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Experimental Analysis of Absorptive Removal of Dissolved Fluoride Ion using Bone Char

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ABSTRACT

Fluoride is notably occurring in drinking water sources, and has a considerable effect on human health.Failure to remove excess fluoride in drinking water leads to dental and skeletal fluorosis and fluoride toxicity that can cause non-skeletal diseases. The objective of this research is to determine whether or not the dissolved fluoride level in water could be reduced to acceptable levels by bone char. The bone was burned at 500ºC, for 2 hr to produce the bone char. After charring, the bone was crushed at different size and sieved into a different wire mesh. Generally the research study design was focused on experimental analysis including effect of pH, contact time, dose removal efficiency of the bone char. It was found that the highest fluoride removal (85.39%) using smallest size of bone char media, (0.6 mm), with 25g dose on fresh water containing 20.6 mg/l of fluoride socked for 90 min. Hence, in this study a 25 g bone char dosage/ bone char media with the minimum grain size of 0.6mm showed the maximum removal percentage. This depicts that it is promising to use bone char as an adsorptive media to remove or reduce the fluoride ion in drinking water to allowable level.

Keywords: Bone char, Fluoride, Defluoridation, Adsorption Capacity, Calcination

INTRODUCTION

Fluoride is an ion of the chemical element fluorine, which belongs to the halogen group. It is the most electronegative of all the elements and it is never found in elemental gaseous form except in industrial processes (Kaseva, 2006). Fluoride is notably occurring in drinking water sources, especially in groundwater sources. Consumption of water with high fluoride concentration exposes for risk of fluorosis (Assefa, et al., 2015). This problem faces many country including India, Sri Lanka, China, the Rift Valley in East Africa, and parts of South Africa (Coetzee, 2006).

The East African Rift Valley, which cuts through Ethiopia, is geomorphologically still an active volcanic region. The volcanic rocks, particularly in the young basalt, contain high concentrations of fluoride and fluorapatite (Assefa, et al., 2015). Large fault systems in the Valley create conditions that allow very deep percolation of infiltrating surface water. The floor of the Rift Valley that is characterized by high hydrothermal activity accelerates the solubility of fluoride. The water supplies in the Ethiopian Rift Valley region come mainly from boreholes with depths from 10 to 100 meter (Haimanot, et al., 1987). Though the concentration of fluoride ion in the groundwater varies from place to place and depth based on the nature of the geologic formation, in general it is beyond the WHO standard in most parts of the rift valley system (Gultu and Belayneh 2018), The hot climate and high fluoride waterbed of the Rift Valley, therefore, favor the development of endemic fluorosis. In the Ethiopian rift valley basin the dominant sources of domestic water supply is groundwater, which resulted in an estimated 8 million people exposed to high levels of naturally occurring fluoride (Datturi, et al., 2017).

MATERIALS AND METHODS

Description of the Study Area

Hawassa is a city in SNNPR on the shores of Lake Awasa in the Great Rift Valley. It is located 285 km south of Addis Ababa, 130 km east of Sodo, and 75 km north of Dilla. The town serves as the capital of the Southern Nations, Nationalities, and Peoples' Region, and is a special zone of this region. It lies on latitude and longitude of 7°3′N 38°28′E Coordinates:

7°3′N 38°28′E and an elevation of 1708 meters above sea level (the Southern Nations, 2014).

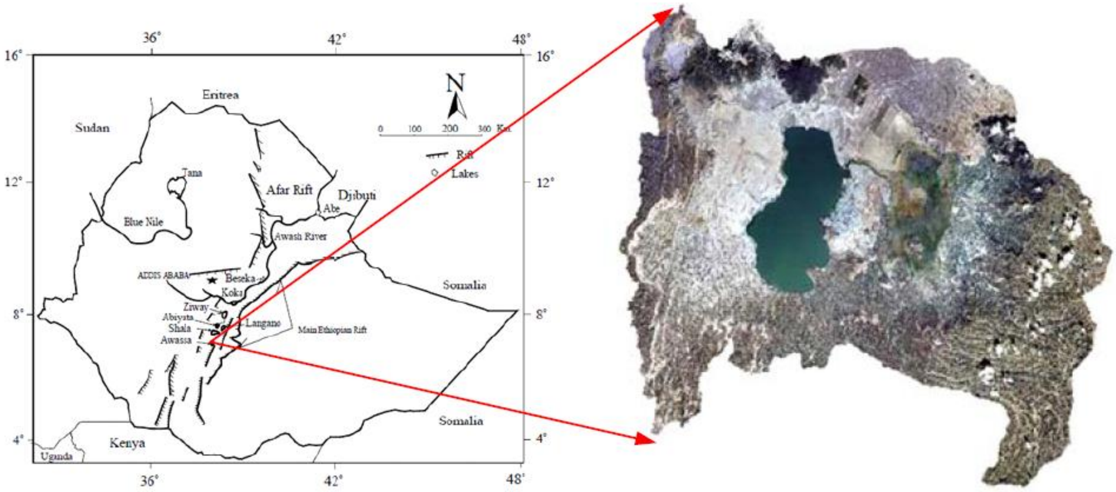

Fig1. *Map of the study area (Belete , et al., 2017)*

Method of Data Collection and Data Analysis

Water samples were taken from four different sites of Hawassa town groundwater from Cheffe formerly an extension of lake Hawassa and currently changed to swampy, Datoa residential site on the North Eastern part of the town and Korem sefer down town of the surface water was taken from Tikur Wuha which is the only perennial river that flows in to Lake Hawassa. Fresh cow bone which is not stay long in soil was used to prepare the bone char (Fig. 1).

Figure2. *Fresh cow bone used for bone char preparation*

Data analysis was done by evaluating the parameter processing by using ANOVA. All analysis of variance methods are based on the assumptions of normally distributed and independent variables with SPSS. Generally the research study design was focused on experimental analysis including effect of pH, effect of contact time, effect of initial concentration, effect of dose and effect of grain size were studied.

In addition to this, data analysis was done by evaluating the literature and processing the collected data from site in the laboratory by filtration mechanism. Primarily the data organized as keeping the procedure of treatment systemthe collected cattle bones were cleaned from flesh remnants, lipids and tendons, and then open dried in the sun for one week (Puangpiny & Osiripha,2015).

The open dried cow bone was broken in to smaller pieces so as to place it in the furnace. The heat treatment removes the organic matter, which adds unnecessary taste and colour to water. Heating thus makes the bone char hygienically acceptable for defluoridation. In this study heating of the bones in the kiln was controlled and kept to 500^0C for 2 hour, because temperatures higher than 500^0C may damage the apatite structure (Posner, 1987), resulting in poor fluoride removal, while temperatures below 400^0C may result in a bad taste and odour on the treated water (Kaseva, 2006). Atmospheric air was flushed into the furnace chamber from room temperature for approximately 1 hour. Then the firing is stopped and the furnace-gate opened gradually to cool down (Eli, 2015).

The bone char was crushed and sieved in to different grain size in a lab to have a bone char media of different grain size. Hence a bone char media of four grain sizes (0.6mm, 2mm, 5mm, 10mm) were prepared for the test. The sampled water were tested for fluoride ion concentration before test in a laboratory using a photometer. Then the bone char was mixed with water samples with known fluoride concentration waited for certain fixed duration.

Figure3. Crushed bone while being sieved

Independent Variables

In this study the following parameters were considered as an independent variable, that means their effect on the bone char removal efficiency or defluoridation capacity was tested. These are:

1. Contact time of the bone char and water to be treated

2. Initial fluoride concentration of water to be treated

3. Bone char dose to be added in sample water and

4. Grain size of bone char to contact precipitation

In this experimental analysis effect of pH, effect of contact time, effect of initial concentration of fluoride, effect of bone char doseand removal efficiency have been studied.

Experimental Setup

In statistics, a factorial experiment wasdesign consists of two or more factors, each with discrete possible values or "levels", and whose experimental units taken on all possible combinations of these levels across all such factors(Dziak, et al., 2014). In this experimental analysis $4^{!}$ has been used that means experimental conditions there were 256 design as indicated in (Dziak, et al., 2014).

In Table 3.1 below indicates all tests analyzed in the laboratory depend on the different variables. The independent variables play great role in output result experimentally for various level and factor. In this experiment bone char material of grain sizes 0.6-10 mm at a dosage of 10- 25 g were used. The contact time between bone char materials and fluoridated water was between 20–90 min and the initial fluoride concentration of water was 4.87- 20.6 mg/l.

Result And Discussion

Residual Fluoride

It is found from the experimental work that, the residual fluoride concentration reduces as the bone char dose is increasing (Fig.3 a). More over the residual fluoride showed a trend of reduction as the contact time is increasing from the maximum value of 4.45 mg/l for a contact time of 30 min to a minimum value of 3.01 mg/l for 90 min contact time (Fig. 3 b). Which was similar with the result (Kaseva, 2006).

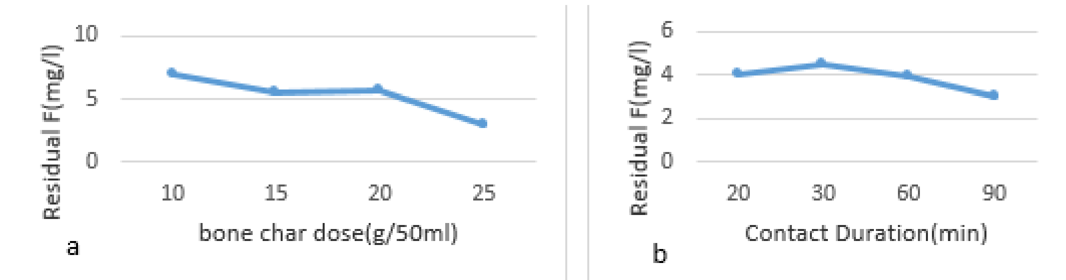

Figure4. Residual fluoride concentration

By using better combination of bone dose and time of contact(soaking duration) of 25 g of bone char and 90 min of contact time respectively. The experimental output showed that, the fluoride concentration in the treated water (20.6 mg/L) reduced to ~ 3.01 mg/L which is a significant reduction(85.4 %).Thus shows that there will be a possibility of reducing the fluoride concentration further to the WHO standard, either by increasing the contact time or the bone char dose.

Residual Fluoride for Cheffe

Based on the experimental setup which consists sample water of fluoride concentration 20.6 mg/l, pH of sample water 7.3, volume of treatment water 50 ml and contact time 20 min the residual fluoride concentration for different sample site were determined. The residual fluoride for the sample site of Cheffe for the shortest contact time in this study (20 min) were found to be ranging from maximum of 12 mg/l to 4.02 mg/l for (10 mm x 10 g) bone char size and dose, and (0.6 mm x25 g) bone char grain size and dose respectively (Table .1).

Table 1 Residual fluoride concentration for Cheffe

Bone dose(g)	Grain size(mm)			
	0.6	**2**	**5**	**10**
10	7.50	9.80	10.20	12.30
15	6.32	7.02	8.30	9.01
20	6.00	5.40	7.03	8.60
25	4.02	5.20	5.00	4.50

In general it is found that the bone char grain size used and level of residual fluoride ion in the treated water have direct relationship. For instance for a given bone dose of 10 g and contact time of 20 min, as the bone size decreases from 10 mm to 0.6 mm the residual fluoride is also reduced from 12.30 mg/l to 7.50 mg/l (Table.1). Hence, the removal efficiency of the bone char is increasing as the bone char grain size is reducing (Fig.4 a, b). This is true as we reduce the size of grain the contact area of the individual grain (the surface area per unit volume of the grain will increase) which facilitate the adsorption.

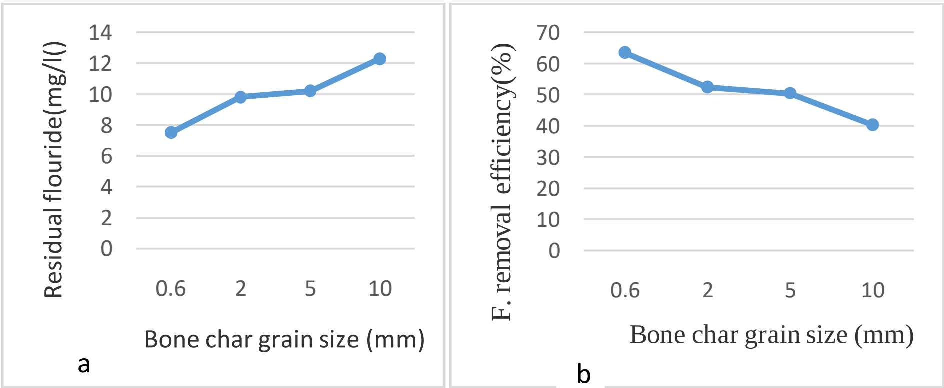

Figure5. *Bone char grain size and its removal efficiency*

Residual Fluoride for Korem Safer

Fluoride concentration of 4.87 mg/l sample water, P^H of sample water 6.5, volume of treatment water 50 ml and contact time 20 min was conducted. Effect of particle size on the adsorptive capacity of fluoride ion was investigated under different particle size and result is presented graphically. From the result adsorption capacity increases as particle size get smaller and smaller (Fig.5) which is supported by the finding of Eli Dahia, (2015), and (Kaseva, 2006). As it is shown in table.2 at particle size >10 mm removal efficiency is 1.10mg/g, but when particle size is <0.6 mm removal efficiency was increased to 4.35 mg/g which is also in line with the finding of Bregnhøj, (2007). Generally, it is obvious that as particle size decrease surface area of adsorbent increases and so is the adsorption capacity.

Table2. Residual fluoride concentration for Kerom Safer

Bone dose(g)	Grain size(mm)			
	0.6	**2**	**5**	**10**
10	4.00	4.30	4.60	4.65
15	3.85	3.90	4.20	4.40
20	3.80	3.95	4.02	4.01
25	3.50	3.59	3.76	3.87

In general it is found that the bone char grain size and fluoride ion residual amount in the treatment water had direct relationship. As the bone char size decreases the residual fluoride is also decreased or more fluoride ion is removed from the water being attached to the bone char surface. For instance for a given bone dose of 25 g and contact time of 20 min, as the bone size decreases from 10 mm to 0.6 mm the residual fluoride is also reduced from 3.87 mg/l to 3.5 mg/l (Table.2). This is true that as we reduce the size the contact area of the individual grain (the surface area per unit volume of the grain will increase) which facilitate the adsorption (Fig .5).

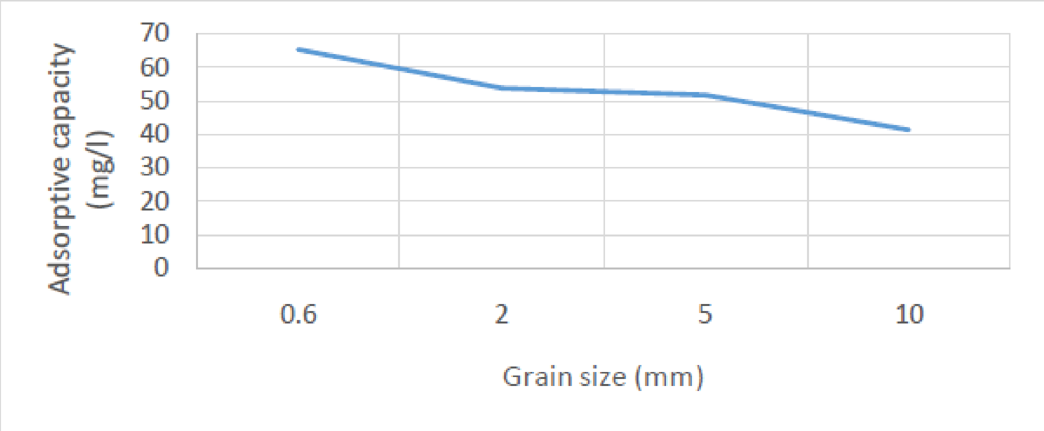

***Figure6.** Effect of grain size on adsorptive capacity Residual Fluoride for Dato*

Fluoride concentration of 10.2 mg/l sample, P^H of sample water 6.8, Volume of treatment water 50 ml and Contact time 20 min, which is based on the actual situation of the water in the particular area was used. Effect of particle size of the adsorptive capacity on fluoride adsorption was investigated under different particle size and result is presented graphically on (Fig. 5). the same relationship between bone char particle size and residual fluoride ion in the treated water was observed as in the other two test site as before. The removal efficiency of the bone char media was found 1.10mg/g for bone char grain size more than 10 mm while this value of removal efficiency dramatically increase to 4.35 mg/g for smaller grain size (less than 0.6 mm) (Table .3).

Table3. Residual fluoride concentration for Dato

Bone dose(g)	Grain size(mm)			
	0.6	2	5	10
10	6.45	7.53	7.68	8.23
15	5.76	6.52	7.32	8.01
20	4.32	4.70	5.01	5.45
25	3.56	4.65	4.01	4.35

Residual Fluoride for Tikur Wuha

Fluoride concentration of 10 mg/l, P^H of sample water 6.95, Volume of treatment water 50 ml and ccontact time 20 min the residual fluoride concentration for different sample site were determined. The residual flouride for the sample site of Tikur Wuha for the shortest contact time in this study (20min) were found to be ranging from maximum of 8.8 mg/l to 4.81mg/l for (10mm x 10g) bone char size and dose and (0.6mm x25 g) bone char grain size and dose respectively (Table .4)

Table 4Residual fluoride concentration for Tikur Wuha

Bone dose(g)	Grain size(mm)			
	0.6	2	5	10
10	7.28	8.16	8.19	8.80
15	5.00	5.35	6.05	7.15
20	4.65	5.12	5.05	5.52
25	4.81	4.95	5.01	5.30

Contact Time and Defluoridation Capacity

Analysis of residual fluoride in a fluoride affected water being treated by or soaked in bone char media for different duration of time (20, 30, 60, and 90 min) was carried out in order to see the f= effect of duration of soaking on the removal efficiency. In the experiment bone char material of four grain sizes initial fluoride in the treated water is 20.6mg/l and 25 g doze were used. It is found that the residual fluoride varied from a maximum value of 4.45 mg/l for a contact time of 30 min to a minimum value of 3.01 mg/l at 90 min duration of soaking time (Table .5)

Table5. The effect of contact time on removal of fluoride

Contact Time (min)	Residual Fluoride after treatment (mg/l)	Removal efficiency fluoride ion (%)	Adsorptive capacity (mg/g)
20	4.02	80.5	33.16
30	4.45	78.4	32.3
60	3.95	80.83	33.30
90	3.01	85.39	35.18

The least fluoride removal was obtained in a sample of bone char media that was removed for a period of 30 min. This resulted in a residual fluoride concentration of 4.45 mg/l at 30 min contact time. As it is shown in fig.6 the highest removal efficiency (85.39 %) was obtained from bone char material which was removed for a duration of 90 min. This finding is similar with (Ragheb, 2013).

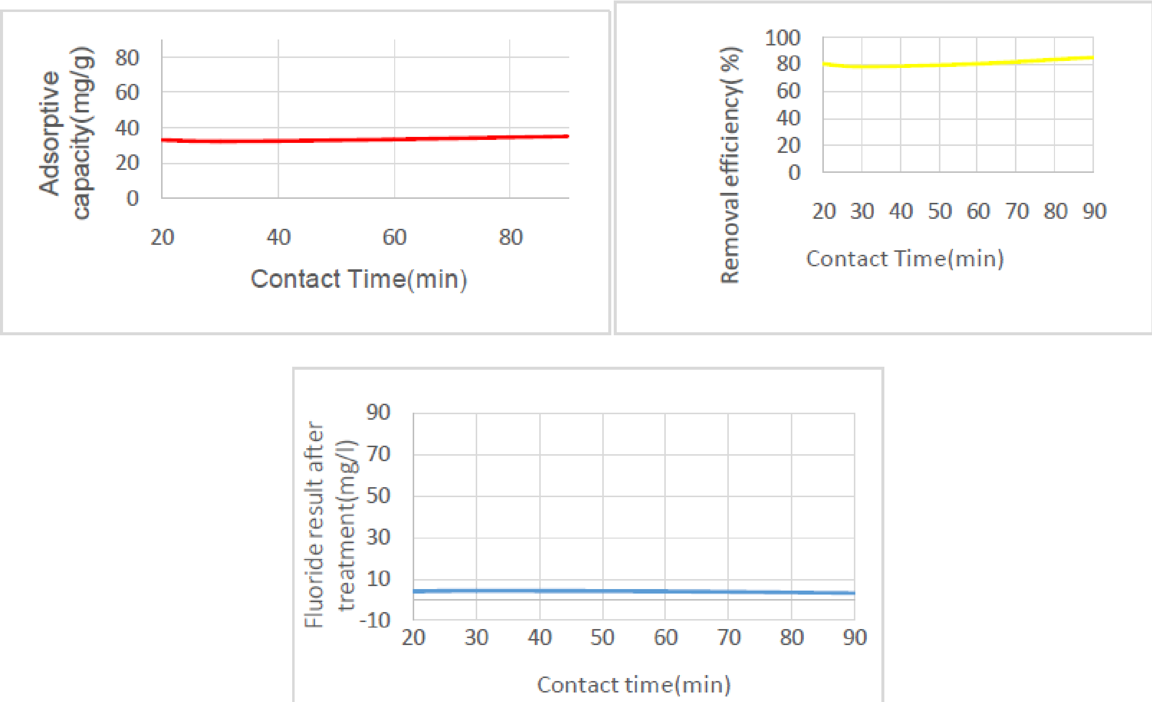

Figure7. *Effect of contact time on removal of fluoride*

The finding of this study shows that the removal amount of fluoride ion was increase over longer duration, Since there is no significant change after 60 min, it was considered as equilibrium with removal efficiency of 79.61 and maximum adsorption capacity of 0.27 mg/g. In general as contact time increases the adsorption efficiency of the adsorbent increases for the first few minutes and it gets constant or no more increase after some time because all active pore sites are occupied and fluoride ions were adsorbed, this finding is inline with (Kebede et al., 2014).

Grain Size and Defluoridation Capacity

Effect of adsorbent on fluoride ions adsorption was investigated under different adsorbent dose and the result is presented graphically on (Fig.1a). It was found that if the adsorbent dose is increased the removal efficiency will also be increased as well. The minimum and maximum removal efficiency obtained were63.59 and 80.49 % for 9 and 4 mg/lof bone char dose respectively (Table.1). This finding is in line with the finding of Albertus, (2006). It is obvious that as dose increase the active surface will also increase which are ready to adsorb fluoride ions in the solutions.

Effect of particle size of the adsorbent on fluoride adsorption was investigated under different particle size. From the result adsorption capacity increases as particle size get smaller and smaller. As it is shown in Table.6 at particle size >2 mm removal efficiency is ~77% but when particle size is 0.6 mm removal efficiency was increases to ~85%. Generally, it is found that as particle size decrease surface area of adsorbent increases.

Table6. *Adsorption capacity and removal efficiency of bone char*

Grain size	Contact time(min)	Initial fluoride concentration(mg/l)	Residual Fluoride (mg/l)	Removal efficiency (%)	Adsorptive capacity(mg/g)
0.6	90.00	20.60	3.01	85.39	35.18
2	90.00	20.60	4.83	76.55	31.54
5	90.00	20.60	4.75	76.94	31.70
10	90.00	20.60	4.79	76.75	31.62

The filtration experiment was carried out for various particle sizes (0.6, 2, 5, 10 mm) of bone material charred at 500 ^{0}C to establish the effect of grain size of the bone char material on fluoride removal capacity, it is found that bone char of 0.6 mm diameter had the least residue fluoride or hieghest removal capacity(Fig. 7). This finding is line with(Bregnhøj, 2007).

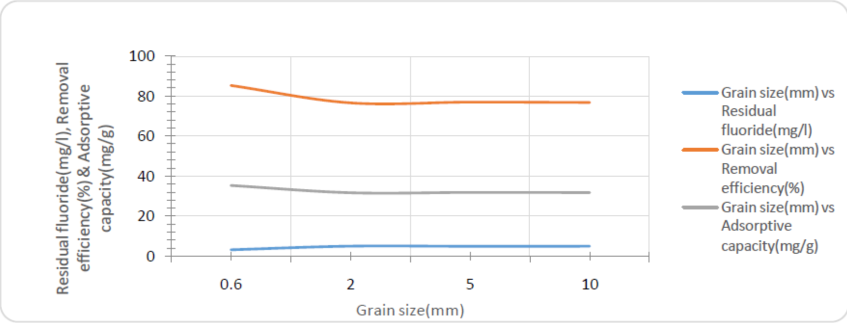

Figure8. *Fluoride result after treatment at different grain size*

Grain size is another important parameter which significantly influences adsorption efficiency of adsorbent which is similar with (Vyshak and Jayalekshmi, 2014). For this study as plotted on (Fig. 7**), t**he removal efficiency increased as the particle size decreased. This is due to the increases of surface area associated with decreased particle size. The presence of large number of smaller particles provides high sorption efficiency with the presence of larger surface area for Fluoride ions and it also reduces the external mass transfer resistance as indicated in (T.Mohammed and Rashid, 2012, Jamode et al., 2004).This can be pointed to the fact that the smaller adsorbent particles have shortened diffusion paths and increased total surface area, and therefore the ability to penetrate all internal pore structures of adsorbent is very high (Gupta et al., 2009). For larger particles, the diffusion opposition to mass transport is higher and the majority of the internal surface of the particle may not be utilized for adsorption and as a result, the amount of Fluoride ion adsorbed is small.

Dosage of Bone Char and Defluoridation Capacity

Fluoride removal capacity with respect to the dosage of bone media was studied by using different dosages (10, 15, 20 and 25 g) of bone char material with particle grain sizes of 0.6, 2, 5, 10 mm diameters for a different contact time. The initial fluoride concentration was 20.6 mg/l and the media had been charred at 500 ^{0}C for 120 min (Bregnhøj, 2007). Figure 8 indicates that fluoride removal capacity is dependent on the dosage of the bone char media and that the higher the dosage the higher the removal efficiency. In this study, the highest removal efficiency is found with 25 gm bone char media for soaking duration of 90 min at the smallest grain size of 0.6 mm. This could show providing more media of bone char will give better efficiency of removing the fluoride ion (Table 7).

Table2. Effect of bone dose on removal efficiency

Bone dose(gm)	Contact time(min)	Initial fluorideconcentration (mg/l)	Residual Fluoride (mg/l)	Removal efficiency (%)
10.00	90.00	20.60	6.95	66.26
15.00	90.00	20.60	5.45	73.54
20.00	90.00	20.60	5.64	72.62
25.00	90.00	20.60	3.01	85.39

According to the result of this study increasing of bone char dose resulted in high removal efficiency. This result was expected because as the dose increases the more active site will be available in solutions and it is similar with the finding of(Baraka et al., 2012, Guan, 2005). From (table 7), it was observed that the adsorption percentage of fluoride ions onto the bone char increased rapidly with the increasing of adsorbent concentration. This result is expected because the increase of adsorbent dose leads to greater surface area.

As a result was presented graphically on (Fig.8) fluoride removal was high at lower initial concentration and start to decrease as initial concentrations start to increases. Result of removal efficiency for 4.78 mg/L was 22.79 % but it was decrease to the contrast when fluoride concentration increase and result was 81.31 % for 20.6 mg/L in (Table .8). This can be concluded that the active site of adsorbent was limited with constant adsorbent dose but continues increases of fluoride ions remain excess in solution.

Effect of pH

The effect of pH on Fluoride ions removal from water solution analyzed with synthetic solution was investigated and presented on (Fig.8). The lowest adsorption was observed at pH ~6.5 and the highest is at pH ~ 7.3. The minimum and maximum adsorption efficiency was ~ 50.9 and 80% respectively. From the result it is observed that adsorbent can effectively remove >80% ofF ions. The trend shows that there will be a direct relationship between the removal efficiency of the bone char and the pH of the media or the treated water. Hence, one has to know the pH of water to be treated whenever to use bone chare as a defloration media. That means the removal efficiency of bone char is limited by the pH of the treated water. This finding is in line with the finding of Bregnhøj, (2007).

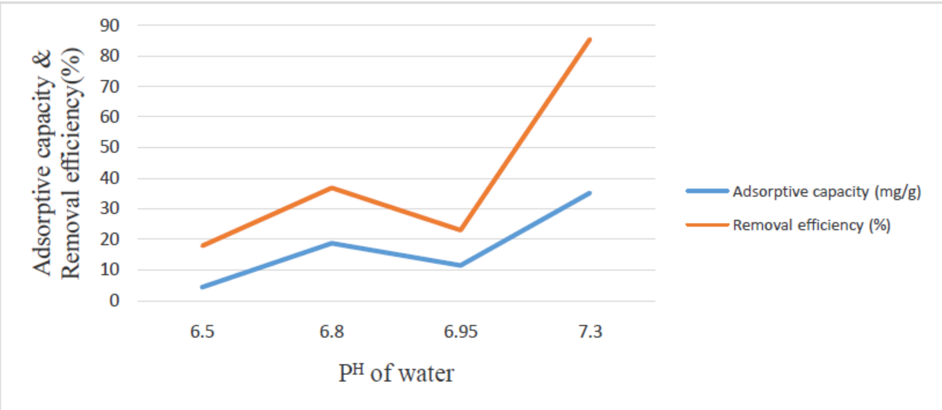

Fig9. *Effect of pH on the removal ofFfluoride ion by bone char.*

To Identify the Significant Factor of Defluoridation Based on ANOVA

The significant factor in this study investigated were bone dose, contact time and grain size. This independent variables has a significant effect on the residual fluoride concentration.

Significant Effect of Bone Dose

The residual fluoride or the defloration capacity of the bone dose is significantly affected by the bone char dose to be used. That means when the bone dose is increased the fluoride amount was decrease.

$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_5$------------------- (3)

Where,

β - is coefficient of the independent variables (constants) for each independent variable

x- is independent variable (bone char doze grain size, pH level of the treated water and the contact time)

y- is dependent variable (residual fluoride)

$Y = \beta_0 + \beta_1 BD + \beta_2 CT + \beta_3 GS + \beta_4 PH + \beta_5 F$............................ (3.1)

Y=104.463 - 4.39BD + 0.024CT- 0.51GS - 16.97PH + 3.383F....... (3.2)

From the above relationships give in equation 3.2 except the soaking duration of the bone char and initial fluoride ion concentration in the treated water other factors will have a negative relationship with the residual fluoride in the treated water. In the other way, the capacity of the bone char to remove the dissolved fluoride ion in the water is higher for higher bon dose,smaller grain size and higher pH value.

Table8. Analysis removal of fluoride result

ANOVA						
Model		Sum of Squares	Df	Mean Square	F	p>(t) Sig.
1	Regression	57760.637	5	11552.127	459.284	.0005
	Residual	6288.120	250	25.152		
	Total	64048.756	255			

Table9. The summary of variable in statistic model of SPSS

Model Summary									
Model	R	R^2	Adjusted R^2	SE Estimate	Change Statistics				
					R^2	F Change	df1	df2	Sig. F Change
1	0.950	0.902	0.90	5.01522	.902	459.284	5	250	0.0005

In general if the R^2 is defined the distances of estimated and actual value depend on its mean. That means $R^2=\frac{\sum(\hat{Y}-\bar{Y})^2}{\sum(y-\bar{Y})^2}$ it should be equal to 0.9 or 1 to be a significant value in experimental result. Hence, based on this truth from (Table 9) it was conclude that R^2=0.902 which means significant.

Conclusion

This study aimed at achieving goals of removing fluoride from water by using bone char technology. During this study the impact of different independent variables were tested with regards to its effect in reducingthe dissolved fluoride ion concentration in water. These factors were bone dose (BD), contact time (CT), grain size (GS), initial concentration of fluoride (F), and PH of water. It was found that the highest fluoride removal (85.39 %) using smallest size of bone char media, (0.6 mm), with 25 g dose soaked in water with dissolved fluoride ion for 90min, which reduces Fluoride ion concentration from 20.6 mg/l to 3.01 mg/. However. The trend shows that it will be possible to further reduce the residual fluoride concentration by increasing the bone char doze and soaking time duration. More over reducing the bone char grain size will have also a positive effect on the defloration capacity of the media, though it has a negative effect on the movement of water. Hence, it is promising that one can remove or reduce the fluoride ion in drinking water to allowable level using bone char which is locally available material. Therefore, it can be likely that bone char (BC) can be used as locally availablematerial together with highly efficient for removal of fluoride from water.

During this study compatible result with WHO standard guideline was not recorded, hence it was recommended to bring this standard, further detail investigation have to be continued by varying bone dose, contact time and grain size .Using different techniques like pyrolysis to compare the results with calcinations technique that means the heating bone with presence of atmospheric oxygen or no access to oxygen;

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