Production of Aluminum Sulphate from Indigenous Bauxite without Precalcination of the Ore

SHAH ALAM KHAN AND MUMTAZ HUSSAIN
PCSIR Laboratories, Peshawar-25120, Pakistan

(Received 30th July, 1994, revised 3rd April, 1995)

Summary: Reaction parameters for the production of aluminum sulphate (aluminoferric grade) from Khushab bauxite without precalcination of the ore, were studied using a lead-lined reaction vessel. Commercial sulphuric acid was used for the dissolution of alumina (Al_2O_3) present in the ore. Acid concentrations ranged from 30-65 wt.% H_2SO_4 and the acid quantity was of the stoichiometric requirement of bauxite. The reaction temperature was adjusted at 120°C for a time period of 0.5-6.5 hrs. whereas the particle size of the ore was -120 mesh. It has been found that 70% conversion of Al_2O_3 with 97% purity can be achieved after 5 hrs. reaction time at acid concentration of 60 wt. % H_2SO_4. This conversion is associated with iron and free acid as impurities, while the free acid can be neutralized by adding bauxite in excess.

Introduction

Pakistan has large reserves of bauxite or high aluminous clays in salt range and kalachatta range of Khushab and Attock areas of Pakistan, which are scattered over an area of 150 sq. miles of these places [1-4]. At the same time Pakistan imports large quantities of $Al(OH)_3$ and Al_2O_3 for production of aluminum sulphate (commercially known as Filter Alum), which is mainly used for water and sewage purification, for sizing paper in paper industries in pharmaceutical and other allied chemical industries. Annual import of $Al(OH)_3$ and Al_2O_3 during 1991-92 and 1992-93 has been made against a sum of Rs.91.87 million in foreign exchange [5]. This contributes heavy drain on the national wealth.

A number of processes are available for the extraction of alumina from bauxite by sulphuric acid treatment after calcining the ore at higher temperature [6-8]. On the other hand this work is mainly intended to investigate the possibility of producing the aluminoferric type of Filter Alum [9] from indigenous low-iron λ bauxite without pretreatment of the ore, for use in water and sewage purification, and to study the effect of the different factors on the dissolution of alumina in sulphuric acid in order to reduce import of the raw materials and save our foreign exchange which will be a step towards development of indigenous technologies.

Results and Discussion

Effect of initial acid concentration

Fig. 1 and 2 depict the percentage of Al_2O_3 extraction with different acid concentrations of 52.5, 57.0, 60.0 and 65.0 wt. % H_2SO_4 and for different time periods. It is observed that for a certain initial acid concentration, the % conversion increases with the increase of the reaction time. In almost all cases, the rate of increase is higher during the initial stages of the dissolution, then it gradually levels off. The higher conversion was recorded for 60 wt.%H_2SO_4 where the rate of reaction was more than 90% in 3.5 hours, attributed to the fact that using such high acid concentration at the boiling point results in a vigorous reaction which being an exothermic reaction causes local thermal effects in the solid particles. These results are also presented in Table-1.

Table-1: Rate of reaction with respect to time and initial acid concentration expressed as % age reaction.

	Percentage of Al_2O_3 extraction			
Reaction time (hours)	52.5 wt.% H_2SO_4	57.0wt.% H_2SO_4	60.0 wt.% H_2SO_4	65.0 wt.% H_2SO_4
0.5	36.4	46.9	61.4	63.7
1.0	54.6	59.9	69.1	75.2
1.5	62.6	73.2	76.8	78.6
2.0	66.1	78.2	82.8	81.8
2.5	70.0	82.0	87.7	83.2
3.5	75.9	86.7	92.3	85.0
4.5	80.0	88.3	94.5	87.2
5.5	84.2	88.8	95.5	88.3
6.5	88.2	88.9	96.0	89.7

Free acid during reaction

The free acid present at the end of reaction is an important factor which affects the quality of aluminum sulphate. It was carefully monitored and

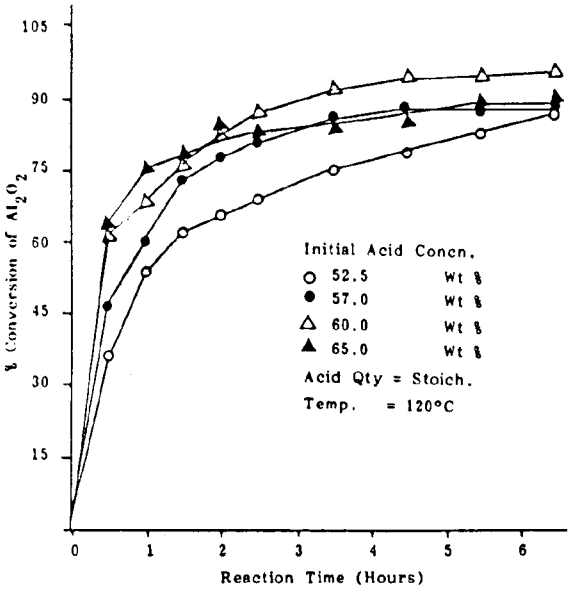

Fig. 1: Effect of the initial acid concentration on conversion of Al_2O_3.

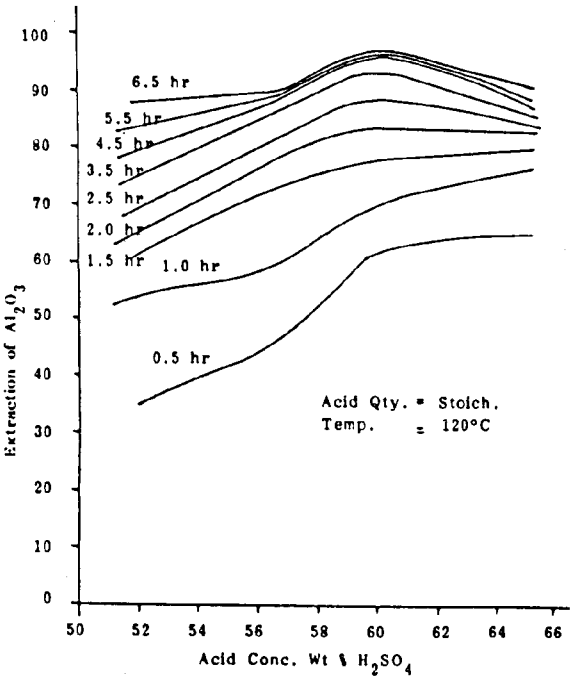

Fig. 2: Effect of the initial acid concentration on conversion of Al_2O_3 for different time periods.

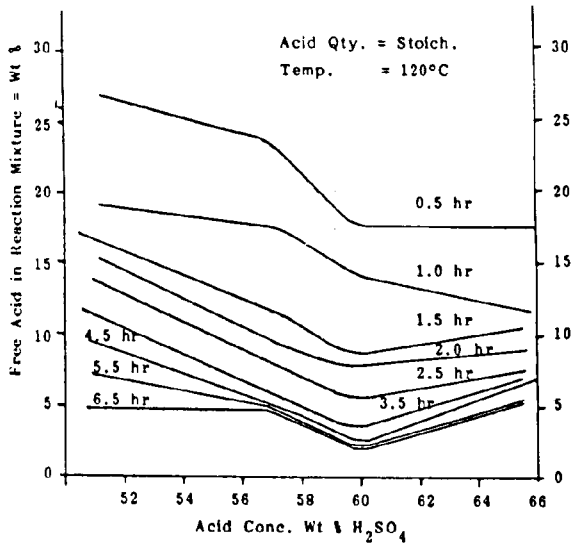

Fig. 3: Free acid during reaction.

the results are expressed in Fig. 3 and Table-2. The results are calculated as wt. % H_2SO_4 for different reaction time and initial acid concentrations. It was found that sulphuric acid having 60 wt. % concentration produces better results where the free acid contents range from 2.0-3.5 wt. % H_2SO_4 between 3.5-6.5 hours reaction time.

Table-2: Free acid during reaction expressed as wt. % H_2SO_4

Reaction time (hours)	Free acid (wt. % H_2SO_4			
	52.5 wt.% H_2SO_4	57.0wt.% H_2SO_4	60.0 wt.% H_2SO_4	65.0 wt.% H_2SO_4
0.0	41.2	43.9	45.7	48.5
0.5	26.3	23.4	17.7	17.7
1.0	18.8	17.7	14.2	12.1
1.5	15.5	11.8	8.8	10.4
2.0	14.0	9.6	7.9	8.8
2.5	12.4	7.9	5.7	7.3
3.5	10.0	5.9	3.5	6.7
4.5	8.3	5.1	2.5	6.2
5.5	6.5	5.0	2.1	5.2
6.5	4.9	4.9	1.8	5.0

Behaviour of foam

The reaction of sulphuric acid and bauxite ore is exothermic as a result of which foam is produced. It plays an important role in studying the behaviour and other physical constants of the reaction. Therefore, a careful examination of foam

formation was studied during the course of reaction. The experimental data are presented in Fig. 4 as foam volume ratio (V_f/V) versus reaction time, where V_f and V are volumes of foam and reaction mixture respectively. From the figure it is evident that the foam formation during the initial reaction time of 1-3 hours is high and later on it decreases. This increase is consistent for various initial acid concentrations studied.

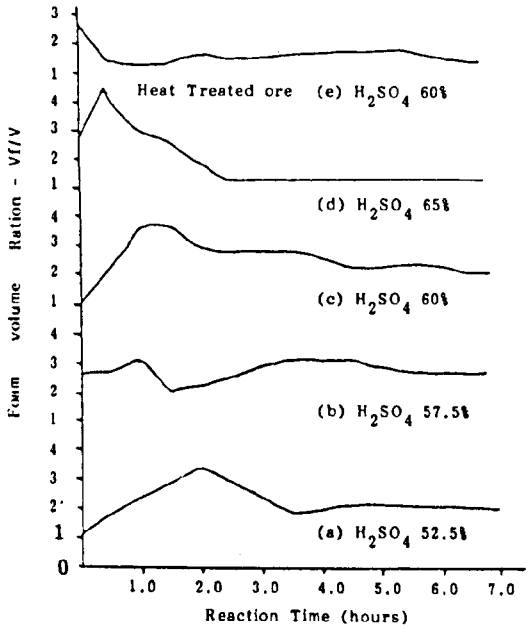

Fig. 4: Behaviour of Foam.

Experimental

Raw material and apparatus

Two types of raw materials were used i.e. sulphuric acid and bauxite ore. Locally manufactured commercial grade sulphuric acid having a strength of 96 wt.% H_2SO_4 was used. Bauxite from the deposits of Tariq Mines of kalachatta/Khushab areas was selected for these studies because of its clayey nature and suitability for the production of chemicals [10]. The chemical and mineralogical composition of the bauxite from Tariq Mines is as follow [1-4]:

Al_2O_3 = 67.0%; Fe_2O_3 = 4.0%; SiO_2 = 10.7%; TiO_2 = 1.4%; CaO = 1.3%; Loss on ignition = 15.2% and Minerals: Boehmite, diaspore, kaolinite, anatase, limonite, calcite, quartz. The reaction vessel used in this investigation consisted mainly of a lead-lined batch reactor with dimension of 15 cm. dia. x 30 cm. and provided with all necessary auxiliaries. This was fabricated in our own workshop.

Procedure

The bauxite ore (ca. 400 gms) was ground to a fine powder (95% passes 120 mesh) and charged into open lead-lined steel reaction vessel followed by calculated amount of water and was stirred to form a uniform slurry. Known volume of sulphuric acid (96 wt. % H_2SO_4) was then added at the rate of 20 ml./min. and the raw materials were thoroughly agitated. The reaction mixture was kept at a temperature of 120°C by the jacket steam for a time period of 0.5-6.5 hrs. A series of clay samples were thus treated with sulphuric acid, of different concentrations of 52.5, 57.0, 60.0 and 65.0 weight percent H_2SO_4 obtained by mixing of concentrated acid with the desired volume of water.

Intermittently the sample of reaction product amounting to 3-5 gms. was collected and was analyzed accordingly so that kinetics of the reaction could be studied. In each experiment, the weight percent of Al_2O_3 dissolution (percentage conversion) was determined by a complexiметric method [11]. Iron and free acid contents were determined by the standard analytical methods [12]. At the end of reaction the product was diluted with calculated amount of water while stirring and heating was continued. The reaction product was finally drawn out of reaction vessel and filtered through filter paper. The clear solution was concentrated to the desired density, which was later on solidified.

Conclusion

Production of aluminum sulphate (alumino-ferric grade) from Khushab bauxite ore without precalcination, is practically possible when reacted with sulphuric acid. The percentage recovery of the product increases with increase in the reaction time, for a certain acid concentration. The rate of this increase is high during the initial stages of dissolution. Alumina dissolution is also associated with iron and free acids as impurities. However, the free acid content can be neutralized with excess bauxite.

The maximum yield of aluminium sulphate is attained when the dissolution was carried out at the boiling temperature of the slurry, using dilute or moderate concentration of the acid.

References

1. M.R. Cheema, Geol. Surv. Pak., Inf. Release No. 76 (1973).
2. T. Hussain and A.A. Naqvi, Geol. Surv. Pak., Inf. Release No. 59 (1973).
3. S.M.I. Shah, *Geol. Surv. Pak. Records*, **52**, 91 (1980).
4. Persons/RUJV, Feasibility Study of Bauxite-Preleminary Survey Report under Technical Services Project U.S. Aid Loan No. 391-W-165, PUNJMIN (1981).
5. Foreign Trade, Statistics Division, Govt. of Pakistan (1992, 1993) Vols. 18-20.
6. Ya. Ya. Dodonov, G.V. Medoks and E.M. Soshestvenskaya, *J. App. Chem., (USSR)* **20**, 870 (1947).
7. L. Dubnil, *Verre Silicate Ind.*, **12**, 22 (1947).
8. F.H. Aly, I.El. Shawarby, S.H. Eissa and M.F. Chalabi, *J. Min. Met. Fuel.*, **29**, 316 (1981).
9. The Wealth of India, Industrial Products, Part-1, p. 83 (1948).
10. J. Benbow, Industrial Minerals, p. 67 (1988).
11. E. Shura, Methods of Analysis of Silicate Rocks, Series No. 104, Complexiметric Methods, Moscow (1965).
12. W.W. Scott, Standard Methods of Chemical Analysis, N.Y. (1956).