

Efficiency of various sodium solutions in regeneration of fluoride saturated bone char for de-fluoridation

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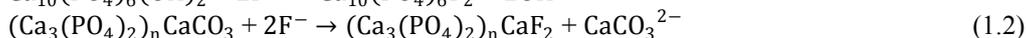
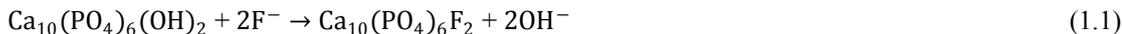
Abstract: High levels of fluoride in drinking water have adverse health effects, that include dental, skeletal and crippling fluorosis, bone cancer, reduced IQ and increased bone fractures. Reuse of fluoride exhausted bone char as a way of reducing fluoride was investigated to make full utilization of the bone char before its disposal. This study aimed at determining the effectiveness of sodium solutions (NaOH, Na₂CO₃, NaHCO₃ and Na₃PO₄) in reactivating fluoride-saturated bone char to be reused in the removal of fluoride from water with a view to finding a solution to the problem of excessive fluoride in drinking water. Determine the effect of temperature on regeneration process. Samples of 40 g each were packed in different columns, to it, 50 ml of Na solutions of different concentrations of 1, 2 and 4 % was added and samples taken at different duration of 0.5, 1, 2, 4 and 24 hours and fluoride analysed using fluoride meter. Data was analysed using M-Stat software. NaOH solution had the highest fluoride released of 130.55-186.14 ppm while the lowest was NaHCO₃ with 4.35-6.03 ppm of fluoride removed from fluoride saturated. Increase in temperature during regeneration increased the amount of fluoride released from bone char. The study confirmed that NaOH is the most effective for regenerating fluoride saturated bone char.

Keywords: sodium solutions, regeneration, fluorosis, bone char, defluoridation

I. Introduction

Bone char contains about 10% carbon (C) by weight with the remainder comprising mainly hydroxyapatite, (Ca₁₀(PO₄)₆(OH)₂) but also a significant percentage of calcium carbonate (Lewis, 1995; Guedes *et al.*, 2007).

Bone char ability to take up fluoride involves more than one reaction and varies with fluoride concentration, pH and available surface area (Bregnhøj, 1995). Reactions involved are direct adsorption of fluoride on the empty sites on the bone char surface. Ion exchange mechanisms where fluoride ion exchange position with OH⁻ (Eqn 1.1) or it exchanges with hydrogen carbonate/carbonate ion/ phosphate ion (Eqn 1.2). Recrystallisation processes where the hydroxyapatite and bone minerals dissolve and precipitate with fluoride as fluorapatite (Bailey, 1972; Bregnhøj and Dahi, 1995; Jorgen, 2005; Kawasaki *et al.*, 2009). The principal reaction is:



Upon saturation with fluoride, it is possible to regenerate bone char through; surface coating, contact precipitation, and use of sodium hydroxide. Christoffersen *et al.*, (1991) studied surface coating where fluoride saturated bone char was immersed in an acidic solution of calcium and phosphate or of bone char powder, it take up a fresh layer of hydroxyapatite, (Ca₁₀(PO₄)₆(OH)₂) on its surface. The surface coated bone char behaves as unused bone char and can to absorb a new amount of fluorides.

Contact precipitation process involves the addition of calcium and phosphate compounds to the raw water prior to its flow through the fluoride saturated bone char filter (Dahi, 1996; Jacobsen and Muller, 2007 b; Koriret *et al.*, 2009). In a mixed solution of calcium phosphate and fluoride, the precipitation of calcium fluoride and/or fluorapatite is theoretically possible, but practically impossible due to reaction inertness (Fawell *et al.*, 2006). The precipitation is easily catalysed in a contact bed that acts as a filter for the precipitate, using calcium chloride and sodium dihydrogen phosphate or “monosodium phosphate.” The following equations illustrate the removal of fluoride, equation 1.3 and 1.4 involve dissolution to get calcium and phosphate ions and equation 1.5 and 1.6 precipitations of calcium fluoride and fluorapatite.



However, if some soluble parameters are changed by the treatment process such as total phosphorus levels may exceed Kenya standard limits for 2.2 mg/L as PO_4^{3-} pH increases. Water quality taste and smell have been sometimes reported to be poor (CCEFW, 2010).

Studies on the use of a 1-8% solution of sodium hydroxide for the bone char regeneration have been reported (Horowitz *et al.*, 1972; Mcharo, 1986; Christoffersen *et al.*, 1991; Jacobsen and Muller, 2007 a), where the process is assumed anion exchange (Eqn 1.7) between the fluoride ions and the hydroxyl ions:



Residual caustic soda is rinsed with an acid. Behrman and Gustafson (1938) developed a carbonic acid (dissolved CO_2) method for neutralizing excess residual caustic that prolongs the active life of both tricalcium phosphate and hydroxyl apatite.

In this project, sodium solutions for regeneration were chosen based on the previous studies that showed carbonate, hydroxyl, hydrogen carbonate, and phosphate ions are exchanged with fluoride ion from fluoride-contaminated water during de-fluoridation. Nevertheless, little has been done to determine effectiveness of sodium solutions in regeneration of fluoride saturated bone chars. There is thus a need to explore the use of different sodium solutions to maximize use of bone char in reducing fluoride to recommended standards before its disposal.

II. Materials and methods

2.1 Materials

Grey-brown bone char with average diameter of 0.6-2 mm obtained from Catholic Diocese of Nakuru De-fluoridation Company, Kenya was used. Bones are heated in a kiln to high temperatures of 400-500°C in an oxygen-depleted atmosphere to control the quality. The required temperature and duration of heating expected depend largely on the batch size and the packing rather than the type or the nature of the bone (Dahi *et al.*, 1997).

Before crushing, bone chars are manually separated from the metal pieces according to its colour, where black ones are stored and added to the next charring batch. Grey-brownish and white bone char are crushed using crushing machine in which three sieves are attached enabling separation of three different particle sizes. Powder and fine fraction < 0.63 mm used for the production of calcium phosphate pellets for contact precipitation, 0.63-2 mm filter medium is used in community and household filters and coarse (2-4 mm) is used in community filter. Homogenizing the size of the particles is to optimize both flow rate and removal capacity. Impurities and dust from the charring and crushing process are removed by washing and then bone char was dried for safe storage (Jacobsen and Muller, 2007 a).

All the different types of sodium solutions used (NaOH, NaF, NaHCO_3 , Na_2CO_3 , and Na_3PO_4) were of analytical grade.

2.2 Preparation of solutions

1000-ppm Fluoride Solution: In a 1 litre volumetric flask, 2.21g of NaF were added and diluted to mark with distilled water. Rest of the standards was made by appropriate dilution of stock solution.

Four Percent Sodium solutions: NaOH solution was made by dissolving 40 g of NaOH pellets in 1000 ml distilled water. 1% and 2% was prepared by appropriate dilution of 4% stock solution. The same procedure was used for preparation of Na_2CO_3 , NaHCO_3 and Na_3PO_4 solutions.

Fluoride analysis

The concentration of fluoride ions in the solutions was determined using a fluoride ion selective electrode Metrohm 6.0502.150 and reference electrode (Ag/AgCl) Metrohm 6.0733.100.

Reference standards were made from appropriate dilutions of a stock solution of sodium fluoride (NaF) 100 mg/l and Total Ion Strength Adjusting Buffer (TISAB) solution. A water sample of 5 ml was transferred into a 25 ml plastic beaker by means of a measuring pipette. After rinsing the pipette by distilled water, 5 ml of TISAB was measured and transferred into the beaker containing the water sample. The electrodes were immersed into the sample and stirred slowly for 30 s, and then the specific pH Ion meter was switched on in order to read the millivolts when a steady state was reached. A TISAB buffer was added prior to measurement to attain constant pH and break up fluoride complexes.

2.3 Determining effectiveness of each solution in removing fluoride from bone char

About 3 kg of unused bone char (0.63-2 mm) was soaked with 1000-ppm fluoride solution in a plastic basin to saturate the bone for five days. To make sure that all bone char was uniformly saturated, stirring was done after every 24 hours. After saturation, it was followed by rinsing the bone char with tap water to remove all the free fluoride ions and it was finally dried. The bone char was ready for use. The process of regeneration was carried out by exposing the fluoride saturated bone char to NaOH solution in batch through the column. Samples of 40 g each were packed in several columns, to it, 50 ml of NaOH concentration of 1% NaOH was added and

effluents taken at different duration of 30 minutes, 1, 2, 4 and 24 hours. The procedure was repeated using 2% and 4% NaOH solutions to determine the effect of varying NaOH concentration. The same procedure was repeated for the other sodium solutions (NaHCO₃, Na₃PO₄ and Na₂CO₃). All the effluents collected were stored in plastic bottles before the analysed for fluoride concentration. All the experiments were conducted in triplicate. Results are expressed as mean ± standard deviation. Two-way ANOVA was carried out to determine significance difference in varying concentrations of different sodium solutions.

III. Results and Discussions

3.1 Reactivation of Fluoride Saturated Bone Char

Fluoride ion is known to exchange with hydroxyl, carbonate, hydrogen carbonate, and phosphate ions of bone char during fluoride removal from water using bone char (Bailey, 1972; Abe *et al.*, 2004; Kawasaki *et al.*, 2009). During reactivation of fluoride saturated bone char, three different concentrations of the Na solutions: 1%, 2%, and 4% were used. Figures 3.3 through 3.6 shows the results obtained for fluoride released from fluoride saturated bone char for the four solutions at different concentrations.

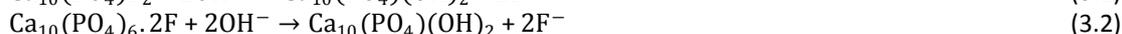
At 95% confidence interval, comparison 1% vs. 2% concentrations were not statistically different, $p > 0.05$ different while 2% vs. 4% were statistically different, $p < 0.05$.

Figure 3.1 shows that the highest fluoride concentration released was 6.03 ppm at 4% NaHCO₃, 5.83, and 5.12 ppm for 2% NaHCO₃ and 1% NaHCO₃ respectively. The minimum concentrations were 4.35, 4.65, and 4.20 ppm for 4%, 2%, and 1% respectively. ANOVA study at 95% in Figure 4.1 show that the means for the 1% vs. 2% NaHCO₃ concentrations were not significantly different that is, increasing the concentration of sodium bicarbonate from 1% to 2% had no effect on the fluoride removed. For 2% vs. 4%, there is no significant difference at 0.5 and 2 hours respectively. These low concentrations of fluorides released may be attributed to the fact that hydrogen carbonate in both fluorapatite and hydroxyapatite is not a functional group in ion exchange, or the concentration of OH⁻ ions is very low.

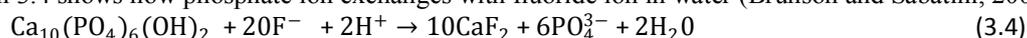
At 95% confidence interval, 1% vs. 2% concentrations were statistically different i.e. $p < 0.05$ while 2% vs. 4% concentrations were not statistically different i.e. $p > 0.05$.

From Figure 3.2, the higher the NaOH concentration, the higher the fluoride effluent released from the bone char. The optimum effluent fluoride concentration was found to be 186.1 ppm and 172.0 minimum using 4% NaOH, 2% NaOH, 181.4 maximum and 175.9 ppm minimum and 1% NaOH, 145.1 maximum and 130.6 ppm respectively. Optimum contact time for regeneration was obtained at two hours for all the concentrations used. According to the ANOVA analysis in Figure 3.2, at 95% indicated that the mean concentrations are statistically significant for 1% vs. 2% NaOH concentrations. This suggests that increasing the concentration of sodium hydroxide, increases the fluoride concentration removed. However, no significant difference was obtained for 2% vs. 4% NaOH, suggesting that increasing the concentration of sodium hydroxide from 2% to 4% does not increase the concentration of fluoride ions removed from the saturated bone char. The reaction involved is ion exchange between fluoride ions from fluorapatite and hydroxyl ion from sodium hydroxide (Bailey, 1972; Dahi, 1997; Jacobsen and Muller, 2007 a).

In regeneration, using NaOH the fluoride in the molecule of fluorapatite is displaced by OH⁻ as follows (Equations 3.1-3.3)

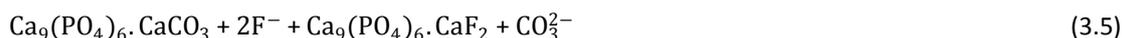


At 95% confidence interval, 1% vs. 2% and 2% and 4% concentrations were significantly different. For sodium phosphate, maximum fluoride removed was 74.43 ppm and minimum was 28.64 ppm in Figure 3.3. There is significant difference for 1% vs. 2% and 2% vs. 4% thus increasing the concentration from 1% to 2% or from 2% to 4%, the concentration of the fluoride removed from bone char increases. The concentrations of phosphate ions eluted during defluoridation were compared to fluoride ions removed by bone char, and phosphate ions were found to be very low depending on the animal bone char used (Kawasaki *et al.*, 2009). Equation 3.4 shows how phosphate ion exchanges with fluoride ion in water (Brunson and Sabatini, 2009).



At 95% confidence interval, 1% vs. 2% and 2% vs. 4% were found to be significantly different.

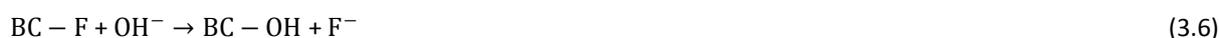
At 95% confidence interval, 1% vs. 2% and 2% vs. 4% were found to be significantly different. The removal mechanism was via ion exchange in which the carbonate ion of apatite was replaced by fluoride ion to form fluorapatite as (Ayoob *et al.*, 2008; Shrikant and Nitin, 2012). The carbonate ion is thought to be the active part of the apatite (equation 3.5).



Regeneration using sodium carbonate solution is the reverse of the equation 3.5. From Figure 3.4, maximum fluoride removed was 47.46 ppm while minimum was 31.58 ppm. At 95%, the means for 1% vs. 2% concentrations were statistically significant. This suggests that increasing concentration of sodium carbonate from 1% to 2%, increases the amount of fluoride concentration removed from bone char. Increasing concentration of sodium carbonate had no significant effect for 2% vs. 4% between 0.5 and 1 hour.

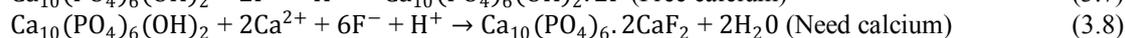
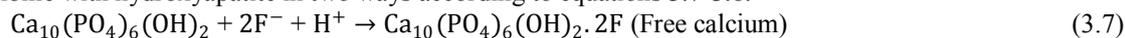
The pH values of the solutions were measured to determine whether pH has any effect on the amount of fluoride released from the bone char. The pH values of the four solutions were observed to follow the following order $\text{NaHCO}_3 < \text{Na}_2\text{CO}_3 \approx \text{Na}_3\text{PO}_4 < \text{NaOH}$. The pH of the solution was found to be the most important factor affecting the amount of fluoride removed. Hydrogen carbonate provided the lowest concentration of fluoride (4.12-6.03 ppm) and it has the lowest pH and sodium hydroxide the highest (130.55-182.18 ppm). Phosphate had a concentration of between 22.60-74.43 ppm and carbonates a concentration of between 31.58-47.46 ppm of fluoride removed. The results obtained indicate that the best desorption was achieved in the solution with highest pH.

One of the reasons for better desorption at high pH values may be attributed to a large number of OH^- ions present at these pH values, which in turn increases diffusion and mobility of fluoride ions. At low pH values, the reduction in desorption may be possible due to the abundance of H^+ ions thus fluoride ions are immobile and this hinders diffusion. The surface desorbed anions favorably in high pH range due to the presence of OH^- ions, whereas the surface is active for the adsorption of anions at low pH values due to the accumulation of H^+ ions (Tembhurkar and Shilpa, 2006). Thus, greater pH gives maximum fluoride removal. The major regeneration process therefore, involves ion exchange between hydroxyl ions in solution and fluoride ions from the fluorapatite according to equation 3.6.

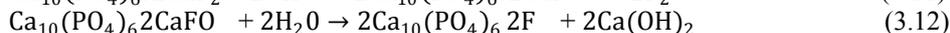
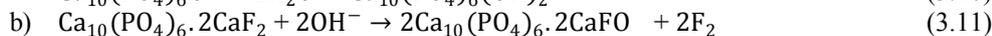
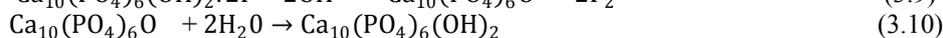
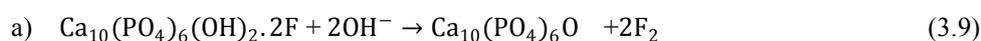


This reaction readily occurs because fluoride ion and hydroxide ion have the same charge and radius (Bregnhøj, 1995; Chidambaram *et al.*, 2003). Regeneration process also involves other reactions such as diffusion, precipitation and desorption.

Regeneration can be carried out by heating fluoride saturated bone char in the presence of hydroxide (Kaseva, 2006). Wang *et al.*, 2001 suggested that during fluoride removal from water, fluoride ion might combine with hydroxyapatite in two ways according to equations 3.7-3.8.



In regeneration using sodium hydroxide, the F^- in the $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2 \cdot 2\text{F}$ can be replaced by OH^- (Equations 3.9-3.10). The molecule $\text{Ca}_{10}(\text{PO}_4)_6 \cdot 2\text{CaF}_2$ cannot react with OH^- but dissolves and releases fluoride from the molecule heating as shown in equations 3.11-12. The reaction may be as follows:



3.2 Effect of Temperature in Regeneration

Study on the effect of temperature was conducted by varying it from 20-60 °C keeping bone char of 40 g/50 mL and 1-hour contact time.

The effect of temperature on the regeneration of fluoride saturated bone char was studied at temperatures of 20, 30, 40, 50 and 60°C. It was found that fluoride released was essentially dependent on temperature as seen in Figure 3.5. At 50 °C, it was found to be the optimum temperature for regeneration since it provided the highest concentration of fluoride released 159.87 ppm. At higher temperature, the hydroxyl ions move faster and more can penetrate into cavities of the porous bone char's structure. Hence, this result in more exchange of hydroxyl ions with the fluoride ions of the bone char is fluorapatite. The amount of fluoride ions that were desorbed increased at higher temperatures. This result indicated that desorption mechanism of fluoride ion from bone char is an endothermic reaction; that is, the fluoride in bone char consumes heat in exchanging with a hydroxyl ion. Diffusion of hydroxyl ions seems also to increase with increased temperature (Meena *et al.*, 2005).

IV. Conclusions

Sodium hydroxide was found to be the most effective solution for regeneration of fluoride saturated bone char. Increasing the pH, increases the concentration of fluoride released from bone char; 130.55-182.18 for NaOH, 31.58-47.56 for Na₂CO₃, 22.60-74.43 for Na₃PO₄ and 4.12-6.03 for NaHCO₃ ppm fluoride.

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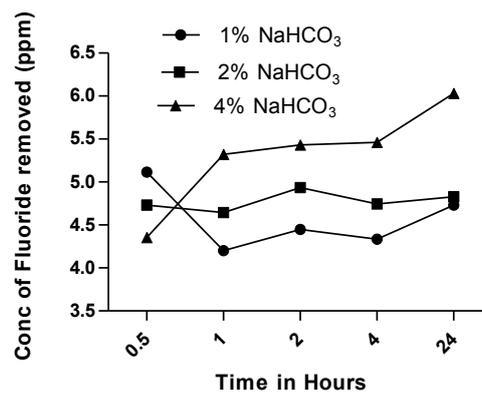


Figure 1: Concentration of Fluoride removed at various times by 1%, 2% and 4% NaHCO₃

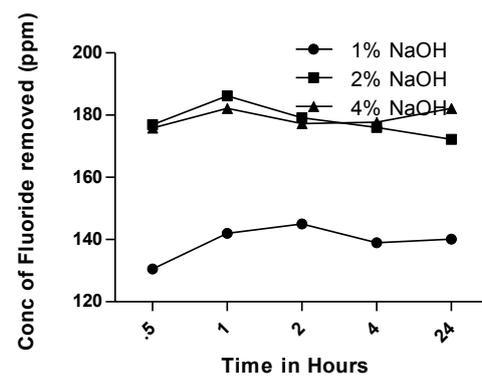


Figure 2: Concentration of Fluoride removed at various times by 1%, 2% and 4% NaOH

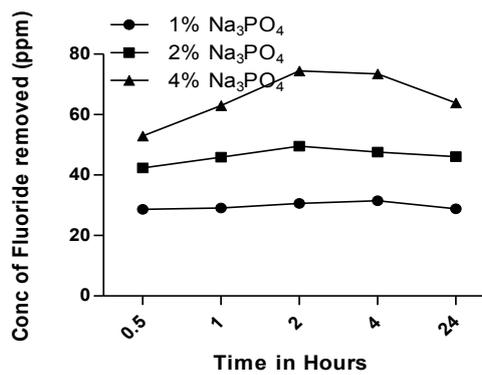


Figure 3: Concentration of Fluoride removed at different times by 1%, 2% and 4% Na₃PO₄

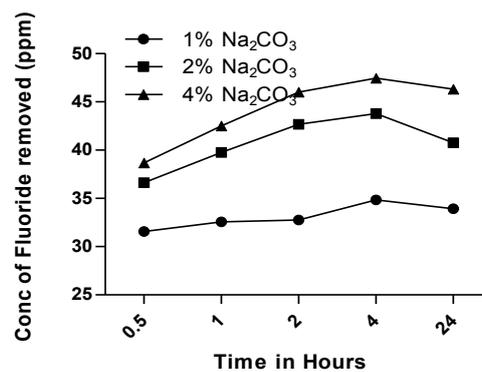


Figure 4: Concentration of Fluoride removed at various times by 1%, 2% and 4% Na₂CO₃

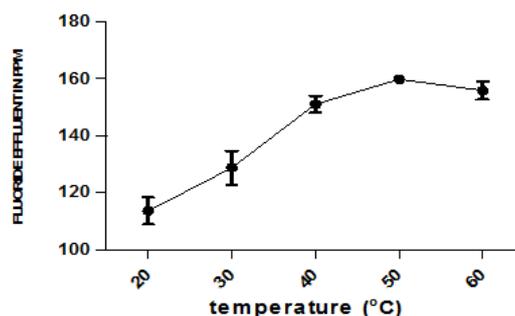


Figure 5: Concentration of fluoride removed at different temperatures of regeneration.

Table 1: Fluoride concentration released from bone char using 1%, 2% and 4% of NaHCO₃ solution for different contact times

Time in Hours	Fluoride concentration in ppm		
	1% NaHCO ₃	2% NaHCO ₃	4% NaHCO ₃
0.5	5.12 ± 0.41	4.73 ± 0.38	4.35 ± 0.09
1	4.20 ± 0.11	4.65 ± 0.21	5.32 ± 0.25
2	4.45 ± 0.07	4.94 ± 0.43	5.43 ± 0.08
4	4.34 ± 0.20	4.75 ± 0.18	5.46 ± 0.20
24	4.73 ± 0.42	4.83 ± 0.06	6.03 ± 0.10

Table 2: Fluoride Concentration released from Bone Char using 1%, 2% and 4 % of NaOH solution for different contact times

Time in Hours	Fluoride concentration in ppm		
	1% NaOH	2% NaOH	4% NaOH
0.5	130.55 ± 3.04	176.96 ± 9.79	175.89 ± 3.08
1	142.02 ± 4.24	186.14 ± 12.55	182.18 ± 5.52
2	145.06 ± 1.99	179.14 ± 3.20	177.33 ± 4.38
4	138.96 ± 5.90	176.08 ± 0.70	177.78 ± 5.91
24	140.17 ± 1.79	172.18 ± 1.72	182.16 ± 4.75

Table 3: Fluoride Concentration released from Bone Char using 1%, 2% and 4 % of Na₃PO₄ solution for various contact times

Time in Hours	Fluoride concentration in ppm		
	1% Na ₃ PO ₄	2% Na ₃ PO ₄	4% Na ₃ PO ₄
0.5	28.64 ± 1.16	42.33 ± 1.27	52.97 ± 6.97
1	29.10 ± 0.51	45.87 ± 1.63	62.98 ± 0.54
2	30.64 ± 0.96	49.60 ± 0.62	74.43 ± 1.90
4	31.51 ± 0.40	47.60 ± 0.46	73.50 ± 0.53
24	28.82 ± 0.53	46.06 ± 0.84	63.85 ± 0.55

Table 4: Fluoride Concentration released from Bone Char using 1%, 2% and 4 % of Na₂CO₃ solution for different contact times

Time in Hours	Concentration in ppm		
	1% Na ₂ CO ₃	2% Na ₂ CO ₃	4% Na ₂ CO ₃
0.5	31.58 ± 1.31	36.63 ± 0.67	38.69 ± 1.73
1	32.56 ± 0.59	39.75 ± 1.89	42.51 ± 0.86
2	32.77 ± 1.96	42.68 ± 0.96	46.02 ± 0.22
4	34.85 ± 1.08	43.79 ± 0.21	47.46 ± 0.39
24	33.92 ± 0.91	40.77 ± 1.45	46.31 ± 1.67

Table 5: Effect of Temperature on Regeneration

Effect of temperature on regeneration using 1% NaOH	
Temperature (°C)	Fluoride effluent (ppm)
60	155.92 ± 5.43
50	159.87 ± 2.62
40	151.14 ± 5.04
30	128.80 ± 8.44
20	113.67 ± 8.25