



Activated alumina-based adsorption and recovery of excess fluoride ions subsequent to calcium and magnesium removal in base metal leach circuits

by L. Lorenzen*‡, J.J. Eksteen*†, M. Pelser*‡, C. Aldrich*, and G. Georgalli*

Synopsis

An effective electrowinning process in hydrometallurgical industry requires fluoride levels in the base metal solution to be less than 10 mg/l. Selective removal of the fluoride ions from base solution is thus desired, if fluoride was added to control calcium and magnesium in the circuit. Consequently, adsorption of fluoride onto activated alumina was studied in a batch and a column set-up. The effects of base metal solution pH, temperature, initial concentration and flow rate on activated alumina performance were investigated in either a batch or column configuration. A two-level factorial experimental design was implemented in studying column dynamics. Results demonstrate that activated alumina is an effective adsorbent for selective removal of fluoride from base solution. In the batch operation, fluoride was removed to values below the maximum allowable concentration (10 mg/l) when pH was ≤ 8 . In the column adsorption step at 55°C and 600mg/l initial concentration, up to 16 bed volumes were processed before breakthrough level was reached. Desorption step using 1% sodium hydroxide solution achieved an elution of 8 bed volumes. The activated alumina (AA) had a capacity of 8.65 gF/l AA at the 10 mgF/l fluoride breakthrough level during the column adsorption test.

Keywords: Fluoride; activated alumina; adsorption; recovery; regeneration.

Introduction

The presence of calcium and magnesium in the hydrometallurgical processing of base metals sulphates is responsible for problematic gypsum formation during solvent extraction of nickel and large bleed streams during electrowinning. Thus, to ensure good operation of various unit processes, it is important to control the levels of calcium and magnesium. One method that can check the levels of these alkaline earth metals is fluoride precipitation. This technique is, however, criticized for the excess amount of unreacted fluoride in the production line (Babjak, 1986). Typically, the unreacted fluoride concentrations can be as high as 1000 mg/l. At such high concentration, the fluoride ions would adversely affect the electrowinning process. In the zinc processing industry for instance, the presence of fluoride during electrowinning would result in the firm

sticking of the zinc to the aluminium starting sheets (Booster *et al.*, 2001). To minimize such effects, it is desirable to lower the levels of fluoride in the production line to acceptable values, which should be below 10 mgF/l. Several technologies for removal of fluoride from solution, such as the use of filtration membranes, precipitation/coagulation, ion exchange and adsorption, are available. Unfortunately, most of these techniques may not be applicable to the hydrometallurgical industry for reducing the levels of fluoride because they are not sufficiently selective.

Adsorption technology is frequently used as a robust technique to remove water-soluble ions from aqueous solution. To remove fluoride from solution by adsorption technique, locally available, efficient and selective adsorbents are required. Presently, the WHO recognizes activated alumina (AA) adsorption as one of the best demonstrated available technology (BDAT) for water defluoridation. Consequently, a number of researchers have in the recent past investigated the performance of activated alumina in water defluoridation under varying process conditions. For instance, Ghorai and Pant (2004), Li *et al.* (2001), Lounici *et al.* (1997), Ku and Chiuo (2002), Rubel and Woosley (1979), Wasay *et al.* (1996) investigated the fluoride sorption behaviour of activated alumina in either batch or column experiments. The investigations were limited to low concentration of fluoride in water in a single component system. Moreover, a wide variation in fluoride sorption capacity of activated alumina was reported. In industrial concerns such as the hydrometallurgical processing of base metal, the concen-

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tration of fluoride in the process stream is very high as stated earlier. Moreover, the stream contains other water-soluble species that may influence the sorption process. Before applying an adsorbent to such a stream, it is important to generate data that give a preliminary indication of the performance of the adsorbent. Furthermore, the adsorbent should be regenerable and where the adsorbing species has reuse value, it should be recovered.

In recent years there has been a large impetus given to the recovery of valuable resources as a consequence of deriving maximum benefit from such resources. In the hydrometallurgical industry, the recovery of adsorbent-bound fluoride is not only an economic necessity but also a means of minimizing the environmental impacts that would arise from used-adsorbent waste disposal. In general, there exists a number of techniques for recovering/desorbing a probe ion and regenerating the sorption media. These techniques include: electrodeposition (Lounici *et al.*, 2001), chemical method (Vieira and Beppu, 2006), thermal methods (Wang *et al.*, 2006; Luo *et al.*, 2006), and the use of physical waves e.g. ultrasonication (Breitbach *et al.*, 2002), among others. Of these techniques, the chemical method using a strong base such as NaOH, because of its simplicity, is the most commonly applied in desorbing fluoride from the sorption media (Ruixia *et al.*, 2002; Lounici *et al.*, 2001).

The objectives of this study were, therefore, to investigate the feasibility of removing by adsorption and recovering un-reacted fluoride during the hydrometallurgical processing of base metal so that the fluoride-containing stream can be reused in the production line. A batch adsorption unit and a continuous flow adsorption column packed with activated alumina were used to remove fluoride from the base metal solution. The batch adsorption experiment investigated the effect of pH on fluoride sorption. For the continuous flow adsorption unit, the influence of the flow rate, temperature and initial concentration was investigated and is presented in terms of breakthrough curves. A two-level (low and high) factorial experimental design method was used to investigate the influence of the above operating parameters in an activated alumina fixed-bed sorption column. Then, the fluoride-loaded activated alumina was desorbed *in situ* using NaOH solution at various concentration levels. Finally, the regeneration of the used activated alumina adsorption media was done by passing H₂SO₄ solution through the bed.

Materials and methods

Composition of base metal solution

Table I summarizes the composition of the base metal solution, which is typical of solution leaving the iron precipitation unit during the hydrometallurgical processing of nickel-copper-cobalt ores. Calcium and magnesium were not included since their concentrations were very low. The base metal solution was made from analytical grade sulphate salts obtained from Merck and Sigma-Aldrich. The salts were weighed and dissolved in distilled water using 2 l volumetric flasks. The solution was then heated to 55°C while dissolving the salts and made up to the 2 l mark at this temperature. This mode of preparation was found to give the most consistent composition of base metals sulphate solution.

Batch adsorption: effect of solution pH

Experimental studies were conducted in a batch reactor to evaluate the effect of solution pH on the removal of fluoride from solution whose composition is summarized in Table I. The batch reactor consisted of a cylindrical container made of polypropylene (PP) material, which was resistant to the fluoride solution, especially when operation was done at low pH. Table II summarizes the dimensions of the reactor and some batch experimental conditions.

Before the fluoride removal experiment was conducted, the activated alumina (100 g for each experiment) adsorbent was soaked in distilled water overnight to ensure complete wetting. The activated alumina was then brought into contact with sulphate solution (see Table I for composition) containing fluoride at a fixed concentration of 600 mg/l. Ammonium fluoride was used as the fluoride source. The adsorbent-solution was allowed to equilibrate for a predefined period (24 hours). At the end of the experiment, samples were taken from the reactor and residual concentration of fluoride measured. Operation at a different pH value (in the range of 5–12) was achieved by adjusting the pH of freshly prepared solution by using either sulphuric acid (H₂SO₄) or sodium hydroxide (NaOH).

Fluoride removal and recovery in column: adsorption-desorption cycle

Figure 1 shows the schematic of the jacketed activated alumina fixed-bed column used to perform fluoride adsorption-desorption experiments. In the adsorption step, a preheated base metal sulphate solution containing fluoride was fed under gravity from the top of the column containing 50 g of activated alumina for which the flow rate was controlled at 1000 l/m²·min (when flow was not a variable) by two valves positioned at the top and bottom of the column.

Table I

Composition of the synthetic solution

Metal ion	Concentration (g/l)
Nickel (Ni)	32.7
Cobalt (Co)	1.87
Manganese (Mn)	0.305
Zinc (Zn)	0.121

Table II

Batch reactor configuration

Description	Dimension
Volume	1 l
Diameter	105 mm
Width of baffles	10 mm
Number of baffles	6
Diameter of impeller	60 mm
Material of construction	PP
Type of impeller	Teflon marine propeller
Stirring rate	520 rpm
Contact time	24 hours

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Figure 1—Jacked column packed with activated alumina used for the adsorption-desorption cycle

The variables studied included changes in flow rate, temperature and initial concentration. A two-level factorial experimental design was used to study the effects of these variables on breakthrough curves characteristics and adsorption capacity of activated alumina. During the experimental run, a 100 ml sample was collected at the bottom of the column at a given time interval to determine the residual fluoride concentration. This procedure facilitated the determination of breakthrough curves, which for our case is a plot of percentage approach to initial concentration against bed volume. The column capacity was determined from Equation [1]

$$q_{10} = \frac{C_o Q}{m} \int_0^{t_{10}} \left(1 - \frac{C_t}{C_o}\right) dt \quad [1]$$

Where q_{10} is the column capacity at 10 mgF/l, C_o is the initial concentration (mgF/l), C_t is the concentration exiting the column at any time (mgF/l), m is the activated alumina bed mass (g) Q is the volumetric throughput (l/min) and t_{10} is time at which the column exit concentration reaches a value of 10 mgF/l (min).

Meanwhile, water from a water-bath was circulated upwards through the column jacket to maintain the bed temperature, from where it was returned to the bath. To make a cost-effective and user-friendly process, the activated alumina adsorbent should be regenerated and fluoride recovered. Thus, desorption studies were carried out by passing a 0.1% or 1% NaOH solution through the activated alumina column loaded with fluoride ions.

Fluoride analysis

The residual concentration of fluoride from the batch reactor and adsorption column was measured by a fluoride ion selective electrode. Accordingly, a calibration curve was initially prepared by recording the potential values for a range of known fluoride concentration. To ensure that other ions did not interfere with the fluoride ion measurements, A total ionic adjustment buffer (TISAB, pH 5.5) solution was prepared and used during fluoride ions concentration measurements. The fluoride concentrations were then calculated from the measured potential values. By carrying an appropriate material balance, the quantities of fluoride retained by the activated alumina adsorbent was determined.

Solid phase characterization

Activated alumina manufactured by Unilab was used as the adsorbent. The BET surface area was measured using the nitrogen adsorption-desorption method. The BET surface area was 136 m²/g. To determine the major phase in activated alumina, the adsorbent was subjected to X-ray powder diffraction (XRD) analysis with scanning mode of 2θ/θ. The γ-alumina phase was identified.

Factorial experimental design

A two-level experimental design was used to study the effect of changing the variables during column operation. For each variable (denoted by X_1 or X_2) a high level (coded as +1) and a low level (coded as -1) was selected (see summary in Table III), which was evenly spread around the centre run (coded as 0).

The effect of a variable is defined as the difference between the average response (y) at the high level of the variable and the average response at the low level. For X_1 and X_2 the effects are calculated according to Equations [2] and [3]

$$Effect\ of\ X_1 = \frac{(y_{(+1,-1)} + y_{(+1,+1)})}{2} - \frac{(y_{(-1,-1)} + y_{(-1,+1)})}{2} \quad [2]$$

Table III

Tabulated factorial design

X_1	X_2	y
- 1	-1	$y_{(-1,-1)}$
+1	-1	$y_{(+1,-1)}$
- 1	+1	$y_{(-1,+1)}$
+1	+1	$y_{(+1,+1)}$
0	0	$y(0, 0)$

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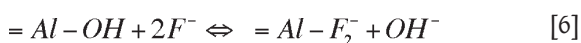
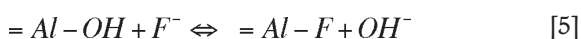
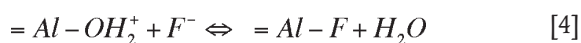
$$\text{Effect of } X_2 = \frac{(y_{(-1,+1)} + y_{(+1,+1)})}{2} - \frac{(y_{(-1,-1)} + y_{(+1,-1)})}{2} \quad [3]$$

Results and discussion

Batch adsorption

Removal of fluoride ions from water by adsorption onto various adsorption media is a robust and effective technique that has been reported by several researchers (Abe *et al.*, 2004; Mjengera and Mkongo, 2003; Moges *et al.*, 1996; Wang and Reardon, 2003; Ramos *et al.*, 1999; Onyango *et al.*, 2004, 2005, 2006, Sujana *et al.*, 1998). In particular, the application of activated alumina (AA) adsorption in removing fluoride ions from water is described by the WHO and USEPA as the best demonstrated available technology (BDAT). Previous studies using AA, however, were limited only to low concentration of fluoride ions in water (Ghorai and Pant, 2004; Li *et al.*, 2001; Lounici *et al.*, 1997; Ku and Chiuo, Rubel, Wasay *et al.*, 1996). South Africa has many hydrometallurgical industries and activated alumina is locally available. Consequently, this work makes an attempt to apply adsorbent in removing fluoride at high concentrations from base metal sulphate solution (see Table I for composition) and to recover the adsorbed fluoride in a desorption step. Removal of fluoride is mandatory to meet the maximum allowable concentration of 10 mgF/l in electrowinning circuits during hydrometallurgical processing of base metals. It has been shown that AA has high affinity for fluoride because in an aqueous environment at pH values below the activated alumina pH_{pzc} —the point of zero charge—it forms protonated ($=Al-OH_2^+$) and neutral ($=Al-OH$) aluminol sites, which are responsible for binding fluoride ions by formation of inner-sphere complexes (Onyango and Matsuda, 2006). The number and activity of these sites depend on pH of operation. Thus, we initially investigated the effect of pH on fluoride removal from base metal solution. The initial concentration of fluoride was fixed at 600 mg/l and activated alumina adsorbent mass at 100 g/l. Figure 2 is a plot of residual concentration of fluoride against equilibrium solution pH.

Two trends are observed. As the equilibrium pH increases from 5.5 to 8, the residual fluoride concentration remains relatively constant and is below the maximum allowable concentration value of 10 mgF/l. This observation may be due to the fact that the reaction vessel contained enough quantity of the adsorption media that provided a large number of terminal aluminol sites for binding fluoride. The aluminol sites-fluoride interaction is expressed by equilibria Equations [4]–[6]



Equation [4] expresses the interaction between protonated aluminol sites and fluoride ions, which is enhanced by coulombic attraction between the unlike charges, leading to the formation of inner-sphere complexes ($=Al-F$). Equation [5] also expresses the formation of inner-sphere complexes and release of hydroxyl ions from the slow

interaction between neutral sites and fluoride ions. Given that the current operation is performed at high concentration, there is a possibility of a reaction that proceeds according to Equation [6]. Above equilibrium pH 8, a rapid increase in residual fluoride concentration with an increase in equilibrium pH is observed. It is known that most activated alumina adsorbents have pH_{pzc} between 8 and 9. Though the reaction vessel contained a large amount of activated alumina, the sites were increasingly becoming negatively charged ($=Al-O^-$) at increased pH. This resulted in coulombic repulsion between the adsorbing fluoride ions and the negatively charged sites, leading to a reduction in fluoride removal efficiency. Results shown in Figure 2 suggest that operation should be done at $\leq pH$ 8.

Characteristics of fluoride adsorption in fixed-bed column

Column experiments were performed using a two-level factorial experimental design to study the effects of temperature, initial concentration and flow rate. The studied variables represented the conditions that can be found in a hydrometallurgical industry.

Effects of temperature and initial concentration

After the successful batch tests, fluoride adsorption behaviour in a fixed-bed column, expressed as breakthrough curves (BTC), was investigated in a continuous down flow mode of operation using 50 g activated alumina bed fed with fluoride-spiked base metal sulphate solution at a flow rate of 1000 l/m².min. The solution temperature and the inlet fluoride concentration were selected as external variables. Temperatures of 45 and 65°C and inlet fluoride concentrations of 400 and 800 mg/l were selected as the high and low levels in the factorial experimental design. A centre run, which was considered to be the standard condition, was an initial fluoride concentration of 600 mg/l and temperature of 55°C. The breakthrough point was taken to be 10 mgF/l and this value corresponded with the maximum allowable concentration of fluoride in the electrowinning circuit. Figure 3 shows the breakthrough curve for the adsorption step of the centre run variables. The variation in pH with bed volume processed is also included. The results are representative of

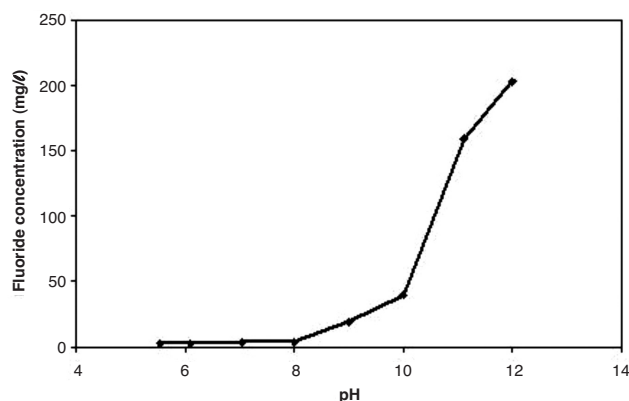


Figure 2—Fluoride concentration as a function of pH (initial: 600 mg/l, adsorbent mass 100 g/l)

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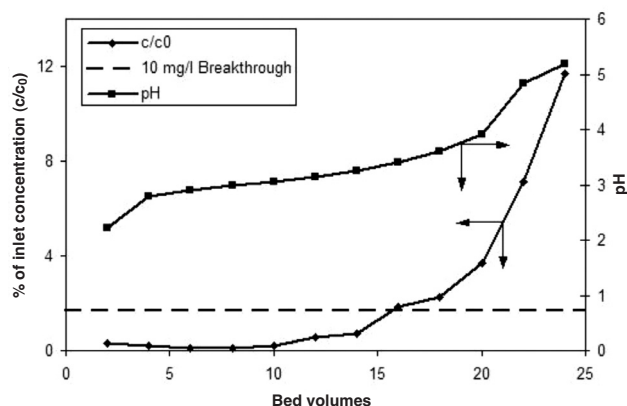


Figure 3—Breakthrough curve for the adsorption cycle of the centre run (55°C, 600 mg/l fluoride)

what were observed in all the runs. The pH dropped from 5.0 at the inlet to ~3.0 for the first few bed volumes processed.

At the beginning of the experiment, the column exiting solution pH was as low as 2.3, which might have been a result of acid solution still present in the bed since the column was acidified before the adsorption step. It appears from the decreasing pH value that the hydrogen ions adsorbed onto the surface were displaced by the base metal species. In Figure 4 the results for the adsorption of fluoride in terms of the fluoride capacity at a breakthrough point of 10 mg/l are presented for changes in initial fluoride concentration and solution temperature. The capacity of the fluoride at the centre run was determined to be 8.65 gF/l activate alumina. This capacity was significantly higher than those reported in literature (Anonymous, 2005; Lounici *et al.*, 1997; Rubel 1979: 1983). The difference can be attributed to the higher fluoride concentrations used in the current study. An examination of Figure 4 reveals that the fluoride adsorption capacity of activated alumina is positively

correlated with both temperature and inlet concentration. The average effect of temperature for changing from its low level (45°C) to its high level (65°C), is calculated (see Equation [2] and [3]) to be an increase of 1.41 gF/l activated alumina while the average effect of initial concentration for changing from its low level (400 mg/l) to its high level (800 mg/L) is determined to be an increase of 0.86 gF/l activate alumina.

Effect of hydrodynamic conditions

The nominal flow rates used in this study were 1 000 l/m².min and 4 500 l/m².min in an 11 mm diameter column loaded with 50 g activated alumina at an initial concentration of 600 mg/l. The breakthrough curves obtained at the two flow rates are shown in Figure 5.

For both curves in Figure 5 it can be seen that the saturation point was not reached. It appears that the system attained a quasi steady state between the 80th and 100th bed volume, which is a symptom of the presence of strong dispersive diffusion resistance. Dispersion resistance increases with an increase in flow rate due to an increase in turbulence in the column. The presence of strong dispersion in the column reduces the efficiency of column operation. This is depicted in Figure 5 by reduced performance at higher flow rate and skewed breakthrough curves for both flow rates. Thus, the results suggest that it is better to operate the column at lower flow rate to maximize the loading of fluoride onto activated alumina.

Desorption: effect of NaOH concentration

Recovery of fluoride is important in hydrometallurgical industry since by doing so savings are made on buying fresh chemicals. Moreover, fluoride-loaded activated alumina needs to be desorbed and regenerated to reduce economic and environmental impacts since activated alumina is a very expensive adsorbent. The desorption of activated alumina-bound fluoride was done using a 0.1 and a 1% sodium

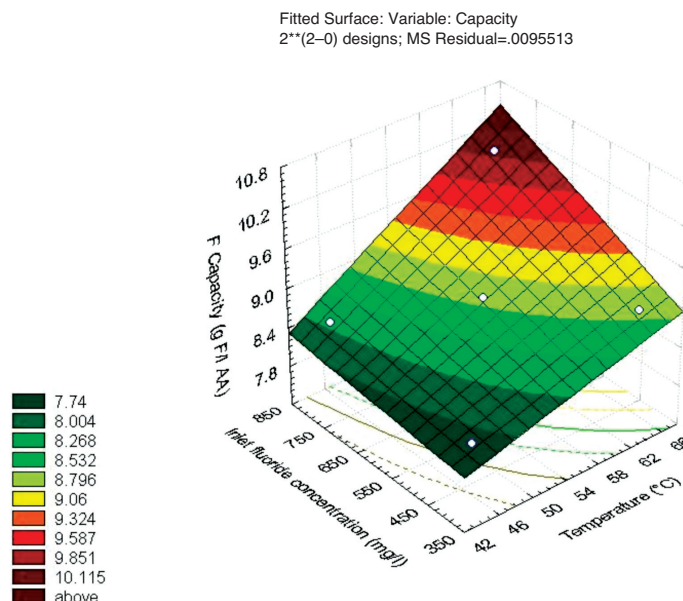


Figure 4—Fluoride capacity of the activated alumina at 10 mg/l breakthrough point

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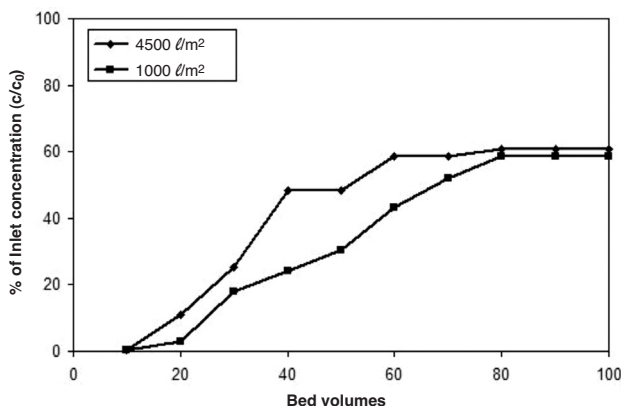
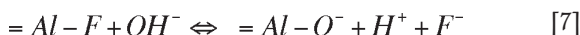


Figure 5—Influence of flow rate on the breakthrough curve

hydroxide solution. A typical elution curve when the desorption agent was 0.1% NaOH is illustrated in Figure 6. A lag period is observed in which negligible fluoride was eluted from the activated alumina bed.

The apparent reason for this lag phase can be seen when inspecting the ionic species (sulphate levels) in the effluent and the pH. It is seen for the initial period that the effluent contained a large number of sulphate ions and the pH was low as a result of the presence of sulphate ions. At low pH, fluoride is tightly bound on the activated alumina active sites. In a similar study, we have shown that fluoride is difficult to desorb because it is tightly chemisorbed on aluminol sites (Onyango *et al.*, 2006a). As the operation was continued, the levels of sulphate ions in the effluent fell to near zero. This phenomenon helped increase the pH of the exiting solution. Apparently, at high pH desorption of fluoride is favoured according to the reaction:



From the practical point of view, the use of 0.1% sodium hydroxide solution is technically not feasible since the volume of hydroxide solution required to desorb the bed would be more than double the volume of solution treated. To this end, it was decided to test a 1% sodium hydroxide solution. The desorption profile obtained for the latter is shown in Figure 7. It is seen that most of the fluoride is desorbed during the first 8 bed volumes processed. The corresponding desorption ratio at this point is 73%. Meanwhile, the trends in the amount of sulphate ions desorbed and the pH value of the column effluent show once more opposite profiles, suggesting as before that sulphate ions were responsible for the low pH in the effluent.

Conclusions

An adsorption-desorption cycle was used to remove and recover unreacted fluoride in a hydrometallurgical process stream. The adsorption medium was activated alumina. From the batch adsorption experiments it was observed that the fluoride was adsorbed optimally when pH values ≤ 8 . For the adsorption cycle in the fixed-bed column, the influence of temperature and inlet fluoride concentration was evaluated. The centre run experiments (initial concentration of 600 mg/l and 55°C) gave a bed capacity of 8.65 gF/l activated

alumina. The capacity of the activated alumina appeared to be positively correlated to both temperature and the inlet concentration. Temperature had a larger effect. The difference in adsorption capacity for a change in the operating temperature from 45°C to 65°C was determined to be 1.41 gF/l activated alumina. This, in part, corroborated the mode of interaction between fluoride and active sites to be that of chemisorption. A desorption step using 1% sodium hydroxide was found to give appreciable performance with a regeneration ratio of 73% achieved after 8 bed volumes were processed. This study gives fundamental information that is useful to the hydrometallurgical industries considering reclaiming back used fluoride. It is suggested that to optimize the regeneration procedure, one should reduce the number of bed volumes eluted from the column.

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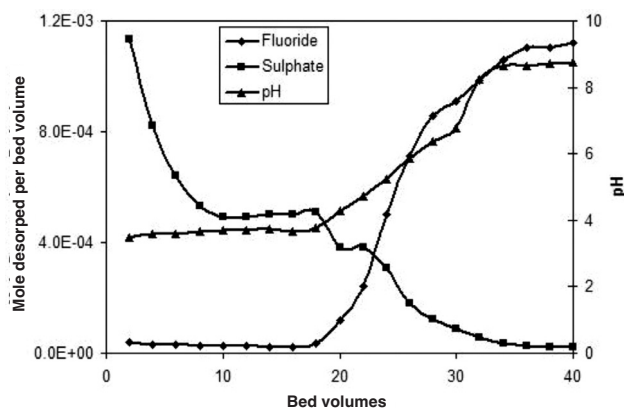


Figure 6—Desorption of fluoride using 0.1% sodium hydroxide solution. Ionic species and pH profiles are also included

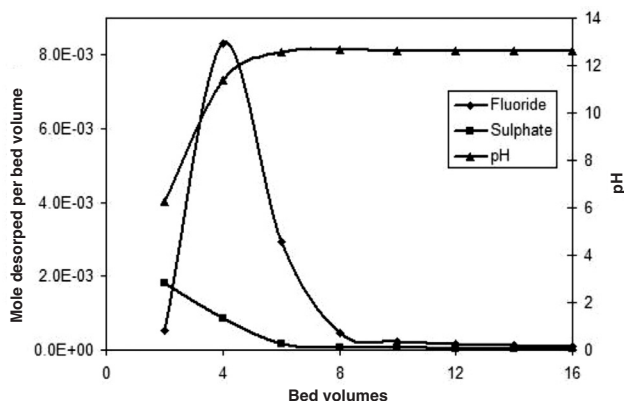


Figure 7—Desorption of fluoride using 1% sodium hydroxide solution. Ionic species and pH profiles are also included

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Flotation '09 programme now available*

It is 103 years since froth flotation was patented, and it is often said to be the 20th century's most important technological development.

It is still the most intensively researched area of mineral processing, as is evidenced by the response to the call for papers for Flotation '09, MEI's 3rd flotation conference, which will be held in Cape Town in November.

Anyone with an interest in flotation should try to be there. So far 10 major sponsors have weighed in with their support and a high-calibre international technical programme for the four days has now been published.

The first two days will be devoted to fundamental physics and chemistry, with Prof. Cyril O'Connor, Chairman of the IMPC, and Dean of Engineering at University of Cape Town, presenting a keynote lecture: The effect of the multiple interactions between reagents and mineral particles on flotation performance – a review.

The final two days will deal with applications and plant practice, with Prof. Graeme Jameson, Laureate Professor at the University of Newcastle, Australia, presenting the keynote lecture: New directions in flotation machine design.

There will also be plenty of time for networking at the beautiful Vineyard Hotel, nestling under Table Mountain, and at the conference dinner at the Spier Wine Estate in Stellenbosch.

Registration forms are now available on the conference website—and it is not too late to submit an abstract.

Full conference details are at www.min-eng.com/flotation09 ♦

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