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Effect of Magnesium on Fluoride Removal

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Abstract: Fluorides in drinking water are known for both beneficial and detrimental effects on health. The fact that the problems associated with the excess fluorides in drinking water is highly endemic and widespread in countries like India prompted many researchers to explore quite a good number of both organic and inorganic materials adopting various processes from coagulation, precipitation through adsorption, Ion exchange etc. for fluoride removal. Some are good under certain conditions while others are good in other conditions. Leaching of Fluoride from the earth crust is the chief source of fluoride content in ground water; however the other sources like food items also add to increase the overall ingestion of fluoride into the human body. The soil at foot of the mountains is particularly likely to be high in fluoride from the weather and leaching of bed rock with a fluoride. The present paper aims to encompass the work carried out by various researchers in various fluoride affected areas and to access the effectiveness of using magnesium for fluoride removal.\
Key Words: Fluorides, coagulation, flocculation, Nalgonda process, fluorosis

I. Introduction

Fluorine is the chemical element with atomic number 9, represented by the symbol F. Under normal conditions, elemental fluorine is a yellow-green gas of diatomic molecules, F_2. The lightest halogen, it is found on Earth in its only stable isotope, fluorine-19. The high affinity of fluorine for electrons leads it to direct reactions with all other elements in which the reaction has been attempted, except for helium and neon. Chemically, fluorine is one of the strongest oxidizing agents known, and is similar to, but even more reactive than elemental chlorine. Ionic compounds of fluorine are water-soluble halide salts known as fluorides. Fluorine (F_2) is a greenish diatomic gas. Fluorine is so highly reactive that it is never encountered in its elemental gaseous state except in some industrial processes. The fluoride occurs notably as Sellaite, fluorspar, CaF_2; Cryolite, Na_3AlF_6; Fluorapatite, $3Ca_3 (PO_4)_2 Ca(F,Cl_2)$. Other minerals containing fluoride are given in the table 1.

Table1. Fluoride bearing minerals

MINERAL	CHEMICAL FORMULA	PERCENTAGE FLOURINE
Sellaite	MgF_2	61%
Villianmite	NaF	55%
Fluorite (Fluorspar)	CaF_2	49%
Cryolite	Na_3AlF_6	45%
Bastnaesite	$(Ce,La) (CO_3)F$	9%
Fluorapatite	$Ca_3(PO_4)_3F$	3-4%

As fluorspar it is found in sedimentary rocks and as Cryolite in igneous rocks. These fluoride minerals are nearly insoluble in water. Hence fluorides will be present in ground water only when conditions favor their solution. It is also present in sea water (0.8-1.4 ppm), in mica and in many drinking water supplies. The current information on fluoride in the environment and its effects on human health and available methods of defluoridation are presented in the following sections. It is evident from the information available that a certain quantity of fluorine is essential for the formation of caries-resistant dental enamel and for the normal process of mineralization in hard tissues. The element is metabolized from both electrovalent and covalent compounds. Low fluoride concentrations stabilize the skeletal systems by increasing the size of the apatite crystals and reducing their solubility. About 95% of the fluoride in the body is deposited in hard tissues and it continues to be deposited in calcified structures even after other bone constituents (Ca, P, Mg, CO_3 and citrate) have reached a steady state. Age is an important factor in deciding to what extent fluorine is incorporated into the skeleton. The uptake almost ceases in dental enamel after the age of about 30 years. Many rivers flowing through more than half a dozen states in India reported to have fluoride contents varying from 0.1 to 12.0 ppm Similarly occurrence of fluoride bearing waters was reported by many in Andhra Pradesh, Rajasthan, Punjab, Haryana, Maharashtra, Tamil Nadu, Karnataka, Madhya Pradesh, Gujarat and Uttar Pradesh. Waste water from phosphate fertilizer plants may contain upto 2% of fluoride. Increased levels of fluoride can also be present in effluent from the fluorine industry, coal power plants, rubber, fertilizer and semiconductor manufacturing industry, glass and ceramic industry and in ground water around aluminum smelters.

According to 1984 guidelines published suggested that in areas with a warm climate, the optimal fluoride concentration in drinking water should remain below 1 mg/L (1ppm), while in cooler climate it could be upto 1.2 mg/L (1.2 ppm). The differentiation drives from the fact that we perspire more in hot weather and consequently drink more water. The guideline value (permissible upper limit) for fluoride in drinking water was set at 1.5 mg/L, considered a threshold where the benefit of resistance of tooth decay did not yet shade into a significant risk of dental fluorosis. It has long been know that excessive fluoride intake carries serious toxic effects. Fluorosis, an endemic disease caused by long term ingestion of high fluoride drinking water, affects many millions of people, particularly children in South Africa, Mongolia, Pakistan, Thailand, India and China. Severe forms of disease typically develop only when the fluoride concentration of the water is greater than 5 to 10 mg/L. However, symptoms of the disease can develop with regular ingestion of the water containing fluoride concentration as low as 1 to 2 mg/L. We purposely fluoridate a range of everyday products, notably toothpaste and drinking water; because for a decade we have believed that fluoride in small dose s have no adverse effects on health to offset its proven benefits in preventing dental decay. It should be noted that fluoride is also found in some foodstuff and in the air (mostly from the production of phosphate fertilizers or burning of fluoride containing fuels), so the amount of fluoride we actually ingest may be higher than assumed. So more and more scientists are now seriously questioning the benefits of fluoride, even in small quantities.

II. Literature Review

Numerous techniques have been developed for defluoridation of water. Numbers of scientists have performed experiments with various concentrations of fluoride and reducing that to a required permissible concentration considering the cost and resource availability factor. The research work related to fluoride concentration, its effect on human health and various techniques related to fluoride removal invented by various researchers around the world are discussed here. Excess fluorides in ground water cause serious environmental problems and health. The main source of fluoride accumulation is Geological formation.

"**Fluorosis -** a disease caused by ingestion of fluoride in excess through water, food, and air and is a serious health problem. Fluoride ingested with water goes on accumulating in bones up to age of 55 years. At high doses fluoride can interfere with carbohydrates, lipid protein, vitamin, enzyme and mineral metabolism."[1] "Long term consumption of water containing 1 mg of fluoride per liter leads to dental fluorosis. White and yellow glistening patches on the teeth are seen which may eventually turn brown."[1] "**Skeletal fluorosis-**This has been observed in persons when water contains more than 3-6 mg/L of fluoride. Skeletal fluorosis affects young and old alike. Fluoride can also damage the foetus- if the mother consumes water and food, with a high concentration of fluoride during pregnancy/breast feeding, infant mortality due to calcification of blood vessels can also occur, Severe pain in the backbone, joints, Stiffness of the backbone, Immobile /Stiff joints"[1] "**Non-Skeletal Manifestations-**This aspect of fluorosis is often over looked because of the misconception prevailing that fluoride will only affect bone and teeth. Fluoride, when consumed in excess can cause several ailments besides skeletal and dental fluorosis viz. Neurological Manifestations, Muscular Manifestations, Allergic Manifestations, Gastro intestinal problems, Head-ache."[1]

According to WHO standards, the fluoride in drinking water should be within a range that slightly varies above and below 1 mg/L (Meenakshi *et al.*, 2004). In temperate regions, where water intake is low, fluoride level up to 1.5 mg/L is acceptable. The Ministry of Health, Government of India, has prescribed 1.0 and 2.0 mg/L as permissive and excessive limits for fluoride in drinking water, respectively. Table 3 shows different health impacts at varying fluoride concentrations in drinking water.

"The fluoride concentration recorded in Allahabad was 0.80 to 6.50 mg/L while it was 1.60 to 1.80 mg/L in Ballia. Fluoride value of 1.60 mg/L observed in Barielly district with 2.00 mg/L and 1.70 mg/L to 4.30 mg/L in Gonda and Pratapgarh district respectively." "Abundance of brick kilns was said to be polluting the ground water in villages of Pratapgarh." [Sandeep K. Malhotra *et al.* (1998)]." "Increase in fluoride during summer due to greater dissolution and depletion of water levels was observed agreeing with the study of Singh (1994)." [Singh (1994)].

"Fluoride content in Unnao district varied from 1.46 to 3.20 mg/L. Top aquifer upto 100 meter was said to yield with fluoride greater than 1.50 mg/L and medium salinity."[Das (1996)]. "Fluoride from 0.12 mg/L to 19.0 mg/L in a few villages of Unnao district. Fluoride observed varied from 8.0 mg/L to 10.0 mg/L in Dih block. Fluoride content variation from 1.60 mg/L to 3.0 mg/L was noticed in Maharajganj and Dalmau blocks." [Mukherjee and Pandey, 1999]. "Fluctuation in fluoride content of ground water over pre-monsoon and post-monsoon was noticed in Aligarh, Etah and Sultanpur districts. Pre-monsoon levels were objectionable and fluoride was below 1.0 mg/L during post-monsoon." [Dhaneswar Rai (1999)]. "A study conducted in 77 villages of Agra district concluded fluoride concentration ranged from 0.28 to 22.0 mg/L .45 % of samples were in the range of 0.0 to 1.00 mg/L, 42% of sample showed a range of 1.0 to 1.50 mg/L and 12% of samples had fluoride

1.50 to 3.00 mg/L. 3 samples were containing above 3.00 mg/L. The highest concentration (22.0 mg/L) was recorded at Bainkhera Village." [Gupta *et al.* (1994a)].

"Excess of fluoride is present in potable water in several parts of India, resulting in the development of deformed limbs and mottled teeth in men and cattle. The present communication illustrates a simple cheap and household fluoride reduction described as Alum Treatment Method. The known amount of potash alum [$K_2 SO_4 .Al_2 (SO_4)_3. 24 H_2O$] were allowed to the sample of water and stirred properly and allowed to settle down. The spectrophotometer readings were constant after five hours and residual fluoride was within acceptable limits. "Defluoridation unit at domestic level for 3 mg/L fluoride water using Activated Alumina are present. The defluoridation capacity of activated alumina was determined by column experiments, fixing the height of column, input water flow. The concentration of fluoride in output water was monitored periodically." [G. Karthikeyan *et al.* (1994)][4] ."The removal of fluoride Ions by AI(III) ion and Ca(II) ion and solids compounds of multivalent metal elements such as AI(III), La(III), Ce(III), Nd(III), Sm(III), Ti(IV), Zr(IV) and Ce(IV) in the form of ozidesi hydrous oxides and basic carbonate is studied. With an excess amount (20 ppm) of Ca or Al ions, fluoride was removed from 5.0 to <0.02 ppm by precipitation (pH 4) and coprecipitation pH(5.5-7.5) respectively."[S. Tokunaga *et al.*][5.] "To find out cost effective alternatives for removing too much fluoride from waters, many different geomaterials have been tested in recent years, including zeolite, heat treated soils, fly ash, bauxite, volcanic ash and lime stone. Limestone was used in two column continuous flow system to reduce fluoride concentration from waste waters to below MCL (Maximum concentration Level) of 4 mg/L. calcite was forced to dissolve and fluoride to precipitate in the first column. The degassing condition in the second column caused the precipitation of calcite dissolved in the first column thus returning the treated water to its approx. initial composition. The major advantage of this technology over existing liming and ion exchange methods to treat waste waters in that system monitoring is minimal, regular column regeneration is not required and the water is returned to its initial chemical composition.[Zanxin Wang][6]

Bone char is an effective and safe material for filtration of fluoride from drinking water. Bone charring in ceramic kiln like those used in the northern part of Thailand is normal calcinations and the efficiency of bone char in fluoride reduction, compared with original bone char that was burnt in electric furnace. The laboratory studies carried out showed that the optimal temperatures for bone char efficiency in fluoride removing while maintaining the quality of drinking water after filtration was 500^0C and then stopping and letting it cool down. The efficiency of bone char calcinated at this optimal temperature is 91.8%.[Winolsri Puangpinyu *et al.*][7]. Retention of fluoride ion in dynamic experiments on column packed with fly ash was studied at 20^0 C with a series of aqueous solutions containing 1, 5,10,20,50 and 100 mg F^- /L. the flow rate through the bed was 2ml/hr. At lowest F^- concentration (1 mg/L) the F^- level in the effluent initially increased and then gradually decreased down to 0 mg/L after 120 hours. With higher F^- concentration in the feed solutions, the F^- concentration in the effluent steadily decreased reaching 0 mg/L after 120-168 hours. We conclude that coal fly ash is an effective sorbent for F^- ions, especially at high concentrations in water.[R. Piekos *et al.*][8]

Bauxite can be used as a defluoriding agent in the domestic water filters in the place of activated alumina, which is comparatively quite expensive. The researchers have concluded that the use of bauxite as fluoride removing medium is encouraging not merely because it can bring down the cost of defluoridation, but also on the account of the fact that bauxite is a natural resource, is available in almost all regions of the country. Another point of great importance is that, there is no loss of this natural resource and the rejected bauxite from differentiation units can be more effectively used for aluminum metallurgy.[Sravani Sarkar *et al.*][9]. The basic principle of the process is the adsorption of fluoride with freshly freshly precipitated aluminum hydroxide which is generated by anodic dissolution of aluminum or its alloys, is an electrochemical cell. The process utilizes 0.3 to 0.6 Kwh of electricity per 1000 litre of water containing 5 to 10 mg/L of fluoride. The anode is continuously consumed and need to be replenished. The process generates sludge at the rate of 80-100 gm per 1000 litres.[CECRI][10]. The potential of reverse osmosis membrane for defluoridation of underground water samples at different solute concentration is studied. Optimum membrane properties and percentage rejection has been determined for the RO system to minimize the overall cost of water treatment. The result indicate, reverse osmosis membrane can be successfully used for the removal of fluorine from underground water at low pressure to desired level.[Meenakshi *et al.*][11]. Application of Donnan Dialysis (DD) and Electro dialysis (ED) for removing fluoride ion from waters where the concentration exceeds acceptable values is done. The techniques both use ion exchange membrane but involve different driving forces: the difference in the electrochemical potential on both sides of the membrane for DD and the difference in the electric potential in ED.[Michour *et al.*][12]. A typical groundwater containing 10 mg/L fluorides, 60 mg/I hardness, 500 mg/L alkalinity and 7.6 pH was studied using magnesia (MgO) concentrations of 10 - 1,500 mg/L. The treated water showed a pH above 9. The average fluoride concentration in the filtrate was 5.8 mg F/L where the dose was 1,000 mg/L. The fluoride at 100, 250 and 500 mg/L doses were 9.5, 8.9 and 8.4 mg F/L, respectively. A dose of 1,500 mg/L magnesia and a contact period of 3 hr was required to reduce the fluoride content in the water to 1 mg/L. The study established that magnesia removed the excess fluorides, but large doses were necessary. Moreover the pH of the treated water was beyond 10 and its correction by acidification or recarbonation was necessary. All this adds to the cost and complexity of operations. The acid requirement can be to the extent of 300 mg/L expressed in terms of CaCO3/L.[Rao *et al.*(2003)][1]

III. Most common methods of fluoride removal

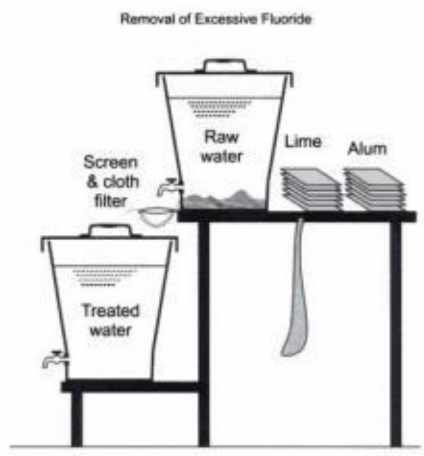
Following are the most common methods of fluoride removal, which are used as per the requirement of defluoridation and availability of resources:

- Nalgonda Technique
- Activated Alumina Process
- Bone char
- Contact Precipitation
- Degreased and alkali treated bones
- Synthetic tri-calcium phosphate
- Florex
- Activated Carbon
- Lime Process
- Ion Exchange Resins
- Cation Exchange Resins
- Magnesia
- Serpentine
- Lime stone, special soils and clay etc
- Fly Ash
- Electro coagulation Electrochemical methods
- Rare earth based materials
- Tamarind Gel
- Plant materials

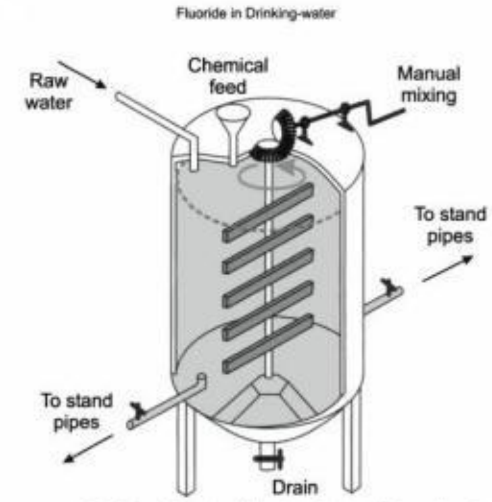
IV. Nalgonda technique

The Nalogonda technique (named after the village in India where the method was pioneered) employs flocculation principle. Nalgonda technique is a combination of several unit operations and the process involves rapid mixing, chemical interaction, flocculation, sedimentation, filtration, disinfection and sludge concentration to recover waters and aluminum salts. Alum (hydrated aluminum salts) - a coagulant commonly used for water treatment is used to flocculate fluoride ions in the water. Since the process is best carried out under alkaline conditions, lime is added. For the disinfection purpose bleaching powder is added. After thorough stirring, the chemical elements coagulate into flocs and settle down in the bottom. The reaction occurs through the following equations:

$$2Al_2(SO_4)_3.18H_2O + NaF + 9Na_2CO_3 \rightarrow [5Al(OH)_3.Al(OH)_2F] + 9Na_2SO_4 + NaHCO_3 + 8CO_2 + 45\ H_2O$$
$$3Al_2(SO_4)_3.\ 18H_2O + NaF + 17NaHCO_3 \rightarrow [5Al(OH)_3.Al(OH)_2F] + 9Na_2SO_4 + 17CO_2 + 18\ H_2O$$



The Nalgonda defluoridation as adopted for domestic use in the United Republic of Tanzania. After Dahi *et al.*, 1996.



The fill and draw type Nalgonda technique for domestic and community defluoridation. After Bulusu *et al.*, 1993.

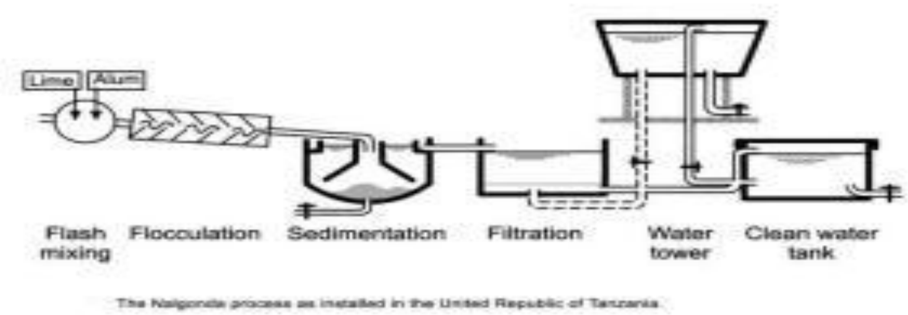


Figure 1.

Salient features of Nalgonda technique:

- No regeneration of media.
- No handling of caustic acids and alkalis.
- Readily available chemicals used in conventional municipal water treatment are only required.
- Adaptable to domestic use.
- Flexible up to several thousands of m^3/d.
- Applicable in batch as well as in continuous operation to suit needs simplicity of design, construction, operation and maintenance.
- Local skills could be readily employed.
- Highly efficient removal of fluorides from 1.5 to 20 mg/L to desirable levels.
- Simultaneous removal of color, odor, turbidity, bacteria and organic contaminants.
- Normally associated alkalinity ensures fluoride removal efficiency.
- Sludge generated is convertible to alum for use elsewhere.
- Little wastage of water and least disposal problem.
- Needs minimum of mechanical and electrical equipment.
- No energy except muscle power for domestic equipment.
- Economical - annual cost of defluoridation (1991 basis) of water at 40 lpcd works out to Rs.20/- for domestic treatment and Rs.85/- for community treatment using fill and draw system based on 5000 population for water with 5 mg/L and 400 mg/L alkalinity which requires 600 mg/L alum dose.
- Provides defluoridated water of uniform acceptable quality.

V. Observations

Table 2

LIME					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	192	80	650	8.53	9.98
10	114	55	500	9.53	8.16
12	98	60	530	9.71	7.38
14	90	67	560	9.94	5.76
16	85	70	580	10.12	5.46
18	80	75	600	10.34	5.22

Table 3

LIME					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	192	80	560	9.53	10.02
16	90	21	460	10.53	5.69

18	80	18	480	10.64	5.1
20	65	15	420	10.82	4.44
22	50	17	490	10.86	2.12
24	150	67	720	10.43	8.4

Table 4

LIME WITH 50 Mg^{2+}					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	263	69	750	8.66	9.97
14	192	30	550	9.84	6.06
16	178	19	520	10.03	5.12
18	159	15	500	10.21	4.25
20	135	9	450	10.36	3.52
22	124	7	410	10.41	2.15

Table 5

LIME WITH 50 Mg^{2+}					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	260	70	700	8.6	10.08
18	138	14	450	10.4	5.82
20	109	10	400	10.49	5.4
22	90	9	380	10.61	4.8
24	67	8	360	10.89	4.02
26	70	10	420	10.2	2.88

Table 6

LIME WITH 75 Mg^{2+}					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	288	74	700	8.58	10.12
18	166	18	500	10.1	8.9
20	137	15	450	10.26	8.25
22	118	13	430	10.46	5.95
24	95	11	400	10.59	5.9
26	70	9	350	10.73	5.65

Table 7

LIME WITH 100 Mg2+					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	305	76	700	8.78	9.96
18	105	10	510	9.73	7.74
20	99	8	460	9.98	6.9
22	92	7	420	9.98	6.3

24	73	6	400	10.1	4.56
26	49	5	380	10.15	2.16

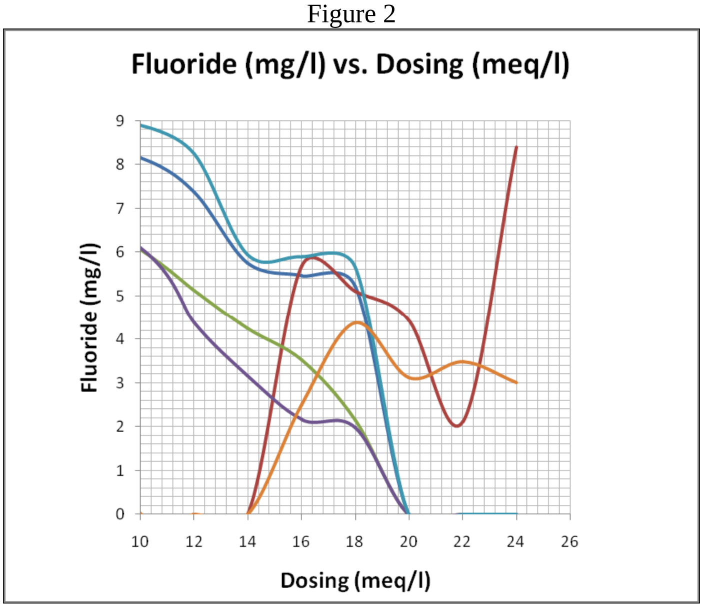
Table 8

ALUM					
DOSING	HARDNESS		ALKALINITY	pH	FLUORIDE
	TOTAL	Ca2+			
-	158	44	780	7.78	10.26
70	210	76	420	9.95	5.1
80	220	85	360	10.12	4.74
90	238	90	330	10.2	1.8
100	236	103	320	10.23	4.14
110	236	104	220	10.56	4.86

Table 9

ALUM+LIME						
DOSING		HARDNESS		ALKALINITY	pH	FLUORIDE
LIME	ALUM	TOTAL	Ca2+			
-	-	182	72	800	7.75	10.2
18	70	234	100	450	7.44	2.52
20	80	240	81	420	7.3	4.38
22	90	244	100	400	7.24	3.12
24	100	269	136	390	7.25	3.48
26	110	300	155	420	7.26	3

Figure 2



VI. Results and Conclusions

- SET-1 Lime dose varied from 10 to18 meq/l.
- Total hardness and Ca^{++} hardness was reduced continuously.
- Alkalinity was almost unaltered at the end of the test while the pH is rising from 8.53 to 10.34.
- Fluoride is reduced from 9.98 mg/l to 5.22 mg/l.
- SET-2 lime dose varied from 16 to 24 meq/l.

- Total hardness was reduced to 50 mg/l as $CaCO_3$ at the dosing of 22 meq/l and suddenly increased to 150 mg/l as $CaCO_3$ at dosing of 24 meq/l, which was 192 mg/l as $CaCO_3$ initially.
- Similarly the Ca^{++} hardness was reduced to 17 mg/l as $CaCO_3$ at the dosing of 22 meq/l and increased to 67 mg/l as $CaCO_3$ at dosing of 24 meq/l, from an initial concentration of 80 mg/L as $CaCO_3$.
- Alkalinity decreased upto dosing of 20 meq/l and after this starts decreasing, likewise the pH was continuously increasing upto dosing of 22 meq/l and at 24 meq/l it increased
- Fluoride was reduced to 2.12 mg/l at dosing of 22 meq/l and this value increased at next dose.
- SET-3 (Varying Dosage from 10 to 18 meq/l of Lime with Mg^{++} 50 mg/l as $CaCO_3$)
- Total hardness and Ca^{++} hardness was reduced continuously.
- Alkalinity was also reducing from an initial level of 750 mg/l as $CaCO_3$ to 410 mg/l as $CaCO_3$.
- pH increased from 8.66 to 10.41.
- Fluoride is reduced to a concentration of 2.15 mg/l at 18 meq/l dosing.
- SET-4 (Varying Dosage From 10 to 18 Meq/l of lime with Mg^{++} 75 Mg/l as $CaCO_3$)
- Total hardness and Ca^{++} hardness was reduced continuously and rapidly.
- Alkalinity was also reducing from an initial level of 700 mg/l as $CaCO_3$ to 350 mg/l as $CaCO_3$.
- pH increased from 8.58 to 10.73.
- Fluoride is reduced to a concentration of 1.98 mg/l at 18 meq/l dosing it was 2.16 mg/l at a dosing of 16 what is almost equal to the residual fluoride at 18 meq/l in SET -3.
- SET-5 (Varying Dosage from 10 to 18 meq/l of lime with Mg^{++} 100 mg/l as $CaCO_3$)
- Hardness and alkalinity are reducing as in the previous Sets and the pH is also increasing likewise.
- But the fluoride removal is not as efficient enough as in the previous sets which shows the limitations of adding Mg^{++}.
- SET -6 (Varying alum dosage from 70 mg /l to 110 mg /l).
- Total hardness and Ca^{++} hardness increased during the process while the Alkalinity reduced and pH increased.
- Residual fluoride concentration was reduced to 1.8 mg/l at dosing of 90 mg /l but starts increasing beyond this.
- SET -7 (Varying alum dosage from 70 mg /l to 110 mg /l & lime from 10 to 18 meq/l)
- Hardness increased, alkalinity reduced, pH remain almost neutral (around 7).
- But the fluoride removal was not significant and unpredictable.

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