

## Recovery of Alumina from Khushab Bauxite by Leaching with Sulphuric Acid and Removal of Iron Impurity by Ethanol

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**Summary:** Bauxite is heterogeneous material principally composed of aluminum oxide minerals and found in all continents. It is being used in chemical, cement, refractory, abrasive, fertilizer, steel and other industries. In order to extract the alumina, the calcined samples of bauxite of Khushab area were ground to -710  $\mu\text{m}$ . Sulphuric acid of purity 40% was used as leaching agent and slurry of pulp density 14% was prepared by dissolving 60 ml acid in 20 gm sample. The leaching was carried out at 90°C for 2 hours. The iron impurity was removed by ethanol of purity 68%. The drying, dehydration and desulphurization temperatures were kept 105°C, 450°C and 850°C respectively in all the stages of the process. Alumina recoveries from four samples of Sultan Mehdhi, Chamil More, Niaz Mine and Nadi locations were 20.8%, 9.81%, 15.47% and 7.78% respectively. Iron was almost completely removed as the analysis shows that the  $\text{Fe}_2\text{O}_3$  removal was from 97.8% to 99.6%. It is concluded that leaching efficiency was quite encouraging except Nadi ore sample. However the iron free alumina recoveries were low as the analysis of  $\text{Fe}_2\text{O}_3$  processed residue shows that it contains 72.72% to 92.94% of leached alumina in all the four experiments.

Key Words: Bauxite, Ethanol, Iron, Leaching, Khushab, Recovery, Texture.

### Introduction

Deposits of bauxite are reported in all the continents [1]. Asian reserves are large and are mostly in China, Malaysia, India, Indonesia, Pakistan and Sarawak. Deposits are also occurred in Iran and Turkey [2]. Bauxite is found in wide variety of rocks including syenite, basalt, limestone and schist [1]. It is formed by weathering of the alumina bearing rocks under tropical or semi-tropical climates with high permeability [3]. The common impurities in bauxite are hematite, goethite, titania, lipidocrosite, kaolinite, pyrophyllite, illite, quartz, sillimanite, corundum, rutile, garnet vanadium, etc. [1, 4-6]. It has found applications in refractory, cement, chemical, abrasion, etc. [4]. Monocrystalline alumina is used for tooth implants and ball and socket joints of artificial hip and knee joints [7]. Bauxite deposits are present at various locations of Khushab and Sarghodha districts of Pakistan. Among these deposits 0.3 million tons of good grade bauxite ore containing  $\text{Al}_2\text{O}_3=40\%$  minimum and  $\text{SiO}_2=15\%$  maximum. Approximately 190 million tons low grade bauxite ore is also present in same area [6]. The present experimental work was of preliminary type and was planned to investigate the recovery of alumina from highly salacious bauxite ore by using

sulfuric acid. So that large deposits of low grade bauxite present at different locations of Pakistan may be exploited. Almost all commercial bauxite has Alkali Feldspar Charnockite (AFCh) content 50-55% [8]. X-Ray diffraction results show that all the four samples of bauxite of Khushab area are boehmite with kaolinite (clay) and quartz as main impurities. Furthermore Opaque minerals of iron and titanium are present [6], the average chemical composition of the Khushab area of different pockets comes to 47.39%  $\text{Al}_2\text{O}_3$ , 24.78%  $\text{SiO}_2$  and 6.77%  $\text{Fe}_2\text{O}_2$  [9]. The chemical analysis of Mehdhi, Chamil More, Niaz Mine and Nadi of Khushab bauxite shows that low grade ore contains  $\text{Al}_2\text{O}_3$  37.17-45.01%; other minor minerals  $\text{Fe}_2\text{O}_3$  0.15 2.14% and  $\text{TiO}_2$  0.59-1.57%; impurities  $\text{CaO} + \text{MgO}$  0.96 1.78%

The bauxite which contains high percentage of silica is of whitish color and other is of reddish which contains higher amount of Fe and  $\text{TiO}_2$  which could be exploited in several ways [10]. For metal grade bauxite, the aluminum content should be more than 40% and for chemical grade it may above 60%, while most Bayer process plants use bauxite having alumina content greater than 40%, however all the

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technical factors from mining to marketing are to be considered [8]. The silica contents of the soil of Khushab area were estimated between 5.2 to 29.35% [5] and the samples of Sultan Mehdhi, Chamil More, Niaz Mine and Nadi contains 36.5 to 44.41% silica [6], So these samples may be characterized as high silica content with low mass ratio of  $\text{Al}_2\text{O}_3$  to  $\text{SiO}_2$  (A/S). It is necessary to improve A/S value to over 10 before bauxite is economically treated by the Bayer process [11-12].

Sulphuric acid dissolve easily alumina in ore samples as aluminum sulphate, which is converted to alumina crystals on calcinations. The use of  $\text{H}_2\text{SO}_4$  can be cost effective if acid is obtained from local fertilizer factories [6]. The calcination temperature range was found between  $450^\circ\text{C}$  to  $900^\circ\text{C}$  and different concentrations of sulfuric acid with leaching Temperature range  $60^\circ\text{C}$  to  $100^\circ\text{C}$  have been reported for the extraction of alumina from calcined clays [13-16], The reaction /leaching time was reported over a range from 15 minutes to 6 hours [17]. The quantity of ethanol used to remove the iron impurity from aluminum sulphate depends upon the quantity of iron contents. Different methods may be applied to remove the iron impurity. These include:

- i. Iron removal by adding ethanol ( $\text{C}_2\text{H}_5\text{-OH}$ ).
- ii. Electrolyte removal method.
- iii. Removal of iron by chemical process [18].

## Results and Discussion

The leaching slurry became dense as a result poor filtration rate was observed during the experimental work.

Table-1 shows that extracted alumina content of ore are from 7.78% to 20.80%, which are quite low. The maximum  $\text{Al}_2\text{O}_3$  recovered is 20.8% from Sultan Mehdhi which is also low. The lowest recovery 7.78% was found from Nadi ore samples although Nadi ore contains greater amount of alumina 45.09%. The Chamil More and Niaz mine samples have 9.81% and 15.47% recoveries. The lower recovery from Nadi sample shows that incomplete leaching has been resulted under the conditions *i.e.*  $\text{H}_2\text{SO}_4$ ,  $t = 2$  h and quantity of acid 60 mL.

Weight of solid residue of Sultan Mehdhi, Chamil More, Niaz Mine and Nadi were 17.15 g, 17.85 g, 18.00 g and 18.20 g, while the weights of filtrate were 88 g, 89 g, 82 g and 90 g respectively.

The loss of material *i.e.* the Material Unaccounted for (MUF) was in minute quantities in each of the experiment and is in a close range of 0.67% to 1.96%

### Problems of Iron Removal

The weight of iron in each of 20 g sample was 0.046 g, 0.428 g, 0.03 g and 0.6 g respectively. The ethanol treated residue of the four samples of Sultan Mehdhi, Chamil More, Niaz Mine and Nadi were 1.10 g, 1.25 g, 0.85 g and 0.9 g respectively.

These were obviously greater than the weight of the iron contents present in the samples. So the samples of ethanol treated residue were analyzed for  $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ . The results are presented in Table-2. The analysis shows that ethanol treated residue contains  $\text{Fe}_2\text{O}_3$  3.49% to 6.64% corresponding to iron removal 98.5%, 97.8%, 99.1% and 99.6% respectively. The total iron removal contents are in the range of 97.8% - 99.6%, which means that iron impurity has almost been removed but at the cost of  $\text{Al}_2\text{O}_3$  as the analysis of  $\text{Fe}_2\text{O}_3$  processed residue shows that it contains 72.72% to 92.94% of leached alumina in all the four experiments. As a result the recovery of iron free alumina was low. Moreover ethanol is costly and may make the process uneconomical. Its availability is also a problem.

The results of Table-1 and 2 show that if the contents of alumina were combined then the recovery was increased from 20.8%, 9.81%, 15.47% and 7.78% to 32.91%, 22.24%, 25.42% and 16.89% for Sultan Mehdhi, Chamil More, Niaz mine, and Nadi samples respectively

## Experimental

### Materials and Instruments

The calcined samples of Sultan Mehdhi, Chamil More, Niaz Mine and Nadi of Khushab area were used in this experiment work. Sulphuric acid of make Sigma Aldrich, Germany available in 2.5L packing and ethanol of make Fluka, Spain available in 2.5L packing were used. Both the chemicals used were of analytical grade. The samples were ground in ball mill of make Retsch Germany. The ground samples were sieved by Taylor series mesh. The drinking water available in laboratory was used. Universal oven Model No. 100-800, Memmert Germany was used for drying purpose. The Filter papers are of Whatman No. 42 types.

Table-1: Weights and Percentages at different stages of the process for recovery of alumina by acid leaching and removal of iron impurities by ethanol of Sultan Mehdi, Chamil More, Niaz and Nadi Mines samples.

Sr.#	Sample Description	Al <sub>2</sub> O <sub>3</sub> Content (%)	Sample Weight (g)	Weight of Filtrate (g)	Residue		Al <sub>2</sub> O <sub>3</sub> extracted (g)	Al <sub>2</sub> O <sub>3</sub> Recovery (%)	Material Unaccounted for (MUF)	
					Leaching Residue (g)	Ethanol Treated Residue (g)			Weight (g)	Weight (%)
1	Sultan Mehdi	39.5	20	88	17.15	1.10	1.65	20.80	0.10	1.27
2	Chamil More	38.23	20	89	17.85	1.25	0.75	9.81	0.15	1.96
3	Niaz	37.17	20	82	18.00	0.85	1.10	15.47	0.05	0.67
4	Nadi	45.01	20	90	18.20	0.90	0.70	7.78	0.10	1.11

Table-2: Percentage of Alumina and Iron in the Ethanol treated residue of Sultan Mehdi, Chamil More, Niaz and Nadi Mines samples.

Sr. No	Sample Description	Iron Contents (%)	Weight of Ethanol Treated Residue (g)	Fe <sub>2</sub> O <sub>3</sub> in Ethanol Treated Residue (%)	Total Fe <sub>2</sub> O <sub>3</sub> Removed (%)	Al <sub>2</sub> O <sub>3</sub> in Ethanol Treated Residue (%)	Others (%)
1	Sultan Mehdi	0.23	1.10	4.09	98.50	86.36	9.09
2	Chamil More	2.14	1.25	33.52	97.80	72.72	2.82
3	Niaz	0.15	0.85	3.49	99.10	92.94	3.56
4	Nadi	0.30	0.90	6.64	99.60	90.56	2.80

### Method

On the basis of reported acid leaching parameters, the flow sheet of sulphuric acid leaching with Khushab bauxite ore and removal of iron impurity by ethanol was planned and executed as shown in Fig. 1

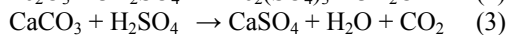
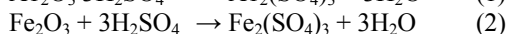
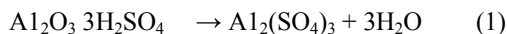
### Sample Preparation

The calcined samples were ground in ball mill. These were then sieved through Taylor series to get the size -710µm

### Leaching of Ore Samples

The samples were leached with 40% sulphuric acid at 90°C and at atmospheric pressure for 2 hours. Slurry of pulp density of 14% was prepared with ore, water and sulphuric acid at ratio 1:3:3 (w/w). Alumina dissolved in acid forming aluminum sulphate.

Depending on the type and nature of the solid material as well as the reaction conditions during the leaching process of bauxite ore with sulphuric acid, the following reactions may occur [19].



### Filtration and Evaporation

In order to remove the clay residue, the leached slurry was cooled down to room temperature and then filtered. The clay residue was dried and dehydrated for one hour at 105°C and 450°C respectively and weighed. Filtrate contains aluminum

sulphate Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and minor iron as impurity was then concentrated by evaporation on Bunsen burner prior to addition of ethanol. Concentrate was obtained in 2 hours. Vigorous effervescence was observed during evaporation process. Also obnoxious fumes of some stack gasses being escaped were plentiful causing nuisance.

### Iron Removal from Pregnant Solution

The contents of the iron were in minor amounts in the samples *i.e.* 0.15% -2.3%. So, ethanol was used to remove iron impurities from concentrate *i.e.* Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>. The ratio of ethanol to concentrate was 3:1 (w/w). The stirring time for mixing the ethanol was 5 minutes. Solution was once again filtered. Iron and ethanol were obtained in the filtrate, while iron free aluminum sulphate was remained on the filter paper.

### Evaporation, Drying, Dehydration and Desulphurization

In order to remove the sulphate, aluminum sulphate was first dried at 105°C and then dehydrated at 450°C for one hour to remove any water content and moisture. Finally it was calcined at 850°C for one hour. As a result sulphates were removed in the form of SO<sub>2</sub> and SO<sub>3</sub> as stack gasses. The iron free white powder of alumina was then weighed.

The ethanol treated filtrate residue contain iron impurity was first concentrated by evaporation on water bath to remove the ethanol and then dried, dehydrated and desulphurized at 105°C, 450°C and 850°C respectively for one hour to remove the sulphates in the form of SO<sub>2</sub> and SO<sub>3</sub> as stack gasses. The iron free white powder of alumina was then weighed.

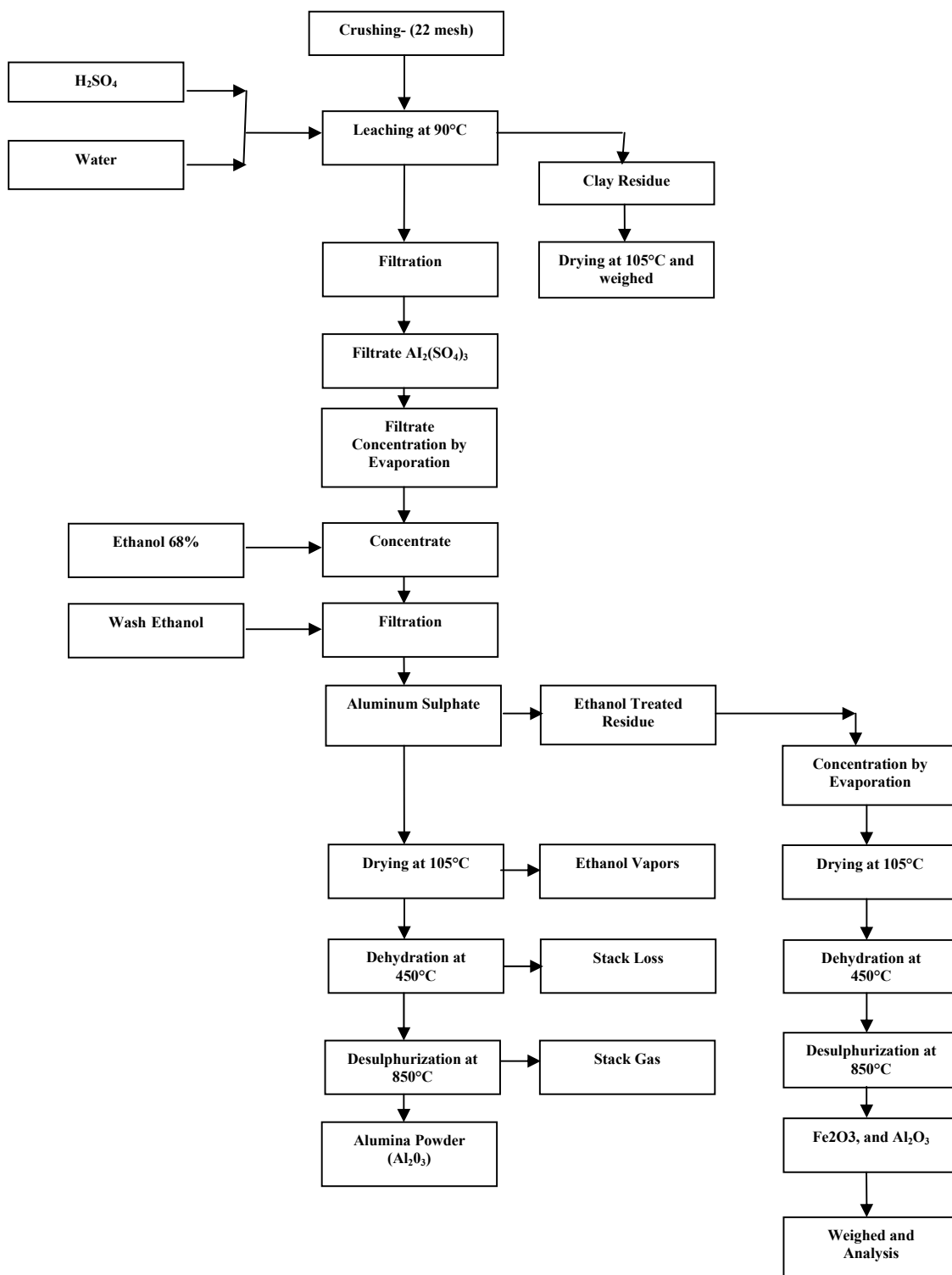


Fig. 1: Flow sheet for the Leaching of Khushab Bauxite Ore with H<sub>2</sub>SO<sub>4</sub> and Removal of Iron Impurity by Ethanol.

*Analysis for Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>*

A 0.8 g powdered sample was digested with 10 mL HF and 5 ml HClO<sub>4</sub> and heated to dryness, the residue was treated twice with 10 ml HF and final volume of 100mL was made in HCl medium. This extract was used for the determination of Al. A 0.4 g of powdered ethanol treated and processed residue sample was extracted with 10 mL of aqua regia and boiled for 3 hours under reflux. After cooling the sample digest was filtered through filter paper and filtrate was transferred into a 100 mL volumetric flask and made volume up to the mark with doubly distilled deionized water (DDDW). This was used for the determination of Fe by using atomic absorption spectrophotometry. For this purpose A Analyst 700 Perkin Elmer spectrophotometer was used in flame mode with air acetylene as flame fuel.

**Conclusions**

The Bayer process cannot be applied to bauxite samples under study because of high contents of silica *i.e.* 36.16- 44%. Therefore Sulphuric Acid process was considered for the experimental investigation as H<sub>2</sub>SO<sub>4</sub> easily dissolve alumina.

The overall recovery of Al<sub>2</sub>O<sub>3</sub> is not very encouraging. Though Sultan Mehdi show comparatively a higher Al<sub>2</sub>O<sub>3</sub> recovery range *i.e.* 20.8 %. The Niaz sample however shows medium range recovery of alumina among the four samples. Chamil More and Nadi show similar response with alumina recoveries 9.81% and 7.78 % respectively. The lower recoveries may be due to mineralogy, texture and hardness of ore or the high contents of silica present in the ore samples that obstructed the leaching of alumina. This needs further research that will make the acid process applicable to leaching most of the ore.

After treated with ethanol the iron was almost completely removed from all the samples and no effervescence was observed during evaporation, drying, dehydration and desulphurization. Since ethanol treated residue contains high amounts of alumina 72.72% to 92.94%. So it may be concluded that the leaching efficiency of bauxite ore samples except Nadi ore was quite reasonable although the recovery of iron free alumina is low. Furthermore iron removal by ethanol was not proved technically sound method when the iron impurities in bauxite are very low.

The material balance shows that only a small fraction is Material Unaccounted for (MUF) *i.e.* varies from 0.67% to 1.96 % in all four experiments. The poor filtration rate of aluminum sulphate means that slurry was thickened during 2 hour leaching, so the quantities of acid and water selected were inefficient and needs to be optimized. Ethanol can be recovered by condensation for reuse to make the process economical. It was not tried to recover the ethanol in the present experimental studies because the main objective of the study was to recover the alumina.

**Recommendations**

- i. The silica content in the ore obstructing the leaching reaction and also poor filtration rate suggest that mineral dressing method like Dense Media Separation (DMS) or reverse froth floatation to float silica may be investigated to remove silica prior to leaching with H<sub>2</sub>SO<sub>4</sub> to get improved results.
- ii. As the laboratory investigations are preliminary, further investigations by varying time, temperature concentration and quantity of sulfuric acid, quantity of water added and particle size of ore may be carried out to optimize the leaching parameter for maximum recovery of alumina.
- iii. Since iron impurities are small and the ethanol treated residue is almost equal in weight to that of weight of Al<sub>2</sub>O<sub>3</sub> extracted, therefore it is recommended that ethanol may be used only in a small amount, *i.e.* 1 mL for 20 gm samples. Or other iron removal methods like use of HCl etc. may be investigated.

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