An evaluation of the activated alumina process for fluoride removal from drinking-water and some factors influencing its performance

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Abstract

The activated alumina defluoridation process was evaluated on an underground mine water containing approximately 8 mg/ℓ of fluoride. The objectives of the study were to compare regeneration methods (caustic soda vs. aluminium sulphate (alum)), determine the importance of feedwater pH adjustment (pH 5 to 6) on process performance, establish design criteria for a 518 m³/d plant, and to determine the economics of the process.

Fluoride could be reduced from approximately 8 to less than 1 mg/ℓ. Approximately one and a half times as much product water of a better quality could be produced with caustic soda than with alum as regenerant. Silicate and hydroxyl ions appeared to compete more strongly with fluoride ions for exchange sites when the pH of the feed was greater than 7. This resulted in a much reduced capacity for fluoride removal. The operating fluoride exchange capacity for design purposes was determined at 3,2 g/ℓ alumina and the total defluoridation costs for a 518 m³/d plant were calculated at R0,33/m³ of treated water.

Introduction

The presence of fluoride in drinking-water may be beneficial or detrimental to public health depending on its concentration (Holden, 1970; Bishop and Sancoucy, 1978). Fluoridation of drinking-water to a level of 1 mg/ℓ has been shown to reduce dental cavities among children. No known deleterious effects of drinking fluoridated water have been observed when the concentration of fluoride is kept within prescribed limits. However, when water supplies contain excessive fluoride concentrations, the teeth of most young consumers become mottled with a permanent black or grey discolouration. Children who have been drinking water containing 5 mg/ℓ or more of fluoride are invariably afflicted and, for many, the enamel becomes so severely pitted that they eventually lose their teeth. Bone changes and crippling fluorosis may also result from the long-term consumption of water containing 8 to 20 mg/ℓ of fluoride (Hodge and Smith, 1954) or from a total intake of 20 mg of fluoride per day for 20 years or more (Roholm, 1937).

There are groundwaters in the Republic of South Africa which contain from about 2 to 20 mg/ℓ fluoride, with levels up to 10 mg/ℓ being fairly prevalent (Macdonald, 1976).

A number of methods can be used for the removal of fluoride from water. These can be divided into two categories – those based upon the addition of chemicals to cause precipitation or co-precipitation during coagulation and those based upon ion exchange or adsorption.

The chemical methods include the use of lime (Boruff, 1934; Maier, 1947; Zabban and Jewett, 1970), magnesium (Sorg, 1978) and aluminium sulphate (alum) (Culp and Stoltenberg,

1958). Theoretically, lime can reduce fluoride to no lower than 8 mg/ℓ while aluminium and magnesium sulphate can reduce fluoride to lower than 1,5 mg/ℓ. However, excessive and costly dosages are required and a sludge disposal problem also arises.

Adsorption methods include the use of activated carbon (McKee and Johnstone, 1934; Seth, 1965), strong base anion exchange resins (Thompson and McGarvey, 1953), bone char (Cillié, 1955), tricalcium phosphate (Sorg, 1978) and activated alumina (Savinelli and Black, 1958; Wu, 1978; Rubel and Woosley, 1978, 1979; Choi and Chen, 1979; Sorg, 1978). Of these methods, the activated alumina process appears to be the most attractive because alumina is somewhat specific for fluoride and has a relatively high fluoride exchange capacity. Activated alumina is not very friable and is not seriously affected by the concentrations of chlorides and sulphates usually encountered. Regeneration, which can be performed with either caustic soda or alum (Savinelli and Black, 1958) is fairly straightforward and the process seems to be reliable, safe and relatively simple to use. A disadvantage, especially for unskilled operators, is that it needs a neutralization step with acid after caustic soda regeneration and that the feedwater pH must be adjusted to between 5 and 6 to create optimum fluoride adsorption conditions (Rubel and Woosley, 1979).

Based on a paper study, the activated alumina defluoridation process was selected for pilot evaluation using an underground mine water containing approximately 8 mg/ℓ of fluoride. The objectives of the study were to –

- compare regeneration methods (caustic soda vs. alum);
- determine the importance of feedwater pH adjustment to between 5 and 6 on process performance;
- establish design criteria for a 518 m³/d plant; and
- determine the economics of the process.

Fluoride quality requirements

The US Environmental Protection Agency has made the maximum allowable fluoride level for drinking purposes dependent on climatic conditions because the amount of water consumed and, consequently, the amount of fluoride ingested, is influenced primarily by ambient temperatures.

The maximum allowable fluoride concentrations are shown in Table 1 (National Interim Primary Drinking Water Regulations, 1975) as are the recommended fluoride concentrations established by the US Public Health Service (*Drinking Water Standards*, 1962).

The SA Bureau of Standards has recommended a limit of 1 mg/ℓ for fluoride in drinking-water supplies. The maximum allowable concentration is 1,5 mg/ℓ (SA Bureau of Standards, 1971).
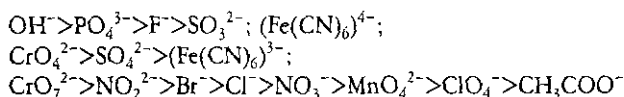
Annual average of maximum daily air temperatures (°C)	Recommended fluoride† concentration (mg/ℓ)			Maximum allowable* fluoride concentration (mg/ℓ)
	Lower	Optimum	Upper	
10 – 12,0	0,9	1,2	1,7	2,4
12,1 – 14,6	0,8	1,1	1,5	2,2
14,7 – 17,7	0,8	1,0	1,3	2,0
17,8 – 21,4	0,7	0,9	1,2	1,8
21,5 – 26,2	0,7	0,8	1,0	1,6
26,3 – 32,5	0,6	0,7	0,8	1,4

*National Interim Primary Drinking Water Regulations, 1975.
†Drinking Water Standards, 1962.

The activated alumina process

Activated alumina (Al_2O_3) is calcined granules of hydrated alumina. This material functions as an anion exchanger and the common anions are selected in the following order by acid-treated activated alumina (Clifford et al., 1978):

$$OH^->PO_4^{3-}>F^->SO_3^{2-}; (Fe(CN)_6)^{4-};$$
$$CrO_4^{2-}>SO_4^{2-}>(Fe(CN)_6)^{3-};$$
$$CrO_7^{2-}>NO_2^->Br^->Cl^->NO_3^->MnO_4^{2-}>ClO_4^->CH_3COO^-$$

The high selectivity of activated alumina for fluoride compared with normal synthetic anion exchangers makes it extremely suitable for fluoride removal from water containing ions such as sulphates, chlorides and bicarbonates, which may compete for the limited adsorption sites.

Activated alumina reacts in a similar way to weak acid or weak base resins in that both acids and bases must be used for the adsorption-regeneration cycle. A typical adsorption-regeneration cycle for the removal of fluoride may be presented as follows, according to the analogy of Clifford et al. (1978):
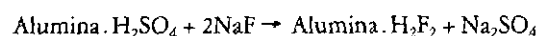
Acidification

When neutral (water washed) alumina, which is represented as alumina HOH, is treated with acid, e.g. sulphuric acid, acidic alumina is formed.

$$Alumina.HOH + H_2SO_4 \rightarrow Alumina.H_2SO_4 + HOH$$
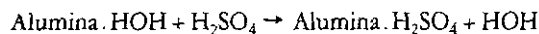
Ion exchange (loading)

If this acidic form is brought into contact with fluoride ions, they displace the sulphate anions.

$$Alumina.H_2SO_4 + 2NaF \rightarrow Alumina.H_2F_2 + Na_2SO_4$$

Loading is usually best performed under slightly acidic conditions (pH 5 to 6) thereby reducing the effects of competing ions such as hydroxyl and silicate and optimizing fluoride adsorption (Rubel and Woosley, 1978). The product water is, therefore, slightly acidic but can be stabilized with either caustic soda, soda ash or lime.

Backwashing

This is carried out upwards (50% bed expansion), after loading to remove suspended solids, to redistribute the alumina particles and break up any tendency towards wall effects and channelling.

Regeneration

To regenerate the alumina which is saturated with fluoride, a dilute solution of the most preferred anion, hydroxide, is used.

$$Alumina.H_2F_2 + 3 NaOH \rightarrow Alumina.NaOH + 2 NaF + 2 H_2O$$
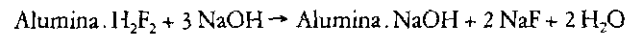
Rinsing

This is necessary to remove excess regenerant prior to neutralization.

Neutralization

To restore the fluoride removal capacity, the basic alumina is contacted with dilute acid.

$$2 Alumina.NaOH + 3 H_2SO_4 \rightarrow 2 Alumina.H_2SO_4 + Na_2SO_4 + 2 H_2O$$
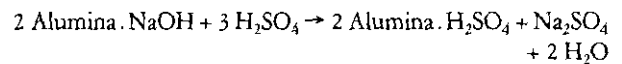
The acidic alumina (alumina.H_2SO_4) is now ready for another adsorption cycle.

Experimental

An activated alumina pilot plant with a capacity of 32 m³/d was designed and built using criteria obtained from the literature (Rubel and Woosley, 1979) and from our own experimental results (Fig. 1).
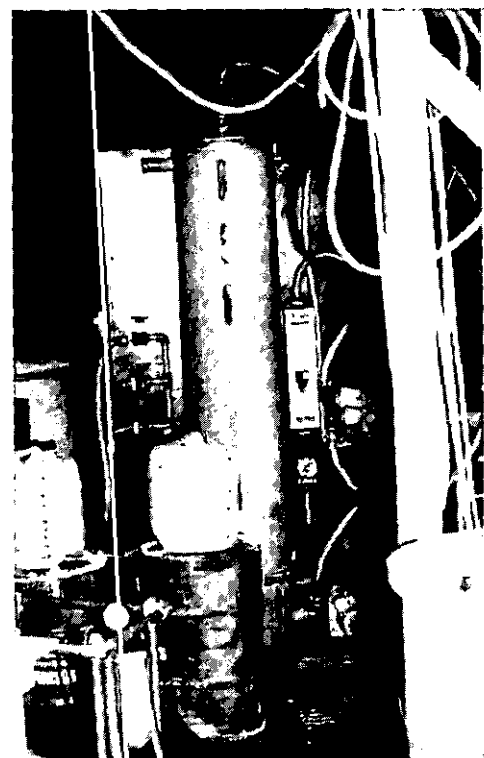
Figure 1
Activated alumina pilot plant.

The column (diameter 380 mm, cross-sectional area 0,113 m²) was filled to a depth of 1 m with 113 ℓ ALCOA grade F-1 activated alumina of particle size 0,5 to 1,0 mm. In order to prevent blockage of the underdrain system with fines, 200 mm of 7 mm stone preceded the activated alumina. About 1 m of free space was left above the alumina to allow for at least 50% bed expansion during backwashing.

The flow through the column could be directed either downwards or upwards by means of a multiport valve. All piping consisted of 2,54 mm plastic hose. Saunders and ball valves were used.

Glass fibre tanks of approximately 2 m³ each were used to store regenerant solution and acid. A galvanized tank (4 m³) was used for product water storage; this water was also available for rinsing purposes. Spent regenerant, backwash and rinse waters could be diverted to waste.

Sandfiltered, chlorinated water from the water treatment plant at the test site was used as feed to the activated alumina pilot plant. Thirty-two runs were carried out with caustic soda and alum as regenerants with and without pH control of the feed water and breakthrough curves established. The breakthrough point for fluoride was taken to be 1,5 mg/ℓ and the number of bed volumes (BV output) produced at breakthrough was determined from these curves. Fluoride and silica determinations were done with a fluoride specific ion electrode and by atomic absorption, respectively.

Results and discussion

Comparison between caustic soda and alum as regenerants

The results achieved using 10 bed volumes of 1 % caustic soda

and of 1 % alum as regenerants with pH adjustment of the feed water (pH 5 to 6) are shown in Fig. 2.

The output of product water using alum as regenerant (run 32) is approximately 65 % of that obtained using caustic soda (run 18) and where less rinse water was used (run 22), it was not possible to produce a product water containing less than 1,5 mg/ℓ fluoride. Excessive amounts of rinse water were necessary to produce a water of the desired quality (<1,5 mg/ℓ F⁻) after alum regeneration and, furthermore, a poorer quality water was produced than after caustic soda regeneration. This poor performance with alum as regenerant may possibly be ascribed to the precipitation on the treatment bed of a partially soluble aluminium-silica-fluoride complex during regeneration. This precipitate appears to be fairly difficult to remove and may explain the high initial fluoride content of the product water as experienced in runs 22 and 32, which may be ascribed to the resolubilization of this compound.

Caustic soda regeneration without pH control

Runs 28 and 29 were performed with 10 bed volumes of 1 % caustic soda as regenerant to determine the effect of operating the plant with a feed water of pH 7,4 to 7,9. The number of bed volumes of satisfactory product decreased from 374 (run 26 with pH control) to 182 (run 28) and 119 for run 29 (Fig. 3).

This clearly shows that output production is reduced when the pH is not controlled. This is most probably due to the adsorption of silica and hydroxyl ions (as indicated in Fig. 3 by the lower silica and pH values of the product water) which compete with fluoride ions for the available exchange sites at higher pH values.
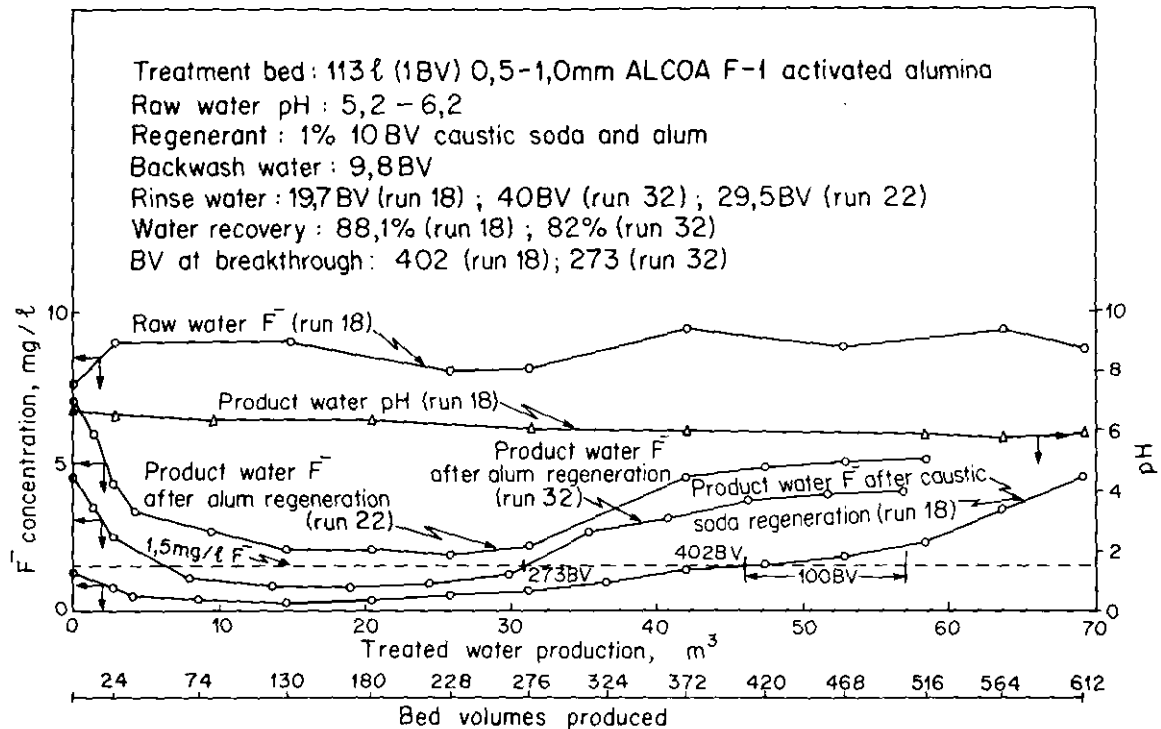

Figure 2
Product F⁻ concentration and pH profile after caustic soda and alum regeneration with pH control of the feed water.
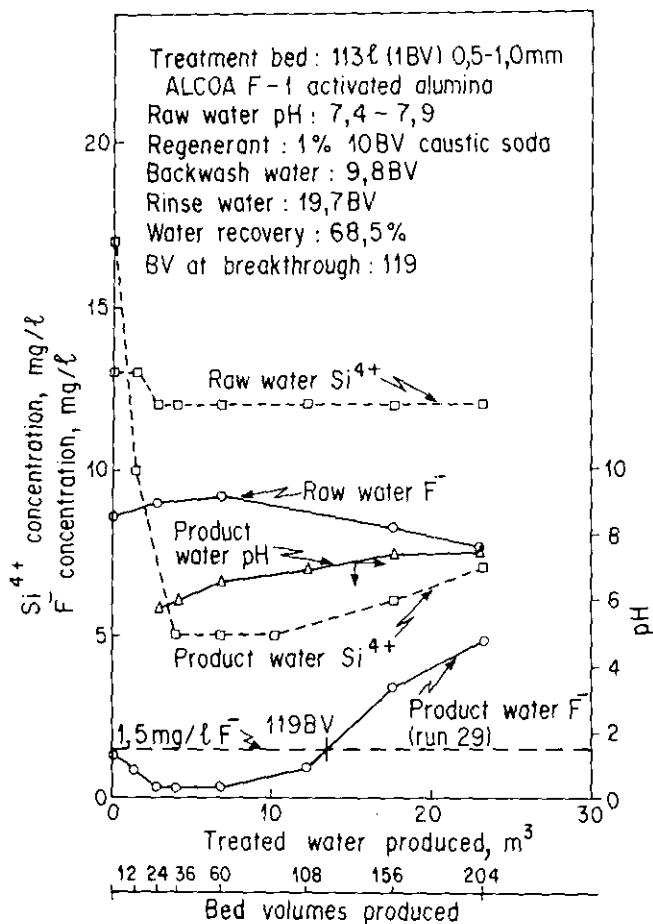
Figure 3
Product F⁻ concentration and pH profile after caustic soda regeneration, without pH control of the feed water.

Caustic soda regeneration with pH control to determine reproducibility

Runs 14 and 19 were performed with 10 bed volumes of 1 % caustic soda as regenerant to determine reproducibility. Treated water bed volumes of 382, 389, 490, 354, 402 and 456 were produced, respectively. Run 16 was exceptionally good while run 17 was comparatively bad. However, the acid dosing pump failed halfway through run 17, causing poor pH control, an increase in silica adsorption and a decrease in output (Fig. 4).

No significant reduction in performance was observed during these runs with pH control of the feed water. Run 18 (Fig. 2) in which 402 bed volumes of treated water were produced at a water recovery of 88, % may be considered as a good average run.

Caustic soda regeneration with 5 bed volumes of regenerant

Runs 20 and 21 were performed using 5 instead of 10 bed volumes of 1 % caustic soda as regenerant to determine whether output would be affected. The numbers of bed volumes produced were 402 and 407, respectively, indicating that a reduction from 10 to 5 bed volumes did not have any significant adverse effect on the output of defluoridated water. Time did not permit further optimization studies to determine the minimum regenerant requirement.

Water quality

A typical chemical analysis of the raw and product water is shown in Table 2.

The increase in sulphate and reduction in bicarbonate are caused by sulphuric acid for pH control of the feed. The Langelier Index of the raw water is positive, which indicates that the raw water is scale-forming while the index of the product water is

Figure 4
Product F⁻ concentration and pH profile after caustic soda regeneration, with and without pH control of the feed water.
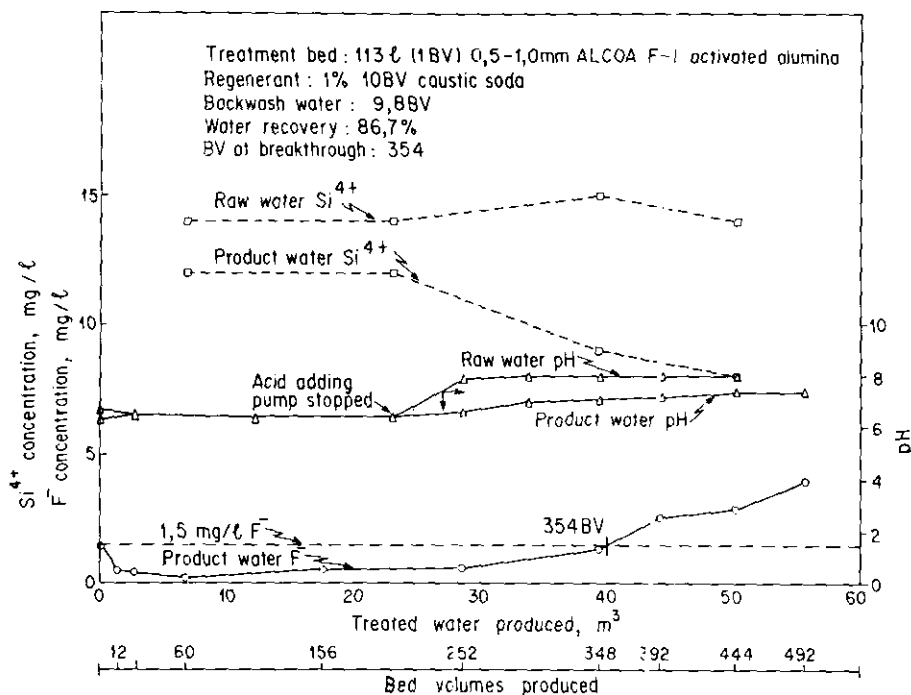
TABLE 2		
CHEMICAL ANALYSIS OF RAW AND ACTIVATED ALUMINA PRODUCT WATER (RESULTS IN mg/ℓ WHERE APPROPRIATE)		
Constituent	Raw water	Product
Na^+	60	56
K^+	6	6
Ca^{2+}	96	94
Mg^{2+}	43	43
Cl^-	27	26
SO_4^{2-}	310	390
HCO_3^-	171	39
F^-	8	<1
pH	7,7	6,5
TDS	720	654
LI*	+0,26	-1,59

*Langelier index

ther reduced by optimizing the rinsing and regeneration requirements.

Flocs in regenerant tank

It was observed during caustic regenerant make-up using raw feed water that cloudy flocculant material formed in the regenerant tank. These flocs (calcium and magnesium hydroxides) settled to the bottom of the tank, necessitating frequent cleaning. Provision should, therefore, be made to remove this material from the regenerant make-up tank.

Attrition losses

The activated alumina visually showed little evidence of attrition losses during the test period. However, such losses may be expected with time, but cannot as yet be quantified.

negative, indicating a corrosive tendency. This water (slightly acidic due to the lowering of the feedwater pH) may be treated with limestone in a suitable contact column to raise the pH and reduce its corrosivity, but this will increase its hardness. Alternatively, caustic soda or soda ash may be used, but at a higher cost.

Design criteria

Design criteria were established for fluoride removal from a typical pilot run, i.e. run 18 (see Fig. 2), details of which are given in Table 4.

Limestone treatment

The performance of a limestone column (0,14 m diameter, 1,2 m depth) loaded with 15,4 ℓ (1 m depth) limestone pebbles through which the activated alumina product water could flow at different rates, was evaluated for neutralization. The results are shown in Table 3.

TABLE 3				
LIMESTONE COLUMN NEUTRALIZATION RESULTS				
pH feed	pH product	pH increase	Flow rate (mℓ/min)	Retention time (min)
6,2*	7,0	0,8	260	59,2
5,9	6,4	0,5	500	30,8
5,9	6,3	0,4	1 080	14,3
6,1	6,4	0,3	1 600	9,6
6,0	6,2	0,2	4 400	3,5

*Ca^{2+} increase from 94 to 130 mg/ℓ

The neutralization process, a slow, time-independent process, is reasonably effective in reducing the corrosivity of the treated water (at pH 7 the Langelier Index had been reduced from -2,33 to -0,96). However, it was found that the calcium content increased from 94 to 130 mg/ℓ in the product water, making the already hard water even harder.

Water recovery

The raw water which is used for backwash prior to regeneration may be circulated to the raw water inlet. This will increase water recovery by about 2 % (from 88 to 90 %) (Fig. 2, run 18). The approximate 10 % waste produced is high in hydroxides, sulphates and fluoride and should be disposed of in such a way as not to pollute the environment. This waste, however, may be fur-

TABLE 4

Activated alumina (ALCOA type F - 1*)
  Grading: 0,5 to 1,0 mm
  Fluoride (F -) exchange capacity: 3,2 g/ℓ
  Bed depth: 1,0 top 1,5 m

Feedwater retention (contact) time in column: 5 min minimum

Backwash (50% bed expansion desirable)
  Flow rate (adjust as required): 980 $\ell\,m^{-2}.min^{-1}$
  Duration: 10 min (approx.)

Regenerant (aqueous caustic soda solution): 10 kg $NaOH/m^3$
  Regenerant volume: 5 bed volumes**
  Regenerant upflow rate: 100 $\ell\,m^{-2}.min^{-1}$

Rinsing (after caustic soda regeneration)
  Flow rate (downwards): 200 $\ell\,m^{-2}.min^{-1}$
  Period: 30 min (approx.)

Acid used for neutralization of alumina: 1,53 kg sulphuric acid (1,836 kg/ℓ) per m^3 feed water
  Volume of dilute sulphuric acid required: approximately 15 BV (until pH is about 8)
  Neutralizing acid flow rate (downward): 200 $\ell\,m^{-2}.min^{-1}$

pH Adjustment of feed water to exchange column using dilute sulphuric acid   The amount of acid required must be determined by calculation or experimentally for each feed water. This may change from time to time and automatic pH control is therefore preferable.

*The activated alumina used was obtained from ICI, 1 Leyds Street, Braamfontein, Johannesburg. (This firm is not necessarily the best or only supplier and any other firm may be approached.)
**1 Bed volume = volume of alumina in column.

The output of defluoridated water per regeneration cycle depends on the fluoride concentration of the feed water. At a fluoride concentration of 8 mg/ℓ, approximately 400 bed volumes are produced.

Activated alumina deteriorates with time owing to abrasion and irreversible exchange reactions. A lifetime of about 4 years is assumed, i.e. approximately 25 % fresh alumina may have to be added annually.

Process design of a 518 m³/d defluoridation plant: design philosophy

For ease of operation, the process design has been based on a. service cycle of 67 h, followed by regeneration taking approximately 5 h. Total cycle time will thus be 72 h (3 days). Two such cycles (6 days) will be necessary to produce the required amount of product water (3 626 m³ per week).

The proposed operating schedule is as follows:
Regeneration: Monday 07h00 to 12h00
First loading cycle: Monday 12h00 to Thursday 07h00 (67 h)
Regeneration: Thursday 07h00 to 12h00
Second loading cycle: Thursday 12h00 to Sunday 07h00

This plant, operating at below maximum throughput rate (9,9 min feedwater retention time) will produce 3 626 m³ water in six days with the possibility of producing an additional 446 m³ (100 BV) of water with a fluoride content of between 1,5 and 2,2 mg/ℓ (Fig. 2), if kept on line on Sundays, should the need arise. This water blended with that already in storage should be perfectly satisfactory for use.

The plant capacity can be increased to about 1 000 m³/d by operating at the designed maximum flow rate (5 min feedwater retention time) and regenerating more frequently (every 32 h). A diagram of the proposed process flow sheet is shown in Fig. 5.

Costs

Total defluoridation costs for a 518 m³/d plant have been determined at R0,33/m³ (Appendix). These include amortization of R146 000 capital over 20 years at 10 %/a interest. Also included are the costs of product water stabilization equipment, chlorination and labour, but excluded are those of product storage and brine disposal. Costs are also affected by factors such as plant size, required regeneration frequency and safety factors. In the case quoted, regeneration was performed after 67 h loading (two regeneration cycles per week) and 25 %/a alumina replacement was assumed. However, with more frequent regeneration, smaller equipment may be used for the same output, with a concomitant reduction in capital cost.

Conclusions

● Fluoride can be reduced from approximately 8 mg/ℓ to less than 1 mg/ℓ by the activated alumina process.

● Approximately one and a half times as much product water of a better quality can be produced with caustic soda than with alum as regenerant.

● The precipitation of a partially soluble aluminium-silica-fluoride complex when using alum for regeneration appears to have an adverse influence on the adsorption capacity of the alumina.

● Silicate and hydroxyl ions compete more strongly with fluoride for exchange sites on the activated alumina when the pH value of the feed is greater than 7. This results in a much reduced capacity for fluoride removal. However, this effect can be remedied by pH adjustment of the feed water to between 5 and 6, at which level fluoride is adsorbed preferentially.

● The product water is slightly acidic and, therefore, corrosive. Limestone treatment has been found to be effective in reducing the acidity and increasing the stability of the defluoridated water. However, this treatment increases calcium hardness. Caustic soda or soda ash may also be used at an increased cost.

● Water recoveries of more than 90 % are possible.

● Provision has to be made to dispose of the regenerant and rinse waters, which amount to about 10 % of the water treated and contain high concentrations of fluoride, caustic soda and sodium sulphate.

● The total defluoridation costs for a 518 m³/d plant were calculated at R0,33/m³ of treated water.

Figure 5
Diagram of proposed flow sheet for activated alumina plant (one of two columns)

Acknowledgements

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Appendix: Water treatment costs

A summary of defluoridation costs with an activated alumina plant is as follows:

Design capacity: 700 m^3/d

Type of plant: Activated Alumina Plant for Fluoride Removal.

Plant life: 20 years

Plant availability: 0,92

Average capacity factor: 0,8

Plant factor: 0,74
∴ Daily output = 700 x 0,74 = 518 m^3

Recovery factor: 90%

Capital costs

Direct:

	R
1. Feed water supply development (not applicable)	—
2. Feed water treatment* (not applicable)	—
3. Activated alumina plant, including buildings (R5 000)	120 000

Two vessels, alumina, pumps, valves, piping, regenerant and acid storage tanks, stabilization equipment, transportation, installation, etc.

4. Site development (not included)	—
5. Energy source development (not applicable)	—
6. Electrical switch gear (Guestimated)	2 500
7. Brine and sludge disposal (not provided)	—
8. Product water storage (not included) and chlorination	2 000
9. Other: Caustic soda and sulphuric acid Storage in 20 m^3 (R3 000) and 5 m^3 (R2 000) tanks (Guestimated costs)	5 000

SUB-TOTAL: Direct Capital Costs (1-9) → 129 500
say 130 000 ...(A)

Indirect Capital costs:

	R
10. Interest during construction (10% of Direct Capital for 1 month)	1 083
11. Project management fees (not applicable)	—
12. Contingencies 10 %	13 000
13. Start-up costs (2 weeks of O & M costs)	1 761
14. Other	—

SUB-TOTAL: Indirect Capital Costs (10-14)   15 844 ... (B)
TOTAL DEPRECIABLE CAPITAL COSTS
(A + B)   → 145 844 ...(C)

*Feed water is the water from the filter plant.

Other capital costs (non depreciable)

	R
15. Land	—
16. Working capital (say 10 % of O & M costs)	4 579

TOTAL OF OTHER CAPITAL COSTS
(15-16) → 4 579 ...(D)
TOTAL OF ALL CAPITAL COSTS (C + D) → 150 423

Annual costs

Recurring costs

	R
17. Taxes (not applicable)	—
18. Insurance (not included)	—
19. Other	—
TOTAL ANNUAL RECURRING COSTS (17-19) →	NIL ... (E)

Operating and maintenance (O & M) costs

	R
20. Labour – Salaries*	15 675
21. Labour – General and Administrative Overhead (40 % of salaries)	6 270
22. Chemicals:	
Sulphuric acid (R104/ton)	3 221
Caustic soda (43 to 50% solution at R597/ton solids)	13 846
Activated alumina replacement (25 % of R11 507)	2 877
23. Supplies and maintenance materials (2 % of direct capital)	2 600
24. Membrane replacement (not applicable)	—
25. Special repairs or overhauls (no provision)	—
26. Energy – 0,23 kWh/m³ at R0,03/kWh	1 301

*Feed water is the water from the filter plant.

	R
27. Energy · Other (steam, etc.) (not applicable)	—
28. Other	—

TOTAL ANNUAL O & M COSTS (20-28) → 45 790 ... (F)

Annual fixed charge

10 % Interest and 20 year Plant Life: Capital
Recovery Factor (CRF) = 0,1175
Depreciable capital (C) x CRF = 145 844 x 0,1175
$$= R17 137 ...(G)$$
TOTAL ANNUAL COST (E + F + G) → R62 927

Unit production costs

Annual production
= Designed capacity x 365 d x Plant Factor
700 m³/d   x 365 d x   0,74   = 189 070 m³

$$\text{Unit cost} = \frac{\text{Total annual cost}}{\text{Actual annual production}} = \frac{R62\ 927}{189\ 070\ m^3}$$

$$= R0,33/m^3$$

Supervisory and labour requirements

	Number	Annual salary	Annual cost
Senior supervisor (part-time)	1 x 0,125	R25 000	R 3 125
Plant engineer (shared service)	1 x 0,05	25 000	1 250
Mechanic (shared service)	1 x 0,10	15 000	1 500
Electrician (shared service)	1 x 0,05	18 000	900
Chemist (shared service)	1 x 0,05	18 000	900
Shift operator (shared service)	4 x 0,5	4 000	8 000
TOTAL ANNUAL LABOUR COST			R15 675