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Removal of Pb^{2+} and Fe^{2+} from Water Samples in Ghana by Synthetic Zeolites as Measured by Atomic Absorption Spectroscopy and Light Transmission Experiments

B. Sefa-Ntiri[1], B. Kwakye-Awuah[2,*], P. Mensah-Amoah[3], C. Williams[4]

[1, 3]Department of Physics, University of Cape Coast, Cape Coast, Ghana

[2]Department of Physics, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana

[4]Faculty of Science and Engineering, University of Wolverhampton, WV1 1LY, United Kingdom

*Corresponding Author's Email: bkwakye-awuah.cos@knust.edu.gh

Abstract: ***The removal efficiencies of laboratory-synthesized zeolite types LTA and LTX on lead and iron in water samples collected from rivers, streams, and wells from Central and Volta regions of Ghana were investigated. The synthesized zeolites were characterized by X-ray diffraction, scanning electron microscopy/energy dispersive X-ray analysis, thermogravimetric analysis and Fourier transformed infrared analysis. We have achieved the following results: for zeolite LTX, lead concentrations in the raw water samples and the treated water samples were respectively 0.192 mgL^{-2} and 0.005 mgL^{-2} (allowable level in drinking water: 0.01 mgL^{-2}); and iron concentrations in the raw water and treated water samples were respectively 2.797 mgL^{-2} and 0.129 mg^{-2} (allowable level in drinking water: 0.3 mgL^{-2}). For zeolite LTA, lead concentrations in the raw water samples and the treated water samples were respectively 0.192 mgL^{-2} and 0.005 mgL^{-2} and iron concentrations in the raw water and treated water samples were 2.797 mgL^{-2} and 0.129 mg^{-2} respectively. The results showed that both zeolite types were effective in removing both lead and iron metal ions in all water samples.***

Keywords: Synthesis, Zeolites, Characterization, Removal Efficiency, Water.

1. Introduction

Good water enhances good health, hence sustainable development for the people. Therefore, it is needless to emphasize the importance of water in our life. However, water for different purposes has its own requirements for the composition and purity, and each body of water has to be analyzed on a regular basis to confirm its suitability [1]. In most countries where water quality regulations pertaining to drinking water are applied before or at the point where pipe water enters the distribution system, often makes it impossible for water supply authorities and consumers to know the quality of portable water reaching their homes [2]. In fact, in most countries of the world, there is shortage in fresh water supply for drinking, for example, in Ghana, illegal mining activities in rivers which provided easy alternative source of water, industrial activities and climatic changes have affected their economic sustainability. Thus, demand for fresh water is a serious economic setback.

As a result, many toxic heavy metals ions have been discharged into the environment as industrial wastes, causing serious soil and water pollution. Cadmium, Cu, Ni, Pb, Fe, Zn and Arsenic are especially common metals that tend to accumulate in water organisms, causing numerous diseases and disorders. They are also common groundwater contaminants at industrial and military installations [12, 13 and 17].

The heavy metal ions frequently contained in industrial and municipal wastewater can be harmful to aquatic and animal health. Due to accumulative toxicity to the human body, these heavy metals, for example, lead and iron, have allowable levels in drinking water since any excess can be of health hazard [2]. Lead, for example, is one of the most toxic metals to man. As it is difficult to detoxify by chemical or biological methods; gradual lead ion accumulation in the nervous and cardiovascular systems of the human body can subsequently cause serious diseases [15]. Iron (as Fe^{2+}) concentration of 40 μgL^{-1} can be detected by taste in distilled water. In mineralized spring water with a total dissolved solid content of 500 mgL^{-1}, the taste threshold value should be 0.12 mgL^{-1}. In well-water, iron concentrations below 0.3 mgL^{-1} have been acceptable [14 – 15]. In drinking-water supplies, iron (II) salts are unstable and are precipitated as insoluble iron (III) hydroxide, which settles out as a rust-coloured silt. This calls for more special and healthy treatment in order to obtain drinking water of high quality as well as to produce environmentally acceptable effluents.

Among the methods use in water treatment such as; boiling, distillation, reverse osmosis, filtration, ozonation, ion exchange, aeration, flotation, neutralization, coagulation, degasification, adsorption, screening, sedimentation, skimming, chlorination, oxidation ponds among others, zeolites have been found to have excellent removal of heavy metals in both drinking water and waste water.

Zeolites are a group of hydrated aluminosilicates of alkali or alkali earth metals: principally sodium, potassium, magnesium, barium, lithium, and calcium [9 – 10, 18, 19] containing pores and channels of molecular dimensions that are used widely in industry as ion-exchange resins, molecular sieves, sorbents and catalysts. Many occur naturally as minerals, and are extensively mined in many parts of the world. Others are synthetic, and are made commercially for specific uses, or produced by research scientists trying to understand their chemistry. Aluminum, silicon, and oxygen are arranged in a regular structure of [SiO_4] and [AlO_4] - tetrahedral units that form a framework with small pores of about 0.1-2 nm diameter running through the material. Their pores hold water and or other molecules.

In this work, zeolite types LTA and LTX were synthesized in the laboratory, characterized by X-ray diffraction, scanning electron microscopy/energy dispersive X-ray analysis, thermogravimetric analysis and Fourier transformed infrared analysis. Finally, the removal efficiency of the zeolites in the removal of iron and lead ions in water samples collected from different geographical parts of Ghana were investigated.

2. Materials and Experimental Method

2.1 Source Materials and Equipment

In this study, water samples were collected from two streams and a hand dug well in the Volta region of Ghana. Most people living in the communities where these water samples were collected, use the water for drinking, cooking, washing and they also use them for other household chores and for their animals. Water samples fetched were kept in clean and very neat containers for storage and transportation to avoid any interference.

The study took place in three institutions in Ghana, comprising scientists and researchers at the LASER and Fibre Optics Centre at the Department of Physics, University of Cape Coast (UCC), Water research centre at the Centre for Scientific and Industrial Research (CSIR) in Accra, and the Material Science Unit of the Department of Physics, Kwame Nkrumah University of Science and Technology (KNUST), Kumasi. Laser light transmission experiments were performed at UCC, physicochemical analyses including, water turbidity, pH/conductivity were conducted at CSIR, and the zeolites applications and measurements, at KNUST. Characterization techniques used in identifying the zeolites were performed at the University of Wolverhampton, in the United Kingdom.

2.2 Synthesis of Zeolite LTA and LTX

Zeolites LTA and LTX were synthesized in the laboratory as per our previous [5 – 8]. To simplify the synthesis protocol the batch composition for the synthesis were given as:
$3.165Na_2O: Al_2O_3: 1.926\ SiO_2: 128H_2O$ for zeolite LTA and
$18Na_2O: Al_2O_3: 4SiO_2: 325H_2O$ for zeolite LTX, respectively.

The synthesized samples were filtered using a vacuum funnel and Whatman No. 45 filter paper and the powder samples obtained were washed copiously with distilled water until the pH of the filtrate was less than 9. Following overnight drying of the powdered zeolite at 100 ^oC in an electrical oven, the zeolite was crushed into uniform powder with pestle and mortar and stored in a plastic bag.

2.3 Characterization of Synthesized Zeolites

To confirm the crystal structure and the composition of the synthesized zeolite, it was essential to characterize the zeolite. The X-ray diifraction XRD pattern of the zeolite X which gives a measure of phase purity were recorded were recorded on Empererean X-ray powder diffractometer over 2θ range of 3^o to 50^o (PANalytical, UK Ltd, Cambridge).

The diffractometer was equipped with a graphite monochromated $\mathrm{Cu}\ \mathrm{K}_\alpha$ radiation source (8987 eV; $\lambda = 1.5418\ \mathring{A}$). The surface morphology of the zeolite as well as the elemental composition was examined by scanning electron microscopy (SEM) using a Zeiss EVO 50 equpped with energy dispersive X-ray spectrometer (EDX) (Zeiss, UK). Aluminium stubs were prepared prior to the analysis with an adhesive coating. The samples were sprinkled on the stubs. Where necessary, the samples were gold-coated using an Emscope SC500 Sputter coater to reduce static charging. Electron micrographs were obtained at various magnifications.

The vibrational properties were investigated by Fourier transformed infrared spectrometry (FTIR). Measurements were done using 100 scans at 4 cm resolution, units of log (1/R) (absorbance), over the mid-IR region of 1200-400 cm . An air background spectrum was collected at the start of the sample analysis A small sample of each zeolite (with or without silver) was centered on the ZnSe plate to ensure that it covered the entire crystal surface, and a pressure clamp was used to apply pressure on the sample. The zeolite samples were analyzed three times for three different samples. A background spectrum was measured before every sample to compensate for atmospheric conditions around the FT-IR instrument. Thermogravimetric (TGA) analysis was performed using a Perkin Elmer TGA 7 (Perkin Elmer, UK). The temperature range for the analysis was 50 ^oC to 800 ^oC.

2.4 Batch Treatment of Water Samples with Zeolites

The batch technique was utilized to monitor the effect of mass of both zeolites on ion exchange. In terms of zeolite LTX, six water samples (three raw samples and three filtrate samples) were further divided into two, making twelve samples in all. An equal volume of 100 ml of each sample was measured into twelve flasks. The mass of zeolite was varied, using 0.2 g and 0.5 g. Zeolite mass to solution ratio was adjusted to obtain significant differences in the removal efficiency of each zeolite mass. Zeolite LTX was used with all the water samples. With varying mass labels, the flasks were then placed on a rotary shaker at an average speed of 200 rpm at room temperature. The zeolite was then filtered from the water samples after one hour using 0.45µm nanofiber filter paper. The above procedure was repeated for zeolite LTA, but with only the raw samples.

3. Results and Discussions

3.1 Characterization of zeolites

Scanning Electron Microscopy (SEM), showed that the LTA zeolite was cubic in morphology whilst zeolite LTX was octahedral as shown Figure 1((a) and (b)) in agreement with our earlier work [5].

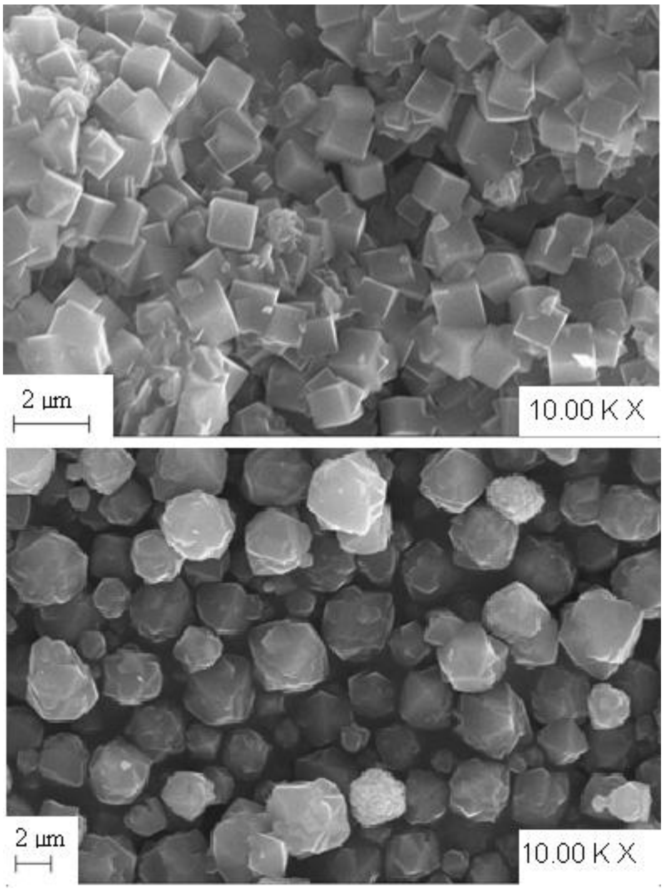
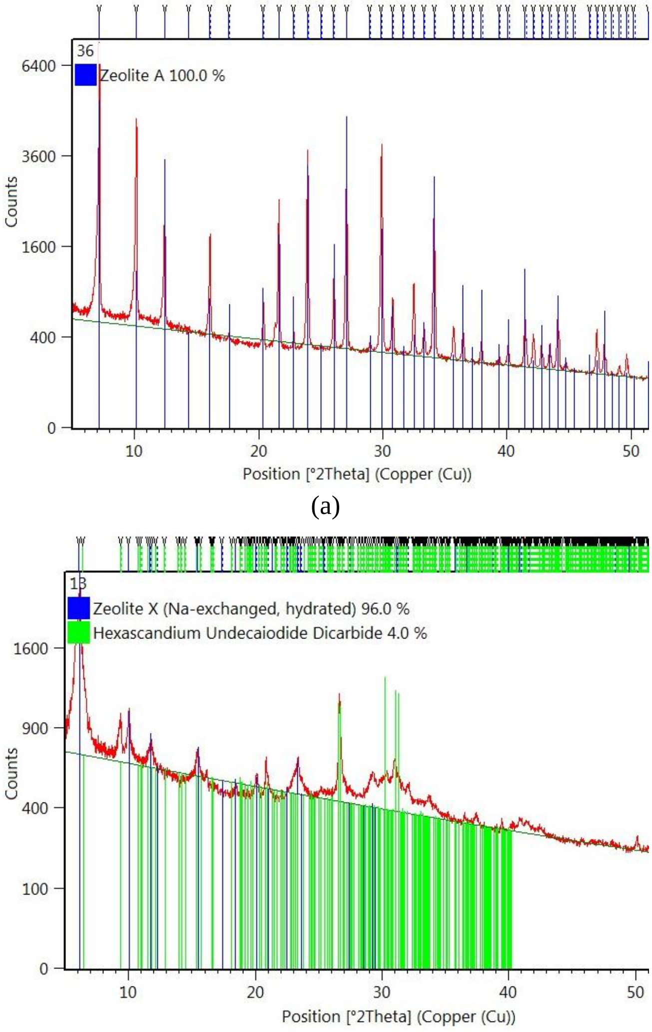


Figure 1: Scanning electron micrograph of top: Zeolite LTA and bottom: Zeolite LTX

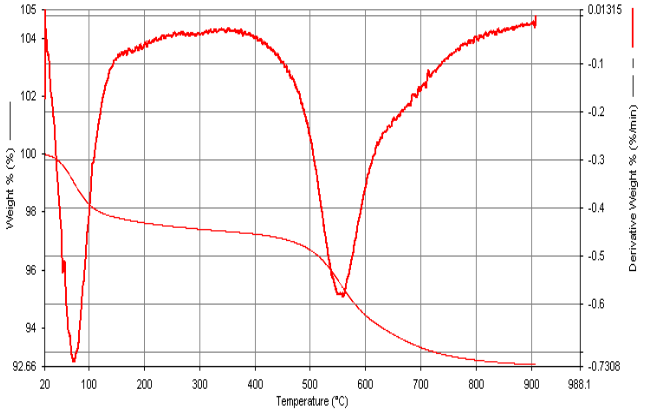
The XRD spectra for Zeolite LTA and Zeolite LTX are shown in Figure 2(a) and Figure 2(b). Rietvield analysis confirmed 100 % composition of zeolite LTA and 99.6 % Zeolite LTX respectively. Thermogravimetric analysis (TGA) of both zeolites showed weight loss of 7 % and 8 % for zeolite LTA and LTX, respectively up to 800 °C. The most apparent weight loss occurred at 90 °C and 550 °C for zeolite LTA and 70 °C and 550 °C for zeolite LTX, respectively. This is attributed to water evaporation on the surface and within the framework of the zeolites (Figure 3) as well as decomposition of calcium carbonate residue that may have remained during the phase transition of bauxite to zeolite (Kwakye-Awuah et al., 2013). The TGA results also showed that zeolite LTA was more thermally stable compared with zeolite LTX.



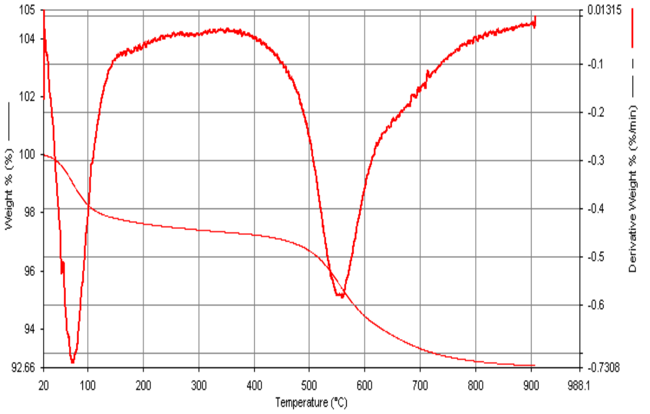
(a)

(b

Figure 2: X-ray diffraction spectrum for (a): Zeolite LTA and (b): Zeolite LTX.



(a)



(b)

Figure 3: Thermogravimetric – differential thermal graph for: (a) Zeolite LTX and (b) Zeolite LTA

The general infrared assignments in structural characterization of the zeolite were done by FTIR to confirm the formation of the zeolite. For zeolite LTX the spectrum exhibited at 1426 cm^{-1} and 968 cm^{-1}. Vibrations associated with double rings of the external T–O linkages occurred at 638 cm^{-1} [5 – 11]. Asymmetric stretching due to the internal vibrations of the framework tetrahedra occurred at 968 cm^{-1} and the band at 426 cm^{-1}. For zeolite LTA, the band attributed to the overlapping of the asymmetric vibrations of Si–O (bridging) and Si–O (non-bridging) bonds occurred at 971 cm^{-1}. Vibrations associated with double rings occurred at 558 cm^{-1} and 519 cm^{-1} whilst the band at 442 cm^{-1} is the vibrations due to the bending of the T–O tetrahedra. According to Mozgawa et al [10] and Aronne et al [11] the two most intense bands of zeolites usually occur at 860–1230 cm^{-1} and 420–500 cm^{-1} agree with the FTIR spectrum of the synthesized zeolites obtained in the present study. The band 1200 cm^{-1} in the spectra represents the presence of substituted Al atoms in the tetrahedral forms of the silica frameworks. T–O bending also occurred at 426 cm^{-1} for LTX, 423 cm^{-1} and 442 cm^{-1} for LTA.

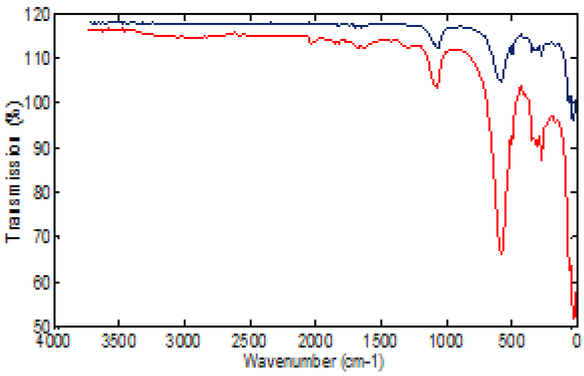


Figure 4: Fourier transformed infrared spectrum for Zeolite LTA (blue) and Zeolite LTX (red).

The elemental composition of zeolite LTA and LTX are presented in Figure 5 and Table 1. The main elements in the zeolite samples were alumino-silicates with minor elements such as copper oxide and sulfur dioxide. Thus the compounds produced were successfully verified.

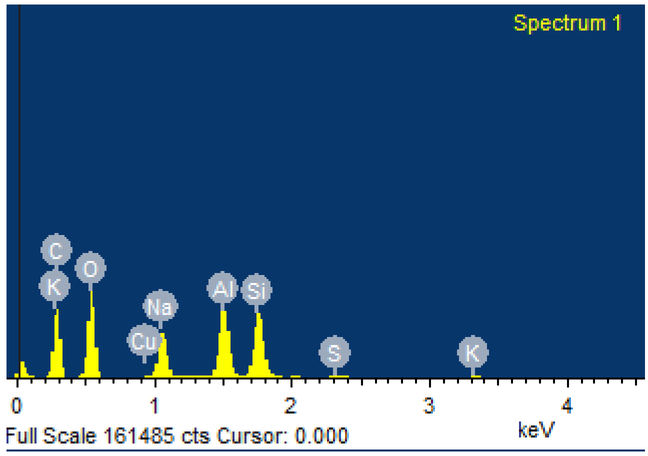


Figure 5: Energy dispersive X-ray spectrum obtained for both zeolites. The percentage composition of each element is shown for Zeolite LTA and Zeolite LTX in Table 1.

Table 1: Elemental composition of zeolite LTA and Zeolite LTX as determined by EDX

Element	**Atomic weight %**	
	LTA	**LTX**
Na_2O	22.11	20.24
Al_2O_3	35.41	36.78
$Si\ O_2$	37.69	37.63
SO_2	0.98	0.50
K_2O	2.56	3.36
CuO	1.26	1.49
Total	**100.00**	**100.00**

3.2 Metal ion concentrations in the samples

Various metal ion concentrations such as nickel, copper, cadmium, zinc, iron, and lead were determined, as shown in Table 2. However, those of the treated raw and the filtered treated with zeolites have not been shown. From Table 2, it was only the ions of Pb and Fe concentrations exceeded the WHO standard levels. Hence, zeolites LTA and LTX were used to treat them both. In other words, only lead and iron recorded very high concentrations in the samples; the rest had concentrations below the detection limit of the AAS. In Figures 5 – 9, metal ion concentrations for the raw, treated raw and the filtered treated have been plotted against the water samples.

Table 2: Metal ion concentration in the water samples

Water Sample	***Metal ion concentration (mg/L)***					
	Pb	Cu	Cd	Ni	Fe	Zn
Stream 1	0.047	0.01	0.002	0.012	1.708	0.005
Well	0.091	0.01	0.002	0.016	0.686	0.007
Stream 2	0.192	0.01	0.002	0.009	2.797	0.005

Figure 6 shows the lead concentrations in the drinking water samples before and after addition of 0.2 g zeolite LTX. All the three raw drinking water samples had lead concentrations above the WHO acceptable limit of 0.01 mg/L. With the raw samples, stream 2 recorded the highest lead concentration of 0.192 mg/L, stream 1 recorded a value of 0.047 mg/L, and the well water had lead concentration of 0.091 mg/L. The treated samples, however, had lead ions concentrations much lower than the acceptable limit of 0.01 mg/L in drinking water. 0.2 g LTX treated samples of raw and filtrate of stream 1 had the same lead concentrations of 0.005 mg/L. 0.2 g LTZ treated raw and filtrate of hand dug well was recorded lead concentration of 0.005 mg/L and 0.017 mg/L respectively. All the three raw drinking water samples had lead ion concentrations above the WHO acceptable limit of 0.01 mg/L. The treated samples also had two samples having their lead concentrations above the acceptable limits except for LTA treated (Figure 7) well water which had a concentration of 0.002 mg/L. This shows that zeolite LTX was more effective in the reduction of the lead concentrations in the drinking water samples used in this study [12, 13]

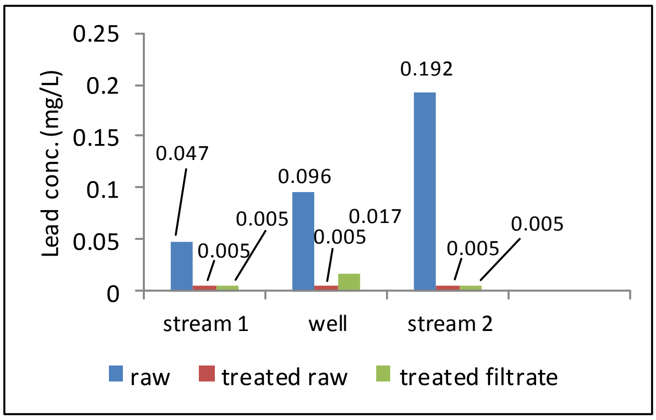


Figure 6: Lead concentrations in samples after 0.2g zeolite LTX treatment

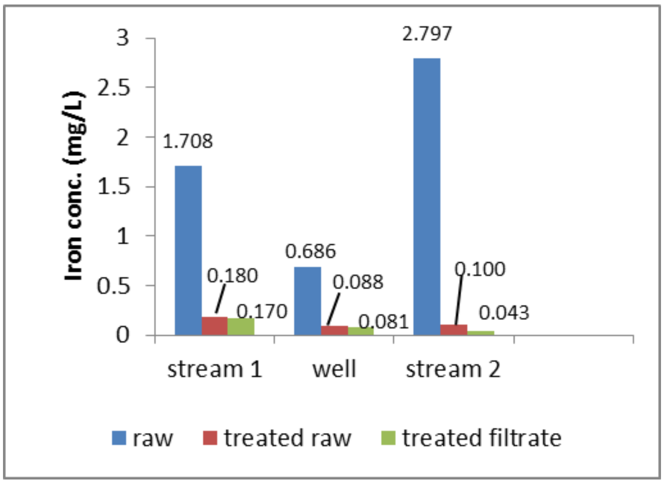


Figure 7: Lead concentrations in samples after 0.5 g zeolite X treatment

For Iron concentrations in the three raw samples as shown in Table 2, all three samples had their iron concentrations above 0.3 mg/L which is the WHO acceptable limit. After treating the samples, the iron concentrations fell within the acceptable limits of 0.3mg/L, per 0. 2g zeolite LTX and 0.5 g zeolite LTX (as in Figure 8 and Figure 10). 0.2 g zeolite LTX treated samples of raw and filtrate of stream 1 had iron concentrations of 0.18 mg/L and 0.17 mg/L, respectively. 0.2 g zeolite LTX treated raw and filtrate samples of the hand dug well water recorded iron concentrations of 0.088 mg/L and 0.081mg/L, respectively. In addition, 0.2 g zeolite LTX treated raw and filtrate of stream 2 recorded iron concentrations of 0.1mg/L and 0.043 mg/L, respectively (Figure 8). The 0.5g zeolite LTX treatment of samples of raw and filtrate of stream 1 had the same iron concentration of 0.244 mg/L. However, 0.5 g zeolite LTX treated raw and filtrate of hand-dug well water recorded iron concentrations of 0.098 mg/L and 0.256 mg/L, respectively. Also, 0.5 g zeolite LTX treated raw and filtrate of stream 2 recorded iron concentrations of 0.165 mg/L and 0.129 mg/L, respectively.

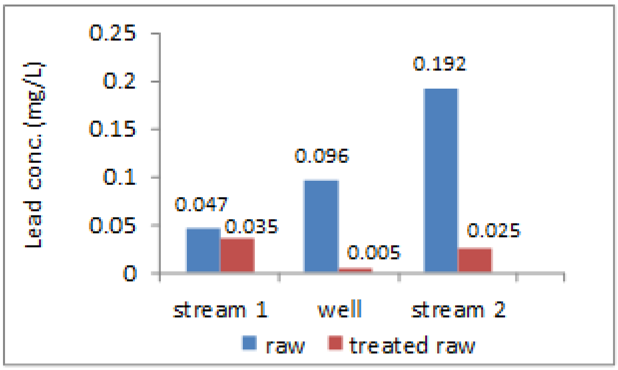


Figure 8: Lead concentrations in water samples after zeolite 0.2 g LTA treatment

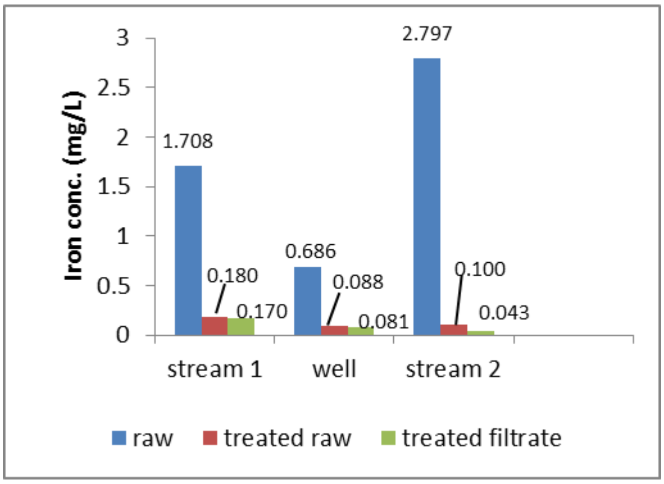


Figure 9: Iron concentrations in samples after 0.2 g zeolite LTX treatment

These means, zeolite LTA treated samples however had 0.012 mg/L, 0.119 mg/L and 0.226 mg/L for treated stream 1, treated well water, and treated stream 2, respectively (Figure 10).

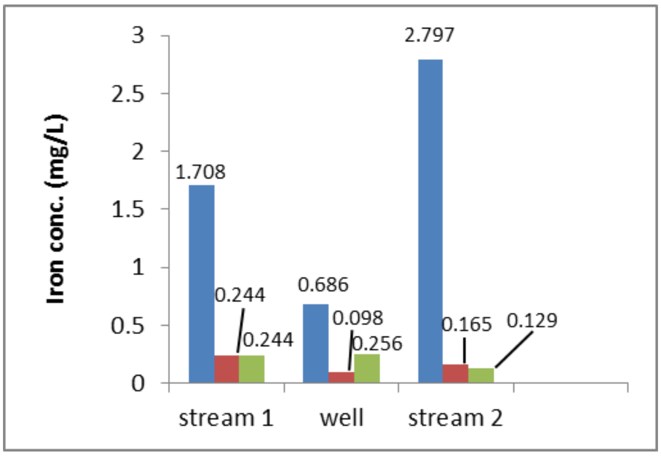


Figure 10: Iron concentrations in samples after 0.5g zeolite X treatment

From the analysis, Zeolite LTA, however, performed better in reducing iron concentrations in the drinking water samples as compared to its performance on reducing lead concentrations in the water samples. To this end, the metal ion removal efficiencies by the two zeolites, LTX and LTA were calculated using the equation:

$$\frac{C_i - C_f}{C_i} \times 100\%$$

Where C_i and C_f are the initial and final concentrations of particular metal ion in the water samples, respectively.

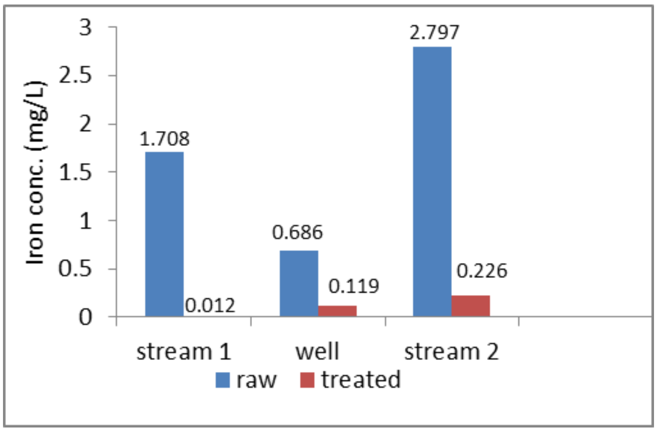


Figure 11: Iron concentrations in samples after zeolite LTA treatment

The values obtained have been shown in Tables 3 – 5. The Lead removal efficiency (Table 3) of 0.2 g LTX in the treated raw samples of stream 1, well and stream 2 were 89.36 %, 94.79 % and 97.4 %, respectively. Those of the treated filtrates for stream 1, well-water and stream 2 were 89.36 %, 82.3 %, and 97.4 %, respectively. Lead removal efficiency of 0.5 g zeolite LTX in the treated raw samples of stream 1, well water and stream 2 were 89.36 %, 94.79 % and 57.8 %, respectively. That of the treated filtrates for stream 1, well-water and stream 2 were 89.36 %, 82.3 %, and 97.4 %, respectively. The lead removals by LTA in the water sample were 25.5 %, 94.8 %, and 85.9 % for stream 1, well-water and stream 2, respectively. Tables 4 and 5 have been used for the comparison. In Table 4, iron removal efficiency of 0.2 g LTX in the treatment of the raw samples of stream1, the well water, and stream 2 were; 89.46 %, 87.2 % and 96.4 % respectively. Those of the treated filtrates for stream1, well-water, and stream 2 were 90.0 %, 88.2 % and 98.46 %, respectively. Iron removal by 0.5 g zeolite LTX in the treated raw samples of stream 1, well-water and stream 2 were 85.7 %, 85.71 % and 94.1 %, respectively. Those of treated filtrates for stream 1, well-water, and stream 2 were; 85.7 %, 62.7 %, and 95.4 %, respectively. The removal efficiency for lead using zeolite LTX in stream 2 had the highest value of 97.4 %. The iron removal efficiency with LTA were; 99.3 %, 82.65 %, and 91.9 % for stream 1, well-water, and stream 2, respectively. It can, however, be noted that, iron removal efficiency with zeolite LTA in stream 1 recorded the highest value of 99.3 % and iron removal efficiency with 0.5 g zeolite LTX in treated filtrate of well-water had a value of 62.7 %. In general, zeolite LTA performed better in reducing iron concentrations as compared with its performance in reducing lead concentrations [16, 20]. On the other hand, with the well water and that from stream 2, the removal efficiency values are extremely high. However, the lead removal efficiency with LTA in stream 1 recorded the least value of 25.5 %. Thus, zeolite removal action favoured a competing cation other than lead [1 – 4]. Another reason could be that the water sample from stream 1 had the highest turbidity compared with the rest of the water sources.

As a consequence, it can be posited that to get very high efficiency applications of zeolites, the water samples could have been filtered from the zeolites, added to a fresh zeolite and the process repeated. This would have removed similar amount of heavy metal ions thus achieving a near 100 % removal efficiency.

Table 3: Lead removal efficiencies of zeolite LTX of masses 0.2 g and 0.5g, respectively

Water Samples		LTX (0.2 g)			LTX (0.5 g)		
		Lead ion concentration			Lead ion concentration		
		C_i (mg/L)	C_f (mg/L)	Removal %	C_i (mg/L)	C_f (mg/L)	Removal %
Stream 1	raw	0.047	0.005	89.4	0.047	0.005	89.4
	filtrate	0.047	0.005	89.4	0.047	0.005	89.4
Well	raw	0.096	0.017	94.8	0.096	0.005	94.8
	filtrate	0.096	0.005	82.3	0.096	0.009	90.6
Stream 2	raw	0.192	0.005	97.4	0.192	0.081	57.8
	filtrate	0.192	0.005	97.4	0.192	0.026	86.5

Table 4: Iron removal efficiencies of zeolite LTX of masses 0.2 g and 0.5 g, respectively

Water		LTX (0.2 g)			LTX (0.5 g)		
		C_i (mg/L)	C_f (mg/L)	Removal %	C_i (mg/L)	C_f (mg/L)	Remova l%
Stream 1	raw	1.708	0.18	89.5	1.708	0.244	85.7
	filtrate	1.708	0.17	90	1.708	0.244	85.7
Well	raw	0.686	0.088	87.2	0.686	0.098	85.7
	filtrate	0.686	0.081	88.2	0.686	0.256	62.7
Stream 2	raw	2.797	0.1	96.4	2.797	0.165	94.1
	filtrate	2.797	0.043	98.4	2.797	0.129	95.4

Table 5: Lead removal efficiencies of zeolite LTA of mass 0.2 g

Samples	Lead concentrations (mg/L)			Iron concentration (mg/L)		
	C_i (mg/L)	C_f (mg/L)	Removal %	C_i (mg/L)	C_f (mg/L)	Removal %
Stream 1	0.047	0.035	25.5	1.708	0.012	99.3
Well	0.096	0.005	94.8	0.686	0.119	82.7
Stream 2	0.192	0.025	85.9	2.797	0.226	91.9

4. Conclusion

The removal of lead (Pb^{2+}) and iron (Fe^{2+}) ions in drinking water samples from Ghana was successful with a selective ion exchange process. The removal efficiency of lead in both treated raw and treated filtrate of stream 2 had the highest value of 97.4% by 0.2 g zeolite X and that of the 0.5g zeolite X. Treatment of the raw of the same stream 2 recorded a lower removal efficiency of 57.8% for lead. This shows that an increase in the mass of zeolite does not necessarily increase the removal efficiency. The removal efficiency of lead by zeolite LTA, however recorded the least value of 25.5% in stream 1. This can be due to a competition between co-existed ions, which favoured another metal ion than lead. In general, zeolite X was better in removing and reducing lead concentrations as compared with zeolite LTA. The iron (Fe) removal efficiency of zeolite LTA in stream 1 had the highest value of 99.3% and that of 0.5g zeolite X treated filtrate of well-water had the least value of 62.7%.

In general, zeolite LTA performed better in removing iron as compared with zeolite LTX. It can also be noted that filtration process to remove suspended solids from the water samples before treatment with zeolite was highly effective.

This work has also confirmed that, one zeolite cannot effectively remove all cations from drinking water samples hence, treating water samples with alternate zeolites and a combination with other water treatment methods might enhance the quality of water. This research has also shown that, zeolite have the capacity to reduce colour values in contaminated drinking water samples to appreciable values. Alternatively, the water samples can be filtered and added to a ‘fresh’ zeolite and the process repeated until all heavy metal ions are removed.

5. Acknowledgement

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Author Profile

Dr. Baah Sefa-Ntiri now is a Lecturer in the Department of Physics, University of Cape Coast. He specializes in water treatment technologies using nanotechnology applications.

Dr. Bright Kwakye-Awuah now is a Lecturer in the Department of Physics, Kwame Nkrumah University of Science and Technology, Kumasi, head of the Materials Science Research Group in the Department, Chief Executive Officer of ZeoTeC Company Limited, the sole producer and distributer of synthetic zeolites in Africa, Member of the British Zeolite Association and Member of the Ghana Science Association. He obtained his BSc in Physic, from the University of Cape Coast, Ghana, MSc in Environmental Physics, from the University of Bremen, Germany and PhD from the University of Wolverhampton, United Kingdom. He has authored over 10 publications in international Journals on zeolite science and technology. He is an Editorial Board Member of World Research Journal of Antimicrobial Agents, Journal of Materials Sciences and Applications and American Journal of Materials Research.

Mr P. K. Mensah Amoah now is a Lecturer in the Department of Physics, University of Cape Coast. He specializes in the laser and fiber optics applications to water treatment.

Professor Craig Williams now is a Professor of Microporous and Mesoporous Materials in the Faculty of Science and Engineering, University of Wolverhampton, UK.