Vol. 39(2), pp. 165-176, Dec. 2020
ISSN 1821-536X (print)
ISSN 2619-8789 (electronic)

Tanzania Journal of Engineering and Technology

Full Length Research Paper

Effects of Calcination Temperature of Naturally Occurring Absorbents on Drinking Water Defluoridation

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ABSTRACT

Currently, in Tanzania, fluoride removal from drinking water is treated mostly using the bone char method. The method has poor acceptability in some religious communities and also causes water quality deterioration in taste and odour if the bones are not properly prepared. The use of local natural adsorbents as an alternative is feasible with limitations of high levels of other impurities in treated water. Locally available gypsum, magnesite and bauxite were converted to adsorbents through calcination. The study was conducted to determine the removal efficiency, best calcination temperature and composite ratio of the three adsorbents for the removal of fluoride from natural drinking water with fluoride concentration as high as 16.7 mg/L. The adsorbent materials were calcined at different temperatures ranging between 350^{0}C and 600^{0}C. Batch experiments were performed and samples were collected at different contact time intervals of 2 minutes to 60 minutes, and residual fluoride was determined. Bauxite had the highest fluoride removal efficiency compared to gypsum and magnesite. The best calcination temperatures were 350^{0}C, 400^{0}C, 600^{0}C for gypsum, bauxite and magnesite, respectively. The best calcination temperatures were used to prepare composites at different ratios of 1:2:3, 2:3:1 and 3:2:1, bauxite: gypsum: magnesite respectively. All the ratios gave low sulphate and iron as impurities within the recommended standards. The composites lowered fluoride concentration level to 1.53 mg/L, 2.07 mg/L, 2.60 mg/L for 1:2:3, 2:3:1, 3:2:1 ratios, respectively. In conclusion the study reveals that, it is possible for composites made of adsorbent calcinated at different optimum temperatures to give good results in fluoride removal from drinking water, as well as standard pH, iron and sulphate values in treated water.

Keywords: *Fluoride, Bauxite, gypsum, magnesite, composite ratio, adsorbent.*

INTRODUCTION

Fluoride is the pollutant that is persistent and cannot be degraded and has the ability to accumulate in the soil, plants, animals and also in human beings body (Tomar and Kumar, 2013). In different parts of the world, contamination of drinking water with excess fluoride content has been described as one of the public health problem (Lavecchia *et al*., 2012). Water with excess fluoride and other chemical and biological contents above the recommended drinking standards is not suitable for domestic use (Rao, 2003). Moderate amount of 1 mg/L of fluoride is of importance in human dental health by preventing dental caries, but too low fluoride concentration is serious especially in children health growth in development of their tooth enamel since it is considered to be less developed at the childhood stage (Owusu-Agyeman *et al*., 2018). Fluoride plays a major role in bone and dental mineralization, but when it is in excess it can cause severe and serious health effects such as dental fluorosis, skeletal fluorosis and long term damage of thyroid, liver, kidney and brain (Lavecchia *et al.*, 2012).

Fluoride in water is unable to bind with cations such as aluminium and iron because of their low reactivity in natural water, hence fluoride occurs as a free ion. Fluoride can occur naturally or due to human activities. Naturally, its occurrence is mainly related to geogenic processes, which govern its concentration in groundwater. It is affected through the contact with the sedimentary carbonates (Sivasankar *et al*., 2016). Moreover, the aquifer characteristics can also contribute to presence of fluoride contamination into groundwater after a prolonged contact with aquifer fluoride rich minerals. In aquifers of volcanic formation, mineral dissolution is inhibited due to the low temperature, high altitude and high transmissivity features of the aquifer hence resulting in low fluoride concentration. For the case of sedimentary formation aquifers they are characterised by low altitude and high temperature which facilitates dissolution of minerals and allow precipitation of CaF_2 hence leading to high fluoride concentration in groundwater (Shen *et al.*, 2016)

Volcanic activities are one of the natural sources of fluoride contribution to the environment, whereby varieties of gases containing HF, NH_4F, SiF_4, $(NH_4)_2SiF_6$, $NaSiF_6$, K_2SiF_6 and KBF_4 and few other minor constituents such as sulphate can be emitted when volcanic activities takes place (Borgnino *et al*., 2013). Also, a significant amount of soluble compounds including fluoride are being released into water when the fresh-erupted tephra gets in contact with water resulting to the contribution of fluoride concentration in water (Ayris and Delmelle, 2012).

Fluoride pollutants can be present in air, dental products, food and beverages, soil as well as in water (Fawell *et al.*, 2006). As a result of human activities, in the process of manufacturing different products, the industries can release fluorine to the environment either in form of gas such as HF, SiF_4, F_2, and H_2SiF_4 or in a form of particulate matters that is CaF_2, NaF, and Na_2SiF_6 (Ozsvath, 2015).

The problems of excessive fluoride amounts in drinking water are highly endemic and hence they have encouraged and motivated more researchers to explore different methods and materials that can be used to solve the problem (Rao, 2003). Over years, as a result of different research conducted in different places worldwide, there have been development of techniques for removal of fluoride (Al-Hawamdeh *et al*., 2013).

Fluoride removal techniques include Nalgonda technique, reverse osmosis and nano-filtration, electro-coagulation technique and precipitation. Similarly using different adsorption materials such as activated alumina, bone char, clay and aluminium have been proven to be effective. However, most of them appeared to be neither sustainable nor effective in remote rural regions of developing countries mainly due to their high cost and dependence on skilled personnel for maintenance (Cherukumilli *et al.*, 2017).

These techniques face setbacks whereas some are not cost effective and others are less expensive but less effective with low quality of the treated water as well as poor sorption capacity (Al-Hawamdeh *et al.*, 2013; Thole and Mtalo, 2012). Each technique has its benefits and shortcomings that limit its use. For instance, membrane separation process which includes reverse osmosis and nano-filtration, are very efficient but it suffers from high energy consumption and membrane fouling, hence making it expensive, whereas chemical precipitation tend to produce large amount of sludge (Cherukumilli *et al.*, 2017). Among the setbacks of Nalgonda method is the formation of sludge, high chemical dosage, requires a technical personnel to operate and it is not suitable for water with high fluoride content greater than 10 mg/L, (Wambu *et al.*, 2014). The use of bone char, interferes with some taboos and religious beliefs of some societies hence it is not accepted globally and when not properly prepared produces low water quality with taste and odour of rotten meat to drinking water. Bauxite and clay increases water turbidity, colour as well as some of the residual ions (Thole and Mtalo, 2012; Feenstra and Erkel, 2007). Despite activated alumina method being popular, it has some limitations such as it is not effective when the total dissolved solids in water exceeds 1500 mg/L. It is also pH selective hence it works better under certain pH levels and when it is left out of operation for 2 or 3 days it provides room for microorganism growth (Shrivastava and Vani, 2009).

There is no single method of treating drinking water which meets all the standards. Therefore, there is a need for obtaining knowledge on the removal of fluoride using resourceful techniques under ideal conditions using different naturally occurring materials. The current research aims at improving the fluoride removal process in drinking water using the naturally occurring rock materials using bauxite, gypsum and magnesite obtained in Tanzania. The scope of the work was the calcination of bauxite, gypsum and magnesite at temperature of 350 to 600^0C to improve their adsorbent capability.

MATERIALS AND METHODS

Boulders of magnesite, gypsum and bauxite sourced from Chambogo, Makanya and Lushoto, respectively, were crushed and milled separately and then sieved through standard sieves of ≥ 0.075 mm. The materials were then calcinated in a furnace at different temperatures of 350^oC, 400^oC, 450^oC, 500^oC, 550^{0o}C and 600^oC for duration of 2 hours. The samples were then removed from the furnace and left to cool to room temperature.

Different dosages of 1 to 10 grams of bauxite, gypsum and magnesite were weighed separately using a weighing balance, and then placed in the beaker containing 100 mL of 16.7 mg/L fluoride contaminated water obtained from Maji ya Chai river in Arusha. Samples were collected at location with coordinates -3.37111 S and 36.8964 E, using the polyethylene sampling bottles, then was transported to the laboratory and stored at room temperature of 25^oC as further analysis was on going.

Using the magnetic stirrer at 100 rpm, the material and fluoride contaminated natural water was allowed to stay in contact while stirring at different time intervals for the first 2, 4, 6 ,8 and 10 minutes and then for 20, 30, 40, 50 and 60 minutes. At the end of each contact time, the water sample was filtered, then pH, fluoride concentration and ionic impurities concentration, iron and sulphate parameters were analysed. This was performed at each calcinated temperature. The calcination temperatures at which fluoride removal was the highest for each adsorbent were then mixed together at the ratios of 1:2:3, 3:2:1 and 2:3:1 as mass ratios, bauxite: gypsum: magnesite, respectively. The fluoride removal efficiency by each adsorbent bauxite, magnesite and gypsum, respectively was calculated using the equation.

$$R = \frac{Ci - Cf}{Ci} \times 100\% \;\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where R is removal efficiency, Cf and Ci are the final and initial fluoride concentration in solution (mg/L) respectively. The removal capacity was determined using the equation (2).

$$qe = \frac{(Co - Cf)V}{m} \;\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where q_e is the amount of adsorbed fluoride at equilibrium (mg/g), Co and Cf represents the initial and final fluoride concentration (mg/L), V is the volume of solution (L) and m the mass of adsorbent in grams (g).

The adsorption isotherm was studied by fitting the data with Langmuir and Freundlich isotherms as expressed in equations (3) and (4). In Langmuir isotherm adsorption is assumed to occur at specific homogeneous sites where there is no additional adsorption to take place (Piccin *et al.*, 2011).

$$\frac{1}{q_e} = \left[\frac{1}{Q_m K_l}\right]\frac{1}{C_e} + \frac{1}{Q_m} \;\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where, qe is adsorption capacity (mg g^{-1}), Ce is Equilibrium concentration (mg L^{-1}), Q_m is maximum monolayer adsorption (mol g^{-1}) and Kl is Langmuir constant (L mol^{-1}).

The Freundlich isotherm assumes a multilayer adsorption, mostly used to define adsorption characteristics for heterogeneous surfaces (Kebede *et al.*, 2016).

$$Q = FC^{1/n} \;\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Where Q is adsorption Capacity (mg g^{-1}), F is Freundlich constant (Lkg^{-1}), C is Equilibrium concentration (mg L^{-1}) and 1/n is heterogeneity factor (dimensionless).

RESULTS AND DISCUSSION

The raw materials, which are bauxite, magnesite and gypsum, were collected from Usambara Mountains in Lushoto district, Chambogo in Same district and Makanya in Same district, respectively. The materials contained some sulphite and metal oxide as impurities (Table 1). Bauxite contained about 57.4% of Al_2O_3, gypsum contained 29.4% of CaO and magnesite contained about 47.0% of MgO. These are the major compounds in the raw materials used production of absorbents.

Table 1: Analysis of the material elements (XRF) results in percentage

Material Constituent	Al_2O_3	SiO_2	CaO	MgO
Bauxite	57.41	4.18	<0.01	<0.01
Gypsum	2.07	8.98	29.36	1.27
Magnesite	0.12	2.30	0.22	47.03

Material Calcination

When bauxite (Aluminium hydroxide), gypsum (calcium sulphate dihydrate) and magnesite (magnesium carbonate) were calcinated, the following reactions took place as observed in the equations (5), (6) and (7).

$$2Al(OH)_{3(s)} \xrightarrow{\Delta} Al_2O_{3(s)} + 3H_2O_{(g)} \;\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

$$CaSO_4.2H_2O_{(s)} \xrightarrow{\Delta} CaO_{(s)} + SO_{3(s)} + 2H_2O_{(g)} \;\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

$$MgCO_{3(s)} \xrightarrow{\Delta} MgO_{(s)} + CO_{2(g)} \;\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

Laboratory Analysis

The water quality parameters analysed from the raw water were found to be within WHO and TBS standards except for fluoride which was found to be much higher compared to the standards (Table 2). Different conditions and factors affecting the fluoride removal were observed and the studied factors such as particle size, contact time, dosage of the adsorbent, and calcination temperature were observed. Likewise, the removal capacity of each adsorbent was determined, the best temperature for media calcination and the best composite ratio was also determined through the analysis.

Table 2: Analysis results of raw water

Parameter	Amount in raw water	WHO Standards	TBS Standards
Fluoride (mg/L)	16.7	1.5	1.5-4.0
Chloride (mg/L)	29.9	250	200-800
Nitrate (mg/L)	7.6	50	10-75
pH	8.14	6.5-8.5	6.5-9.2
Sulphate (mg/L)	BDL	500	200-600

Effect of Dosage

Analysis was conducted to establish removal efficiency within 10 minutes using different concentration dosages. Different doses of 1 g to 10 g of adsorbent in 200 ml contaminated water with initial fluoride concentration of 16.7 mg/L were used. Removal efficiency under different dosage conditions was as shown in Figure 1. Similar trend of removal efficiency was observed by (Patnaik *et al.*, 2016). The removal efficiency was observed to increase from 36.5% to 87.7% with increased adsorbent dosage from 1 to 10 g. The increase in fluoride removal efficiency with increase in the adsorbent dose was due to the increase in surface area; hence more active sites were available for adsorption of fluoride. However after a definite adsorbent dose, the percentage of removal did not increase significantly and that dose was considered as best dose which was 10 g.

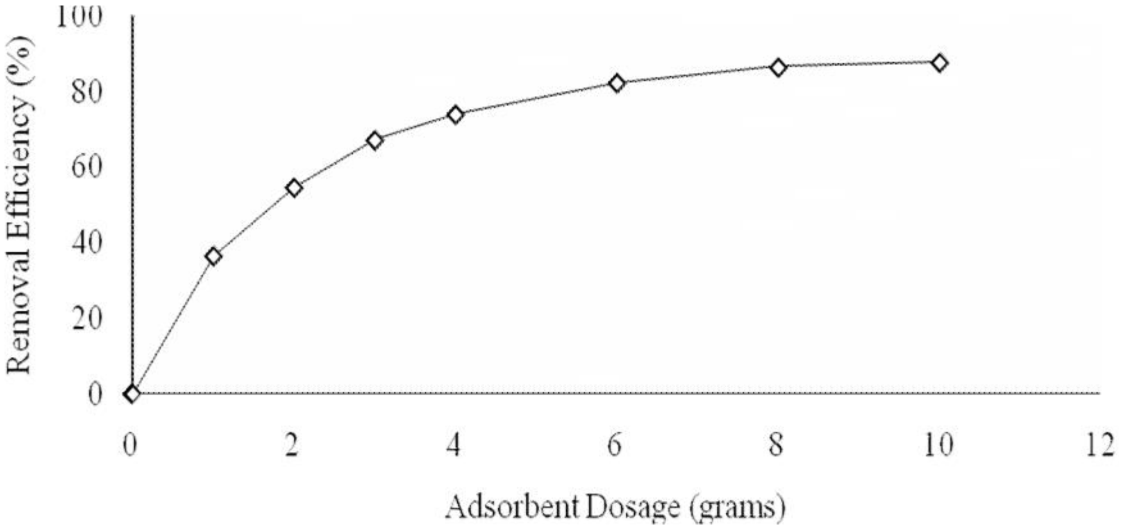

Figure 1: Adsorbent dosage in relation with fluoride removal efficiency

Effect of Adsorbent Dosage on Fluoride Removal Capacity

Figure 2 shows that the removal capacity kept decreasing as the dosage increased at all the contact time. The same trend of result was observed by Patnaik *et al.* (2016). The adsorption process was observed to take place in the first 2 minutes contact time of the reaction. At 10 minutes contact time where the highest removal capacity was observed, the equilibrium removal capacity decreased from 1.13 mg/g to 0.27 mg/g, this was also observed at the contact time of 2, 4, 6, and 8 minutes where removal capacity decreased from 0.9 mg/g to 0.26 mg/g, 0.98 mg/g to 0.27 mg/g, 1.06 mg/g to 0.27 mg/g and 1.08 mg/g to 0.27 mg/g respectively. The removal capacity was observed to decrease to 0.27 mg/g of fluoride as the dosage increased. That is more fluoride ions were adsorbed as the adsorbent dose increases, hence adsorption capacity reached was 0.27 mg/g of fluoride. After reaching 0.27 mg/g, fluoride removal seized. Desorption will begin depending on initial concentration and media volume/weight used.

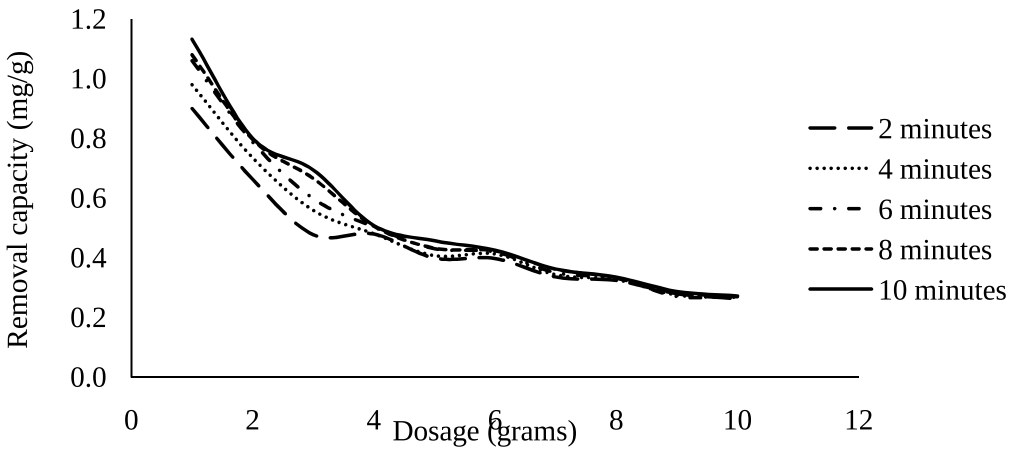

Figure 2: Effect of adsorbent dosage on the fluoride removal capacity at different contact times

Figure 3 shows bauxite had highest removal capacity in all calcination temperatures. Gypsum removal capacity was high at lower calcination temperature of 350^oC and poor at higher calcination temperature of 600^oC. Magnesite had highest capacity at higher temperatures of 600^oC. This was also reported by Singano (2000), that with magnesite fluoride removal was high at higher calcination temperatures. When magnesite is calcinated the decomposition of carbonates takes place and forms magnesium oxide MgO, which is the one responsible for fluoride removal. The decomposition of the carbonates occur starting at temperature of 400^oC and hence proceeds rapidly above temperature of 500^oC (Singano, 2000). Magnesite reaction on fluoride removal can be seen in equation (8).

$$MgO_{(s)} + H_2O_{(l)} + F^-_{(aq)} \longrightarrow Mg(OH)F_{(aq)} + OH^-_{(aq)} \quad \ldots\ldots (8)$$

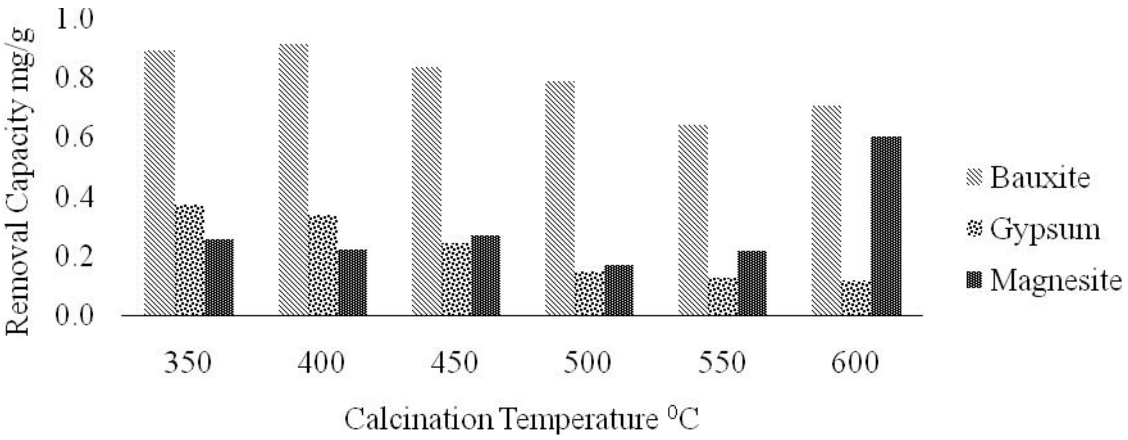

Figure 3: Removal capacity of each adsorbent at different calcination temperatures

Despite the high removal capacity, bauxite increased the water turbidity and colour in the treated water. Also, bauxite and gypsum lowered the pH value to 8.04 and 8.06, respectively, while magnesite raised pH to 8.62 as also reported by Thole *et al.* (2013). The rise of pH by magnesite was also reported by Singano (2000). Bauxite and gypsum lowering pH can be explained by the presence of sulphite in bauxite and gypsum.

The fluoride removal in drinking water using bauxite is through ion exchange processes where fluoride ion with OH^- groups they exchange. Aluminium oxide (Al_2O_3) being in larger content in bauxite as well as sulphite SO_3^-, these are covalent oxides, hence when dissolve in water they bind the water molecules and releases protons which results to the lowering of pH (Cherukumilli *et al.*, 2017; Thole *et al.*, 2013).

Magnesite is mainly $MgCO_3$ compound, when magnesite is calcinated the decomposition of carbonates takes place and forms magnesium oxide MgO which is the one responsible for fluoride removal and the cause of pH rise. The decomposition of the carbonates occur starting at temperature of 400^{0}C and hence proceeds rapidly above temperature of 500^{o}C (Singano, 2000).

Moreover, gypsum calcinated at low temperature of 150^{o}C to 300^{o}C gave better performance in fluoride removal though with the highest residual number of other ions such as sulphate and iron, which are not within the recommended standards. However, gypsum calcinated at temperatures above 300^{o}C introduces less ionic contaminants in treated water (Thole *et al.*, 2011). Results in Table 3 prove the presence of residual sulphate in gypsum calcinated at temperature of 350^{o}C, but the amount is found to be within the recommended standards and iron level was found to be below the detection limit.

Surface area was determined using BET (Brunauer–Emmett–Teller) method and pore diameter was determined using (Barrett-Joyner-Halenda) BJH method. Magnesite prepared at 600^{0}C had a smaller surface area of 83.1 m^2/g compared to bauxite with 103 m^2/g and gypsum 115.5 m^2/g. Bauxite prepared at 400^{0}C had the largest pore diameter of 32.4 Å compared to magnesite 31.8 Å and gypsum with 32.3 Å. The highest removal efficiency for bauxite, gypsum and magnesite were observed at temperatures of 400^{0}C, 350^{0}C and 600^{0}C, respectively. Similarly, Singano (2000), reported best fluoride removal capacity of magnesite to be at 600^{0}C. For the case of gypsum, performance was observed to be best at lower temperature as reported by Thole *et* *al*. (2011), hence 350^0C calcination temperature for gypsum was best in this study.

The composite used comprised adsorbents with best performance calcination temperatures that is bauxite calcinated at 400^0C, gypsum at 350^0C and magnesite calcinated at 600^0C. The ratios used were 1:2:3, 2:3:1 and 3:2:1 bauxite: gypsum: magnesite, respectively. The results are shown in Table 4.

Table 3: Treated water analysis results

Material	Initial Fluoride (mg/L)	Final pH	Fluoride (mg/L)	Iron (mg/L)	Sulphate (mg/L)
Bauxite	16.7	8.04	8.5	1.55	24
Gypsum	16.7	8.06	14.4	BDL	190
Magnesite	16.7	8.62	14.7	0.75	18
WHO (2006) Standards		6.5-8.5	1.5	0.3	500
TBS (2009) Standards		6.5-9.2	1.5 – 4.0	0.3-1.0	200-600

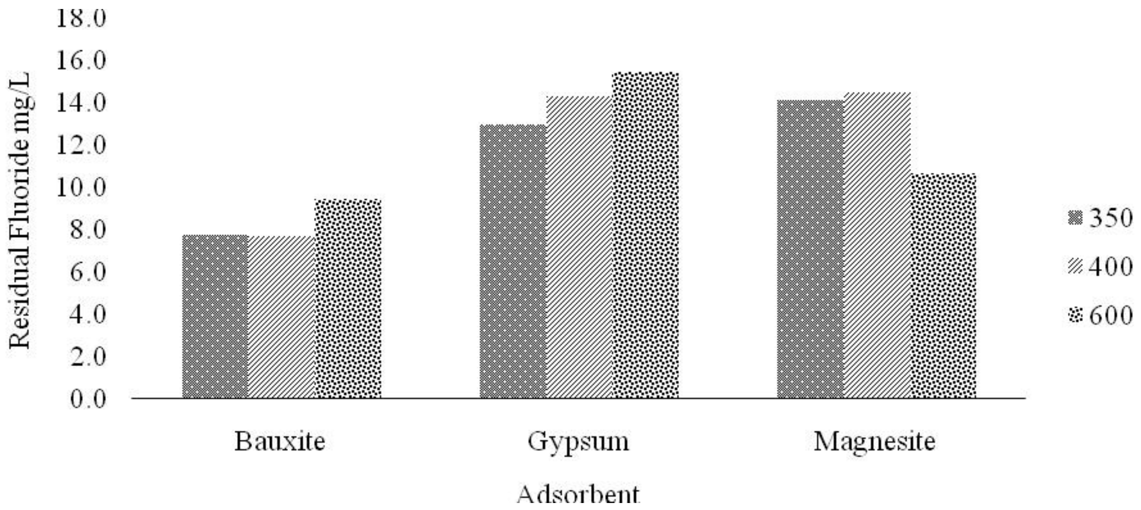

Figure 4: Effect of adsorbent calcination temperature on fluoride removal

Table 4: Treated water test results of the composite ratios

Parameter	Composite Ratio 1:2:3	Composite Ratio 2:3:1	Composite Ratio 3:2:1	WHO Standards	National Standard (TBS)
Removal Efficiency (%)	90.9	87.6	84.4		
Fluoride (mg/L)	1.52	2.07	2.6	1.5	1.5-4.0
Sulphate (mg/L)	175	165	168	500	200-600
Iron (mg/L)	BDL	BDL	BDL	0.3	0.3-1.0

All values were within the WHO and TBS allowable drinking water quality standards.

Figure 5 illustrates the residual fluoride with the respective ratios.

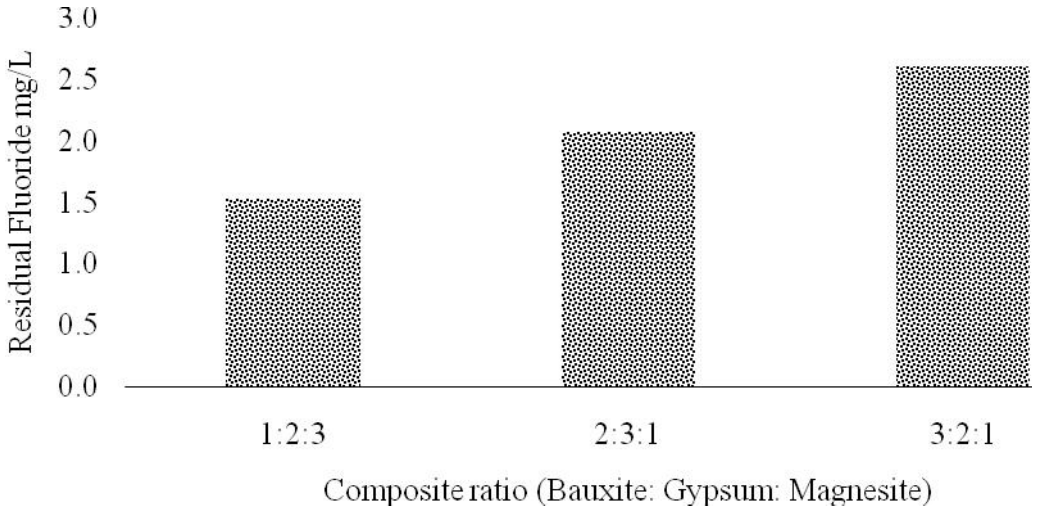

Figure 5: The effect of composite ratio on residual fluoride

Adsorption Isotherms

Composite ratio 1:2:3 bauxite, gypsum and magnesite, respectively, as shown in Figures 6 and 7 demonstrate sorption isotherms obtained from the experimental data fitted. Most of the data set adhered more strongly to Langmuir sorption model which gave the value of $r = 0.00575$ which lies in the range $0 < r < 1$ which indicates a favourable adsorption. For Freundlich model, the value of 1/n was 0.0521 in which the value of n was greater than 10 hence out of range.

The results obtained from fitting the experimental data into the Langmuir and Freundlich equations have been summarized in Table 5, whereas when r value lies in the range $0 < r <$ indicates a favourable adsorption and n value indicates good adsorption characteristics when is in range 2 to 10.

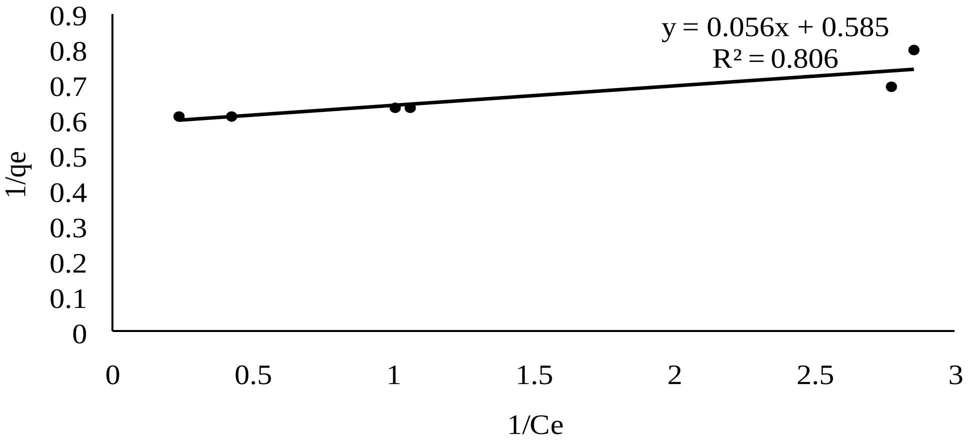

Figure 6: Langmuir isotherm for composite ratio 1:2:3

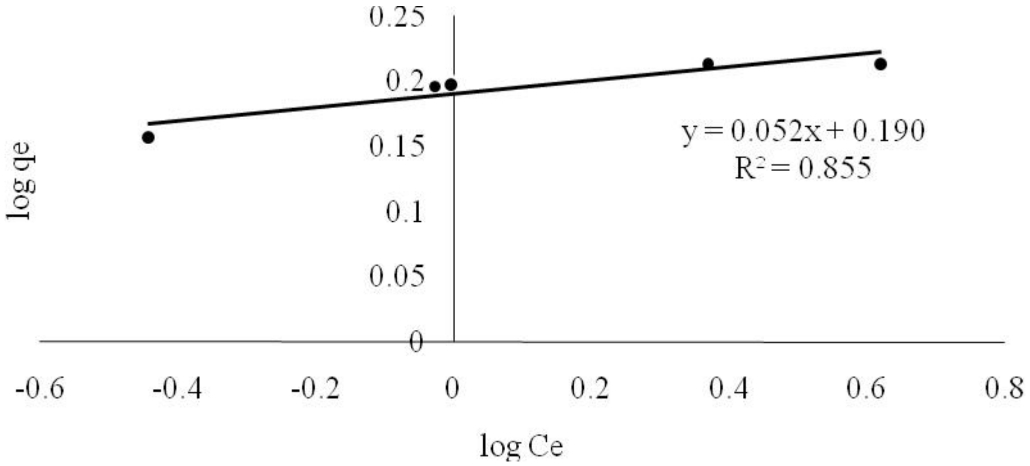

Figure 7: Freundlich isotherm for composite ratio 1:2:3

Table 5: Fitted parameters for Langmuir and Freundlich isotherms of the composites

	Langmuir Isotherm				Freundlich Isotherm	
	R^2	R	$1/K_LQm$	1/Qm	1/n	R^2
1:2:3	0.8065	0.0058	0.0566	0.5859	0.0521	0.8554
2:3:1	0.9697	0.1895	0.1528	0.5965	0.1308	0.9913
3:2:1	0.9525	0.1022	0.3096	0.5886	0.1772	0.9724

CONCLUSIONS

Drinking water defluoridation with the composite materials of bauxite, gypsum and magnesite is possible and provides the best results, where the dosage of the adsorbent is inversely proportional to the removal capacity with constant initial fluoride concentration. The adsorption process was observed in the first 2 minutes contact time of the reaction.

Bauxite gave the highest fluoride removal efficiency compared to gypsum and magnesite at all calcination temperatures tested, though it altered the water quality parameters such as pH, hence with the composite of the three adsorbents the water quality parameters were balanced.

Calcination temperatures of 350^0C for gypsum, 400^0C for bauxite and 600^0C for magnesite were found to be the best in the removal of fluoride. The findings illustrate that the adsorbent material composite of the best calcination temperatures gave the highest removal efficiency at all the ratios tested; also, all gave the lowest sulphate and iron levels as impurities.

The best composite ratio 1:2:3 bauxite, gypsum and magnesite respectively had the highest removal efficiency with low sulphate and iron levels which were found to be within the recommended WHO standards. Whereas adsorption isotherms of Langmuir model had the best fit, the adsorption characteristics were good and the adsorption was favourable. Hence best for the removal of excess fluoride in drinking water.

Therefore, using the naturally occurring materials for the case of bauxite, gypsum, magnesite and its composite of best calcination temperature of each adsorbent, can be used as a treatment technology of drinking water in highly contaminated fluoride regions when upscaled to large municipal treatment plants.

AKNOWLEDGEMENT

Our sincere gratitude and thanks to the Almighty God for his guidance, protection, strength and help throughout the research work. The authors are grateful to DAFWAT project for the support in the laboratory research activities that has led to this output. Thank you and may God Almighty bless you.

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