The production of high purity alumina from solid wastes obtained from aluminium factories

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Abstract

This work is directed to the production of alumina (Al_2O_3), from the solid waste cake produced as a by-product from some aluminium small factories. Aluminium ions in this cake are separated as sodium aluminate solution ($NaAlO_2$), as a result of treating the solid cake with commercial caustic soda solution. Aluminium hydroxide is precipitated from $NaAlO_2$ solution by purging CO_2 gas through the hot solution. The resulting precipitate is separated by filtration, washed with hot water, dried at 105°C, then calcined at 600 or 1000°C to produce pure alumina of γ- or α-form, respectively.
Keywords: alumina, sodium aluminates, aluminum hydroxide, leaching, solid waste.

1 Introduction

Small factories producing aluminum utensils, after shaping, use sodium hydroxide baths for glazing the surface of the product. The spent bath always contains a solid waste cake which contains more than 80% of aluminum compounds mainly as hydroxides and aluminates. This solid waste is generally dried by solar energy and disposed in landfill with remarkable amounts which represents an environmental problem. This solid waste can be recycled and aluminum compounds in pure state can be recovered after a leaching process. These aluminum compounds are of high economic value and have wide applications.

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The aim of the present study is to recover aluminium ions from the solid waste produced from some Egyptian small factories producing aluminum utensils. Production of alumina (Al_2O_3) is the main goal of such study.

Recovery of aluminum from both liquid and solid wastes has been reported by different authors. Rayzman *et al.* [1] produced sodium aluminate solution from alumina-bearing intermediates and wastes including spent potliner and salt cake resulting from aluminium-dross recycling. Pradhan *et al.* [2] developed a process for production of superfine white aluminium trihydroxide powders from actual plant Bayer liquor. Barakat *et al.* [3] removed aluminum and regenerated caustic soda from the spent washing liquor of aluminium etching using lime neutralization processes. Gürel *et al.* [4] produced fine reactive alumina powder using Bayer gibbsite as a starting material.

Alumina is a strong adsorber and can function as an amphoteric ion exchanger depending on the nature of the surface and the solvent [5]. It is also used alone or impregnated with promotes as a catalyst. By changing variables such as the degree of crystallinity, the purity, the activation temperature and the rate of heating, a wide variety of materials can be made to meet special needs [6]. Ceramic industry processes alumina by grinding, adding other materials to form vitreous bonds, forming by any of several processes and firing [7]. Alumina is also used in electronic circuits or can be fused to form blocks used in lining glass furnaces [8]. It can be also used as an adsorbent for purification or treatment of radioactive and industrial waste solutions.

2 Experimental materials, methods and analysis

2.1 Materials

Samples from the solid waste obtained as a by-product from some factories that produce kitchen utensils were collected for characterization and for the leaching process. Sodium hydroxide of commercial grade and tap water were used in the leaching process.

2.2 Characterization of the solid waste

Solid sample is a brownish white powder. When fractionized by sieving it showed that it contains 84% by weight of particle size between 53 to 75 μm, while it contains 16% of particle size between 150 to 300 μm. Analysis of the solid waste under investigation using the XRF is given in table 1.

2.3 Leaching procedure

Different concentrations of sodium hydroxide solutions were tested for leaching the solid waste containing aluminum species (10, 20, 30 and 40%).Different solid to solution ratios (1:1, 1:2, 1:3 and 1:4) were also tested for the leaching process. The following is the standard procedure, unless otherwise stated:

A 100 g representative sample of the solid waste was added into a 1L capacity of Pyrex beaker. Sodium hydroxide solution was transferred to the beaker. The resulting slurry was heated for different defined periods, and a magnetic rod was used for stirring. The liquor obtained, which is mainly sodium aluminate was then filtered.

2.4 Precipitation procedure

Aluminum ions leached in the hot filtrate was precipitated by bubbling CO_2 gas (7L/min) for certain time through the solution till complete precipitation (pH 7). The precipitate formed, $Al(OH)_3$ was then separated by filtration through Whatman filter paper No. I. The precipitate was washed with tap water and dried in an oven at 100°C till constant weight to remove free water. The dried precipitate was subjected to calcination at 600℃ and 1000℃ for 5 hours to produce Al_2O_3 (alumina) in γ- or α-forms, respectively .

Table 1: Characterization of the solid waste.

Elements	Concentration (%)
Al	90.27
Mg	0.92
Si	0.39
Ca	0.41
Fe	0.26
Na	2.71
Ti	0.014
K	0.026
L.O.I	2.92

2.5 Analysis and measurements

Solid samples were analyzed using XRF, DTA of type DTA/DSC, the crystalline phase and microstructure were characterized using SEM, and XRD of type Bruker Axs-D8 Advance. The particle size distribution was determined by a laser scattering technique (Quanta chrome NOVA Automated Gas Sorption System) and analytical Sieve (Fritsch). The optical properties were determined using the whiteness instrument.

3 Results and discussion

3.1 Leaching process

To recover aluminum ions in solution from the solid waste, a leaching process was carried out using NaOH solution. The different parameters affecting the leaching process were studied.

3.1.1 Effect of NaOH concentration

The effect of NaOH concentration on leaching 100 g of the solid waste was studied with four different concentrations (10%, 20%, 30%, and 40%), while the leaching time is kept constant (4 hours) for (1:2) solid to liquid ratio. It was found that as NaOH concentration increases the amount of dissolved waste increases as shown in table 2.

3.1.2 Effect of solid to solution ratio

The effect of solid to solution ratio on leaching of 100 g solid waste was studied with four different ratios (1:1, 1:2, 1:3, and 1:4). The leaching time is kept constant (4 hours) and the concentration of NaOH was 30%. It was found that the amount of dissolved waste increases with increase in the ratios shown in table 3.

Table 2: Effect of NaOH concentration.

NaOH concentration	Amount of sludge remained(g)	leaching %
10%	80.4 g	19.6%
20%	42.0 g	58.0%
30%	12.0 g	88.0%
40%	10.0 g	90.0%

Table 3: Effect of solid to solution ratio.

Solid solution ratio	Amount of sludge remained(g)	leaching %
1:1	16.2 g	83.8%
1:2	12.0 g	88.0%
1:3	10.7 g	89.3%
1:4	9.0 g	91.0%

3.1.3 Effect of time

The effect of time on leaching the solid waste was studied with five different time intervals (1, 2, 3, 4 and 6 hours), while the NaOH concentration is kept constant at 30% for 1:2 solid to liquid ratio. It is clear that as leaching time increases the amount of dissolved waste increases as shown in table 4.

Table 4: Effect of time.

Time	Amount of sludge remained(g)	leaching %
1h.	25.0 g	75.0%
2h.	17.1 g	86.9%
3h.	15.7 g	87.3%
4h.	22.0 g	88.0%
6h.	10.1 g	89.9%

3.1.4 Effect of temperature

The effect of solution temperature on leaching the solid waste was studied with four different temperatures (70°C, 80°C, 90°C and 100°C), while the leaching time is kept constant (4 hours) for (1:2) solid to liquid ratio and for 30% NaOH concentration. By increasing the temperature the amount of dissolved waste increases as shown in table 5.

Table 5: Effect of temperature.

Temperature	Amount of sludge remained(g)	leaching %
70°C	27.2 g	72.8%
80°C	17.0 g	83.0%
90°C	15.0 g	85.0%
100oC	12.0 g	88.0%

It is clear from the obtained results in Tables 2–5 that increasing the concentration of NaOH solution from 30% to 40% shows no significant increase in the leaching process (Table 2), therefore, for economical purposes, 30% of the NaOH solution was found enough for the leaching process. For the same purpose, the solid to solution ratio of 1:4 was found to be suitable (Table 3). In addition, to avoid time consumption, leaching time for 2 hours was found enough for the leaching process at 100°C (Tables 4, 5).

3.2 Precipitation process

Aluminum ions leached in the alkaline solution namely as sodium aluminate (pH 14) were precipitated as aluminum hydroxide by lowering the pH of the leached solution to 7. This was done by bubbling CO_2 gas (7L/min) for a certain time through the hot solution till complete precipitation; the precipitate was washed by hot water to dissolve any precipitated Na_2CO_3.

3.3 Calcination of alumina

The produced precipitate of aluminum hydroxide was first dried at100°C in an oven to eliminate free water. The dried product was calcined at 600°C or 1000°C for 5 hours to produce Al_2O_3 (alumina) in γ- or α- forms, respectively. γ- form of alumina is used as an adsorbent for separation processes.

3.4 Characterization of alumina

Table 6 represents the data obtained from the different analyses on the produced alumina. The peaks of the XRD patterns for the recovered alumina coincided with that of (semi-crystalline) pattern of γ- alumina as shown in Fig. 2. The thermal behavior of γ- alumina was investigated (Fig. 3), in the DTA curve, three endothermic peaks were observed at temperatures in the range 100 –300°C were considered to be due to dehydration [9].

The specific surface area (S_{BET}) of γ- alumina is 140 m^2/g, which is relatively high compared to those reported [9].

Table 6: Characterization of alumina.

Aluminium oxide	
Names	Anhydrous aluminium oxide, γ- alumina
Chemical formula	Al_2O_3
Chemical composition (XRF)	Al_2O_3 – 97.8% MgO – 0.18 SiO_2 – 0.57 CaO – 0.028 Fe_2O_3 – 0.035 TiO_2 – 0.011 L.O.I – 0.67
Physical properties	
Apparent Density	0.3 g/cm^3
Melting point	2015 – 2072^0C
Maximum temperature of use	1600^0C
Chemical properties	
Moisture content	4 – 5%
pH of water suspension	8 – 10
Optical properties	
Color	White
Whiteness	98.01%
Brightness	93.83%
Iso-brightness	96.07%
Morphology	
Particle shape by(SEM)	Irregular (as shown in fig. 1)
Pore diameter	1.947 E + 01 A^0
Pore volume	0.1280 cc/g

Table 6: Continued.

Morphology	
Sieve analysis	28.00% on 850 μm sieve 11.40% on 600 μm sieve 11.56% on 280 μm sieve 10.29% on 420 μm sieve 12.91% on 212 μm sieve 14.14% on 180 μm sieve 9.06% on 152 μm sieve 8.98% on 125 μm sieve 2.73% on 106 μm sieve 0.75% on 63 μm sieve 0.15% on 45 μm sieve 0.18% on < 45 μm sieve
Specific surface area	139.07 ± 1.944 m^2/g

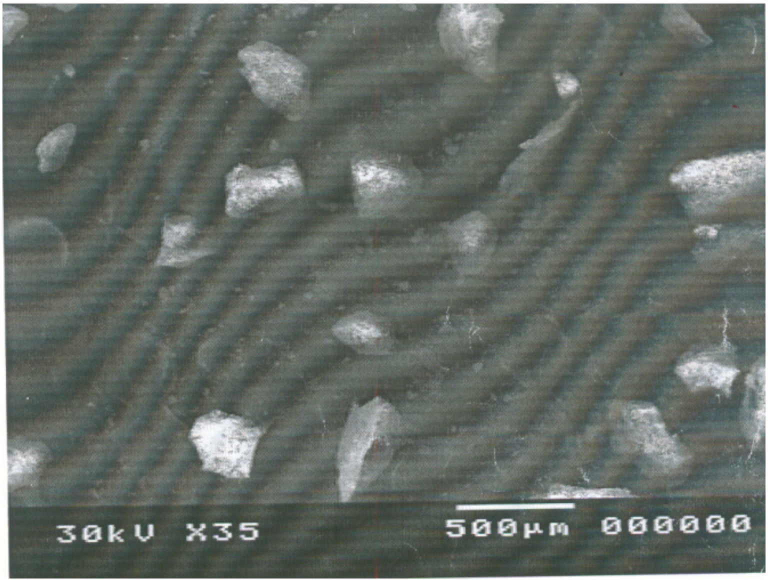

Figure 1: SEM micrograph for alumina particles.

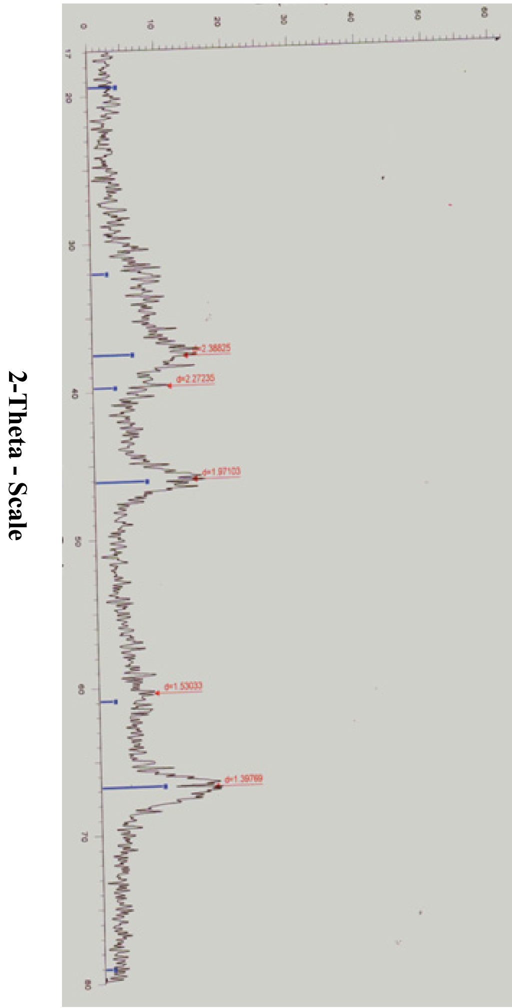

Figure 2: XRD pattern for alumina.

IR patterns for both the solid waste and the recovered alumina are given in Fig. 4 (1) and (2), there are nine IR peaks in the waste (559.1, 800.0, 1028.1, 1419.5, 1559.5, 1653.9, 2016.5, 2360.8, and 3526.1 cm^{-1}) due to organic substances from the lubricants. But for γ-alumina the pattern shows only one peak (746.5 cm^{-1}) due to Al–O bond as in Fig. 4.

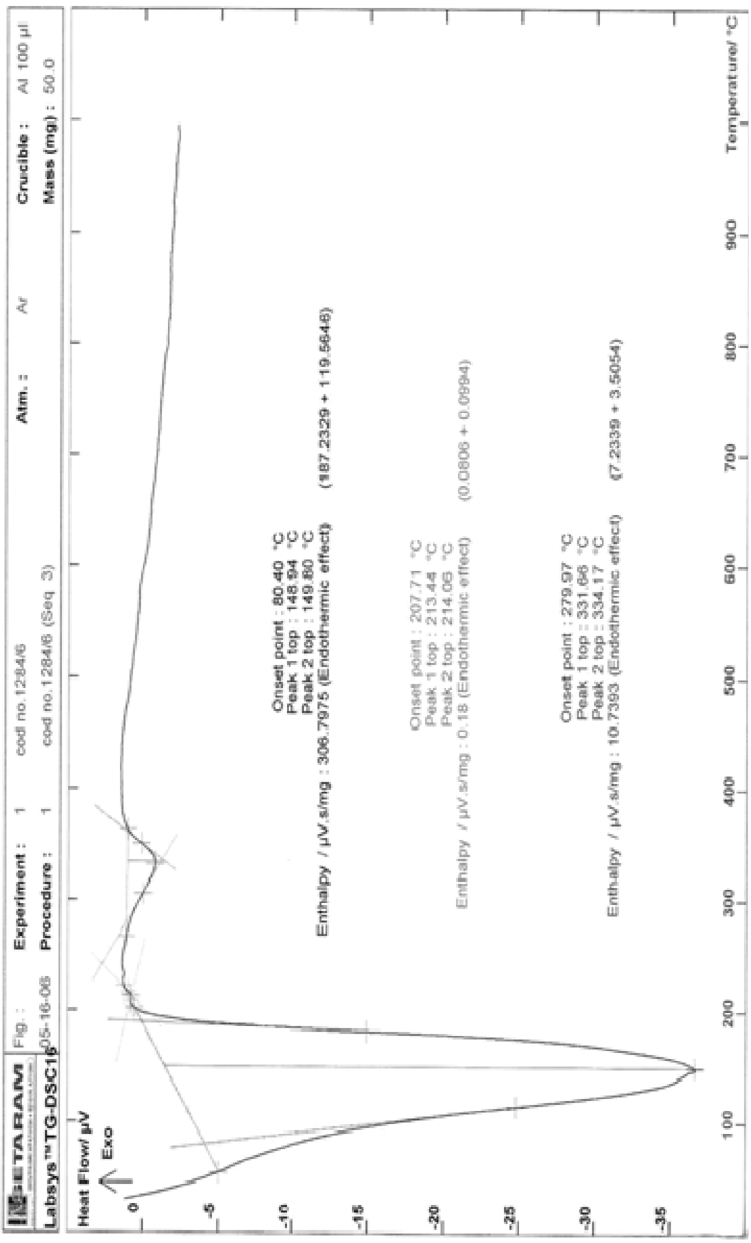

Figure 3: TG/DTA curves for alumina.

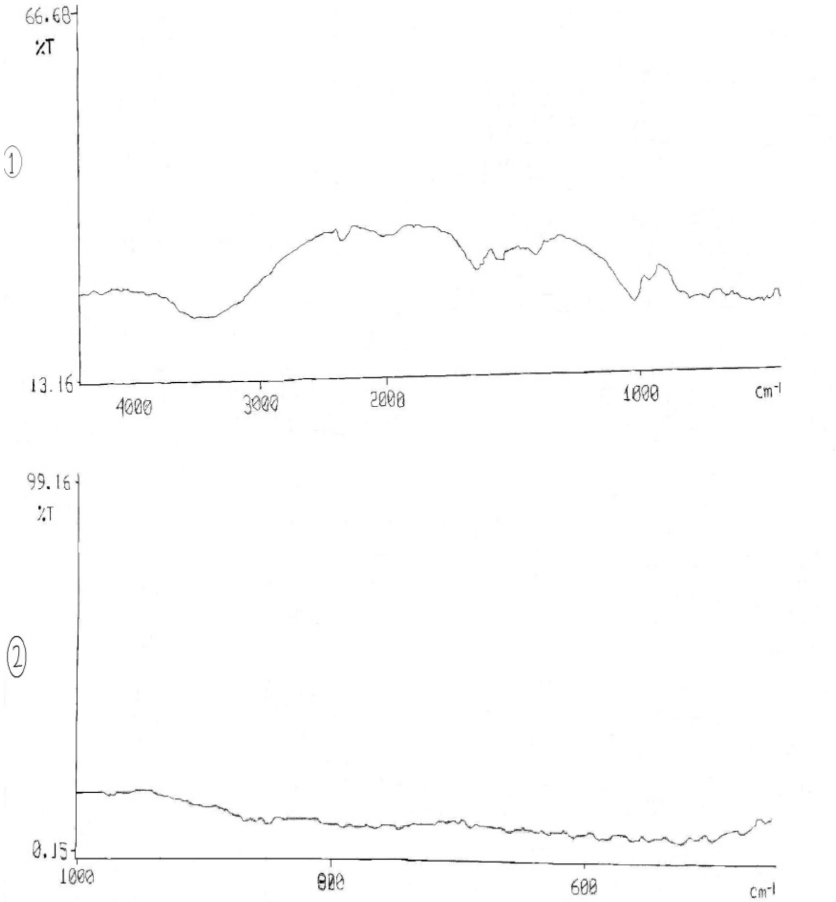

Figure 4: IR patterns for (1) the waste and (2) γ-alumina.

4 Conclusions

In conclusion, it was found that, under the suitable conditions investigated, about 88% of pure alumina (97.8%) can be recovered from the solid waste obtained as a by-product from some aluminum small factories. Fig. 5 illustrates a recommended flow sheet for the whole recycling process required for the production of alumina from the solid waste under investigation. The future work will be done on the uses of alumina as an effective adsorbent for treatment of both industrial and radioactive waste solutions.

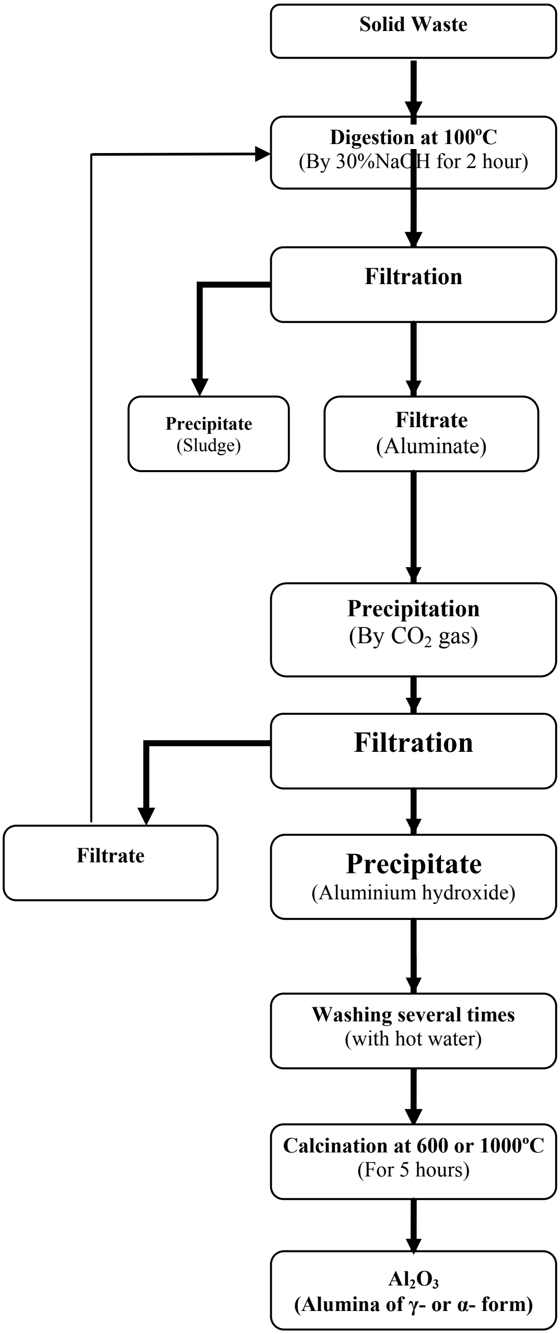

Figure 5: Flow sheet of the recycling process for producing alumina from the solid waste.

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