 2008). The slow uptake observed beyond this time indicates a saturation or depletion of most of the active exchange sites. Fluctuations in removal as observed in both mixed and single solutions are usually due to different exchange sites within the zeolite frameworks. As such, as the reaction proceeds with time, some previously unreached sites with different affinities are accessed whilst other proton exchanges occur.

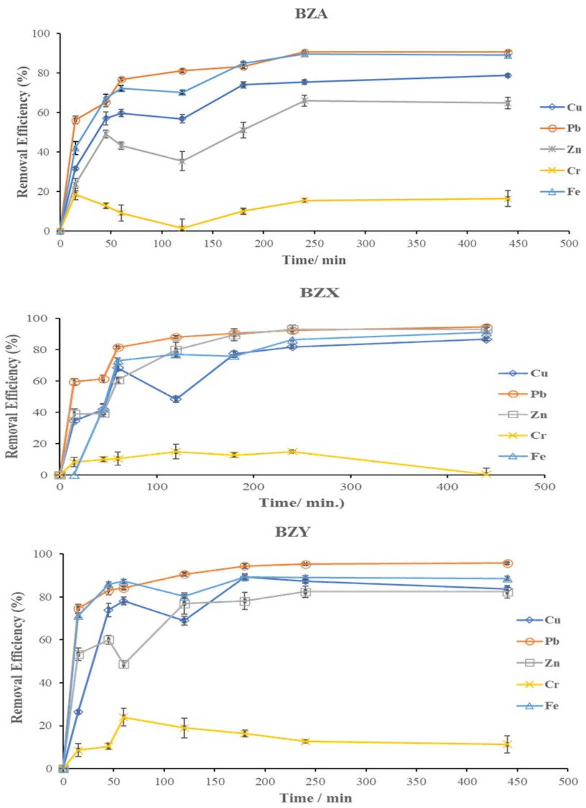

Figure 4. The effect of competing elements on the removal capacities of (a) BZA (b) BZX and (c) BZY.

As observed with single metal solutions, generally, all the zeolites used in the study produced high metal removal efficiencies except Cr^{6+} which exist in solution as an anion CrO_4^{2-} in contrast with the others which are cationic.

Selectivity Sequence

The cation-exchange selectivity depends on framework topology, ion size and shape, charge density on the anion framework, ion valence and electrolyte concentration in aqueous phase (Barrer, 1978). The most preferential ion is removed first by any zeolite. This illuminates why Cr^{6+} appear to be least affected with increasing time. The selectivity sequence

Table 1. Selectivity sequence of the zeolites observed in the study

Zeolite type	Single	Mixed
Zeolite A	Pb > Zn > Fe > Cu > Cr	Pb = Fe > Cu > Zn > Cr
Zeolite X	Pb > Zn > Fe > Cu > Cr	Pb > Fe > Zn > Cu > Cr
Zeolite Y	Pb > Zn > Fe > Cu > Cr	Pb > Fe > Cu > Zn > Cr

(Table 1) revealed that the zeolites show a strong affinity for Pb^{2+} in the mixed metal solution. Similar findings have been reported by (Cheng et al., 2018; Jangkorn et al., 2022; Kucuk et al., 2023).

Relative concentration of each of the metals would significantly affect the removal of each other implying that the metals are interdependent. However, if the exchange capacity of the zeolite is not exceeded, the preferred metal will be completely removed before the other competing metals. This phenomenon can be observed in all three zeolites (Figure 4) with Pb^{2+} and Cr^{6+} being the highest and least preferred ions respectively as shown in Table 1.

Amongst all three zeolites, a similar selectivity sequence in metal removal was observed in the single metal solution. Slight variations are however noted in the removal of the metals in the mixed metal solutions. Common factors known to affect the selectivity of a zeolite for various cations according to Eisenman (1962); Sherry (1979) include the radius of the cation (Pb^{2+} 119 pm, Fe^{2+} 77 pm, Zn^{2+} 74 pm, Cu^{2+} 73 pm and Cr^{6+} 52 pm), its hydrated cationic size, the diameter of the zeolite channels and the amount of cation to zeolite framework interaction that can occur.

Effect Zeolite framework pore on metal removal

Heavy metal removal with zeolite A was lower compared with removal efficiencies obtained for zeolites X and Y. This can be attributed to the pore opening of Zeolite A which is relatively smaller than the pore openings of zeolites X and Y. Depending on the particular zeolite structure, the pore openings of the zeolite cavities may be too small to allow large cations (Table 2). The pores may consist of one type of channel having essentially the same width. This determines the accessibility of the channel to incoming cations from the solution.

Table 2. Pore dimensions of the zeolites used in the study (Xu et al., 2007)

Zeolite type	Framework type	Pore (Å)
Zeolite A	LTA	4.1 × 4.1
Zeolite X	FAU	7.4 × 7.4
Zeolite Y	FAU	7.4 × 7.4

Low removal efficiencies are achieved when the cations in the solution cannot occupy the available ion exchange sites present in the zeolite framework. This is usually due to volume exclusion where the internal volume of the zeolite is filled with charge balancing cations.

On the basis of available exchangeable sites in the zeolite framework, LTA has 3 cation sites. A fully Na-exchanged LTA with Si/Al ratio of 1 has 12 cations per alpha cavity whereas FAU can accommodate up to 48 cations in sites 1 and 2 of its framework (Kulprathipanja, 2010). Hence obtaining a greater removal of cations for zeolite X and Y than zeolite A can be justified.

Ionic charge and hydration enthalpy

Theoretically, the pore opening of zeolite A will exclude hydrated divalent Cu and Zn from entering its framework. Ionic charge and hydration enthalpy are related, in that a higher charge shows a higher enthalpy (Cansever, 2004). A large ion charge/size ratio results in an increase in the hydration energy. Increasing the ionic radius, results in a decrease in the hydration enthalpy.

The radii of unhydrated cations are usually much smaller than their hydrated form (Table 3). The ease with which a cation can shed its hydration sheaths is an additional factor in determining the selectivity of the zeolite for a specific cation. A large hydrated ion can vary its size by losing its water of hydration from the solvated ions to enable them penetrate the pores of the zeolite framework (Weitkamp and Puppe, 1999). The size of the hydration shell at a particular temperature depends on the size and charge of the anhydrous ion. The rate of ion exchange depends on the concentration of the ions of size capable of penetrating the pores of the zeolite (Kuhl, 1999). In a related study (Kirov and Filizova, 2012) it was observed that the degree of ion exchange in a zeolite depends on the structural interactions particularly on the positions of the competing cations.

Table 3. Ionic and hydrated radius of cations Cotton and Wilkinson (1972); Lange (1969)

Ion	Unhydrated (Å)	Hydrated (Å)	Enthalpy of Hydration (kJ mol^{-1})
Pb^{2+}	1.21	4.01	-1481
Fe^{2+}	0.75	5.82	-1946
Zn^{2+}	0.74	4.30	-2046
Cu^{2+}	0.70	4.19	-2100
CrO_4^{2-}	0.52	4.26	-1103

Experimental results obtained in earlier work in literature by (Kucuk et al., 2023; Whitehead, 2000) using zeolite A for heavy metal removal recorded the sequence Pb > Zn > Cu which agrees very well with the results obtained. It can be inferred that the zeolites used in the study exhibit a higher removal for metal ions with lower enthalpy of hydration. Similar studies (Pitcher, 2002; Zamzow and Schultze, 1995) attributed the high affinity for Pb to be due to its low free energy. Also, it has been observed in literature (Yuan et al., 1999) that the partial stripping of the hydration shells of an ion is a requirement for a cation to be able to enter the zeolite framework. This has been observed to occur readily with Pb ions (Pitcher, 2002).

Cr was least removed in all the metal solutions by all zeolites. Earlier work with Mexican clinoptilolite using a pH of 7 in their investigation achieved 12% Cr^{6+} removal (Mier et al., 2001). Studies on cationic metal ions removal processes indicate that Cr^{6+} removal occur differently compared with other heavy metals. Cr^{6+} is found as chromates or dichromates which are anions. The negative charge of chromate or dichromate is responsible for the poor adsorption in some mineral structures and soils thereby giving it its great mobility. Ion exchange of Cr6+ with zeolites is usually not successful as charge repulsion may occur from the negatively charged zeolite framework. This phenomenon has been experienced (Figueiredo and Quintelas, 2014). Cr^{6+} removal can be achieved by modifying the zeolite surface with a surfactant. The modification of zeolites using cationic surfactants such as HDTMA changes the negative basal surface charge of the zeolite. A study by (Jimenez-Castaneda and Medina, 2017) using HDTMA-Cl kaolinites were able to adsorb up to 10 times more Cr^{6+} than the natural form by significantly increasing both the specific surface area and the CEC of the clay. Cr^{3+} on the other hand is directly removed by both natural and synthetic zeolites with a wide variety of treatment methods.

Although this study employed synthetic wastewater for controlled assessment, the use of different zeolites for the remediation of acid mine drainages have been reported in literature by (Motsi et al., 2009; Ramos et al., 2023; Rios et al., 2008; Semosa et al., 2002; Zamzow and Schultze, 1995). When real mine wastewater is used, storage conditions are critical to preserve chemical integrity as acquiring actual mine wastewater samples often required travel to remote mining sites (Le et al., 2020). Exposure to air can cause oxidation of Fe^{2+} to Fe^{3+} and subsequent precipitation, while prolonged storage may alter pH and metal speciation (Hove et al., 2008; Vries et al., 2022). As such, while the synthetic wastewater provides insight into intrinsic removal mechanisms, results from real mine effluents underscore the importance of considering matrix effects, multi-contaminant interactions, and operational parameters when evaluating zeolites for practical treatment applications.

Conclusion

Removal of heavy metal cations from actual and synthetic mine wastewater was successful by all three zeolites synthesized from bauxite and kaolin. Removal occurs as result of ion exchange and adsorption processes with ion exchange occurring due to the weak nature of bonds between the zeolite framework and extra framework cations. Zeolite X and Y was observed to achieve greater than 80% removal of all cations in the synthetic mine wastewater. Pb^{2+} removal was highest in both single and mixed metal solutions with a removal efficiency or uptake of about 99% by all zeolites in single metal solutions and 90, 94 and 96% removal using zeolites A, X and Y respectively in the mixed systems. Zeolite selectivity has been established to be influenced by the hydrated radii and the free hydration energy on the cations as well as the pore dimensions of the zeolite framework

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