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Preparation of Aluminum Oxide from Industrial Waste Can Available in Bangladesh Environment: SEM and EDX Analysis

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Abstract

Aluminum oxide is an important chemical due to its many valuable properties such as hard ware resistance, good thermal conductivity, high strength and stiffness. Aluminum oxide is commonly referred to as alumina, possesses strong ionic interatomic bonding giving rise to its desirable characteristics. It can exist in several crystalline phases which all revert to the most stable hexagonal alpha phase at elevated temperatures. There are different types of Can which are used in everywhere. Most of the energy drinks are marked as canning (in which used mostly aluminum sheet). Can is a waste material which polluted our environment. We have prepared aluminum oxide from industrial Can by two methods. One is acid method and another one is alkali method. UV, thermo gravimetric (TGA), SEM and EDX analysis have been done. It has been showed that acid method is more feasible to prepare aluminum oxide.

Keywords: Aluminum oxide; Thermo gravimetric; SEM; EDX; Environment

Introduction

Aluminum wastes [1] cans recycling provide many environmental, economic and community benefits to individuals, communities, organizations, companies and industries. Recycling aluminum waste cans [2] save precious natural resources, energy, time and money. Money earned from recycling cans helps people help themselves and their communities. Zeepperfeld [3] prepared aluminum hydroxide from alkaline aluminates. Aluminum hydroxide [4-6] also recovered from aluminum containing waste solution [7].

Alpha phase alumina is the strongest and stiffest of the oxide ceramics. Its high hardness, excellent dielectric properties, refractoriness and good thermal properties make it the material of choice for a wide range of applications. High purity alumina is usable in both oxidizing and reducing atmospheres to 1925°C. Weight loss in vacuum ranges from 10⁻⁷ to 10⁻⁶ g/cm²sec is over a temperature range of 1700°C to 2000°C. It resists attack by all gases except wet fluorine and is resistant to all common reagents except hydrofluoric acid and phosphoric acid. Elevated temperature attack occurs in the presence of alkali metal [8,9] vapors particularly at lower purity levels.

The composition of the ceramic body can be changed to enhance particular desirable material characteristics. An example would be additions of chrome oxide or manganese oxide to improve hardness and change color. Other additions can be made to improve the ease and consistency of metal films fired to the ceramic for subsequent brazed and soldered assembly.

Ceramic aluminas are generally produced by calcining Bayer aluminum hydroxide at temperatures high enough for the formation of α -Al₂O₃. By control of calcinations time and temperature and by the addition of mineralizes. Such as fluorine and boron, the crystallite size in the calcined product can be varied from 0.2 to 100 μ m. These calcined aluminas can be categorized broadly according to their sodium content. There are two general types: those having about 0.5% Na₂O and low-soda grades with content 0.1%.

Reactive alumina is a material manufactured by dry grinding calcined alumina [10-12] to particle sizes smaller than 1 μ m. The large

surface area associated with very fine particles and the high packing densities obtainable considerably lower the temperatures [13] required for sintering. Tabular aluminas are manufactured by grinding, shaping, and sintering calcined alumina. The thermal treatment at 1900-2150 k causes the oxide to recrystallize into large, tabular crystals of 0.2-0.3 mm.

The model of industrial ecology emphasizes the containment and reuse of wastes generated by society as an overarching guideline for improving environmental quality. To realize this model, industry and society should work together to recover metals by recycling waste metal from all secondary sources and losing a minimum amount of material from the industrial/social system [14,15]. Aluminum oxide preparation from wastage cans by acid and alkali methods is the aim of this present work.

Experimental

Materials

We have collected three types of cans from our local market. These three types of cans are- (1) Pran juice can, (2) Tiger energy drink can, (3) Wild Brew energy drink can. Reagent grade tartaric acid, thioglycolic acid, ammonia, nitric acids are required for the determination of iron. To prepare aluminum oxide by acid method conc. HCl, NH₄Cl, 1:1 ammonia solution, NaOH, H₂O₂, and distilled water are required. Again conc. HCl, NaOH, H₂O₂, and distilled water are required for alkali method to prepare aluminum oxide.

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Methods

Determination of iron by ammonia method: Ammonia reacts with trivalent iron in presence of thioglycolic acid produces pink color. Depending upon the intensity of pink color, iron content could be determined spectrophotometrically. The maximum molar absorptivity of the above developed color was found at 540 nm using UV visible spectrophotometer (Model: UV-2201 Shimadzu, Japan).

Preparation of standard stock solution: 0.864 g pre-dried A.R. ferric ammonium sulphate was dissolved with double distilled water containing 1 ml conc. HCl into a 100 ml volumetric flask and diluted up to the mark. It produced a solution of iron concentration of mg/ml.

Preparation of reagent:

1. Thioglycolic acid: 10 ml thioglycolic acid was dissolved to 10 ml by double distilled water into a 100 ml volumetric flask.

2. Tartaric acid: 10 g tartaric acid was dissolved with distilled water and diluted to 100 ml into a 100 ml volumetric flask.

3. Ammonia: 1:1 ammonia was used for the purpose.

4. Preparation of standard sample: 1 ml stock solution of concentration 1 mg/ml iron was taken in a 100 ml volumetric flask and diluted up to the mark by water to give a solution of 0.01 mg/ml iron.

The absorption was measured by UV visible spectrophotometer at 535 nm in a 1 cm cell against a reagent blank and the absorption reading were plotted against concentration. A linear straight line was obtained intercepting zero which was used for future sample determination.

Determination of the iron from the sample: 0.5 g sample is dissolved with 10 ml 1:1 HCl. Then it is diluted up to 100 ml. 5 ml solution is taken in a beaker and 25 ml nitric acid is added and it is heated for 20 minutes to convert the entire ferrous ion to ferric ion. After heating the solution it is up to 25 ml. 5 ml sample is taken and dissolve up to the 100 ml. from then 1 ml sample is taken to prepare 10 ml solution in which iron is determined. It is found that the percentage of iron in the Pran juice can is higher (around 60%) than the Tiger energy drink can and in the Wild Brew can (around 0.5%).

Preparation of aluminum oxide by acid method: 5 g of aluminum sheet is taken and it is reacted with 40 ml HCl with diluted 160 ml distilled water. By this reaction aluminum sheet is converted to aluminum chloride.

2Al (impure)+6HCl (aq) \rightarrow 2AlCl₃+3H₂

This aluminum chloride solution is filtered to remove the foreign impurities and this filtrate is diluted up to the about 1000 ml. 5 g NH₄Cl is then added to the solution and boiled. After boiling, about 80 ml 1:1 HCl is added with drop wise by stirring with an electrical stirrer. At this position aluminum hydroxide is produced by the reaction of aluminum chloride with ammonium hydroxide. This aluminum hydroxide is separated by using a pressure filter and in this position sodium hydroxide is added to aluminum hydroxide and diluted it up to 500 ml and heated the solution 10-15 minutes. 5 ml H₂O₂ is added to settle the Fe(OH)₂. Sodium aluminates are formed which is soluble in water, and the insoluble compounds are settled down.

 $Al(OH)_3 + NaOH \rightarrow NaAlO_2 + 2H_2O$

After setting the solution, the solution is filtered by the pickup with help of a pipette and pipette filler. In this filtered solution 1:1 HCl is added drop wise about 80 ml to complete the reaction to form aluminum hydroxide. This aluminum hydroxide is washed at least 6 times in one day to remove sodium chloride to get the pure product. After washing 6 times, the solution is filtered by the filter press. Thus we get aluminum hydroxide which is kept in the dryer at about 105°C. After drying aluminum hydroxide, it is kept in the furnace with about 800°C to form aluminum oxide.

$$2\text{Al(OH)}_3 \xrightarrow{800^{\circ}\text{C}} \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O}_3$$

This aluminum oxide is grinded in a grinder and it is passed through 100 meshes and final product is obtained (Figures 1-4).

Preparation of aluminum oxide by alkali method: 5 g of aluminum sheet is taken and it is reacted with 9 g NaOH and diluted with 250 ml distilled water. NaOH is reacted with aluminum sheet to form sodium aluminate. Then this solution is filtered to remove foreign particles. After heating (until boiling), about 3-5 ml H_2O_2 is added to this solution and it is kept for 24 hours to settle ferric oxide which is brick red in color. After settling, clear solution is picked up by the pipette filler and filtered. About 80 ml 1:1 HCl is added in the filtrate to form aluminum hydroxide.

 $NaAlO_2+2HCl+H_2O \rightarrow Al(OH)_3+2NaCl$

Then the aluminum hydroxide is kept in a woven at 105°C temperature for drying. Again it is kept in a furnace at about 800°C temperature to form aluminum oxide. This aluminum oxide is grinded in a grinder and it is passed through 100 meshes and final product, aluminum oxide is obtained.

Results and Discussion

We have studied about three different types of cans which we have collected from our local market. At first, we analyzed the percentage of iron which is the main impurities of aluminum oxide preparation. In the experiment, we have seen that in the Pran juice can, the percentage of iron is around 60% where as in the Tiger energy drink can and in the Wild Brew can, the percentage of iron is around 0.5%. In case of Pran juice can, extraction of aluminum oxide was not as good as like as other two brands due to the higher percentage of iron. Again it was very difficult to get pure aluminum oxide from the Pran juice can. On the other hand the product yield from the Tiger energy drink can and from the Wild Brew can was higher than the Pran juice can. The product quality as well as product quantity of the Tiger energy drink can and the Wild Brew can were superior to the Pran juice can. Acid method and alkali method were used separately to prepare aluminum

Figure 1: Prepared raw materials.



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Figure 2: Preparation of Aluminum oxide



Figure 3: Aluminum hydroxide prepared in the laboratory.



Figure 4: Aluminum oxide

oxide. We have seen that acid method was better than the alkali method because of the product quality and quantity both was higher in the acid method.

From the Figures 5-7, we have seen that about 300°C temperature is the suitable temperature to convert aluminum hydroxide to aluminum oxide. But about 800°C is the actual temperature to convert aluminum hydroxide to aluminum oxide. So, we can say that water and impurities help to convert aluminum hydroxide to aluminum oxide in less temperature. On the other hand, in case of alkali process for Tiger energy drink can about 275-300°C was needed to convert aluminum hydroxide to aluminum oxide. In the alkali process impurities were more than the acid process.

From the SEM analysis, we have seen that the surface of the grinding aluminum oxide magnifying 1000 times and the surface of the aluminum oxide was not smooth. So, we can say that it was not too much fine particles. May be impurities are responsible for the roughness of the surface (Figures 8-11).

From the analysis of EDX (Energy Dispersive X-ray) and Table 1, we have seen that 52.81% aluminum and 44.28% oxygen were present in the product. In the raw materials percentage of iron was about 0.5% but it was 0.17% in the product. Na and Cl₂ were also present in the product because those were added in washing steps. Because washing procedure was very lengthy and for this reason 1.24% Na and 1.18% Cl₂ was present in the acid method to produce aluminum oxide from Tiger energy drink can. We have also seen that K and Mn were present in the product below detection level (Figure 12).

In case of alkali method of Tiger energy drink can, from EDX analysis and Table 2, we have seen that the percentages of aluminum and oxygen were lower than the acid method. Again in the alkali method we have observed that the final product contained higher amount of Na and Cl_2 due to available NaOH was used in the method. So the product quality was not pure as like as acid method. In this method, K and Mn were absent but Fe was presenting similar to acid method (Figure 13).

Conclusion

Aluminum oxide is a material of commercial importance due to its many valuable properties such as hard ware resistance, excellent ware resistance, good thermal conductivity, excellent size and shape capability, high strength and stiffness. For the above properties it is used adsorbents, catalyst, high temperature electrical insulators, high voltage insulators, furnace liver tubes. Thermal sensors, laboratories



Figure 5: Thermogravimetric analysis of aluminum hydroxide, prepared by acid method of Tiger energy drink can. DTG=Differential Thermo Gravimetry, DTA=Differential Thermal Analysis, TG=Thermo Gravimetry.

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Figure 8: 500 times magnification of aluminum oxide (acid method of Tiger energy drink can).



Figure 9: 500 times magnification of aluminum oxide (alkali method of Tiger energy drink can).



Figure 10: 1000 times magnification of aluminum oxide (acid method of Tiger energy drink can).



Figure 11: 1000 times magnification of aluminum oxide (alkali method of Tiger energy drink can).





Figure 13: Spectrum of prepared aluminum oxide (alkali method) by EDX.

Element	Net Counts	Weight%	Weight% (Error)
0 ₂	7019	44.28	± 0.73
Na	418	1.24	± 0.17
AI	32898	52.81	± 0.51
Cl ₂	444	1.18	± 0.15
К	57	0.16	± 0.12
Mn	21	0.16	± 0.29
Fe	20	0.17	± 0.34
Total		100.00	

 Table 1: Quantitative analysis of aluminum oxide (acid method) which is prepared from Tiger energy drink can.

Element	Net Counts	Weight%	Weight% (Error)
0 ₂	6358	39.47	± 0.70
Na	2120	5.45	± 0.41
AI	33027	48.71	± 0.47
Cl ₂	2609	6.18	± 0.37
ĸ	0	0.00	
Mn	0	0.00	
Fe	24	0.18	± 0.32
Total		100.00	

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 Table 2: Quantitative analysis of aluminum oxide (alkali method) which is prepared from Tiger energy drink can.

instrument tubes and sample holders, grinding media and other ceramic uses. Different types of chemicals such as alum are prepared from aluminum oxide. Wastage cans were collected and aluminum oxide was prepared by acid and alkali methods. In the acid method, product quality was higher. In the alkali method, sodium chloride was the main impurities. UV- spectrometric analysis was done due to study raw materials analysis for iron. From SEM analysis it was seen that the surface of aluminum oxide is rough and from EDX result we were confirmed the purity of the product. We have also studied aluminum hydroxide by TGA and seen that 270°C temperature was needed to convert it to aluminum oxide. It was very low temperature because water and impurities were available there.

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