Proceedings of the
International Conference on Mechanical Engineering and Renewable Energy 2013
(ICMERE2013) 1 – 3 May 2014, Chittagong, Bangladesh

ICMERE2013-PI-293

CRYSTALS GROWN FROM POTASH ALUM BY SLOW EVAPORATION METHOD IN BNGLADESHI ENVIRONMENT

Shourav Deb Nath[1] and Md. Aminul Islam[2,*]

[1, 2]Department of Mechanical Engineering
Chittagong University of Engineering and Technology, Chittagong, Bangladesh

[1]shourav_cuet08@yahoo.com, [2,*]aislam@cuet.ac.bd

Abstract- *Crystal has numerous applications in our day to day life. Along with that, the parameters of crystals vary depending upon the ingredients or elements. Many researches are going on various properties and applications of crystals from manufacturing process to the final product. Evaporation method was used to produce crystals of potassium aluminum sulfate (potash alum) which was synthesized from recycling of aluminum scrap.*

Keywords: Crystal growth, Potassium aluminum sulfate, Recycling.

1. INTRODUCTION

Crystals are defined as pure solids with a regular shape and sharp edges. They tend to be shiny. All crystals of one chemical substance will have the same shape which differs from the crystalline shape of other chemicals. Large crystals are usually identifiable by their macroscopic geometrical shape, consisting of flat faces with specific, characteristic orientations [1].

In crystallography, crystal structure is a unique arrangement of atoms in a crystalline liquid or solid and The unit cell is the smallest structure that repeats itself by translation through the crystal which can be constructed by these symmetrical units with the hard spheres. The most common types of unit cells are the faced-centered cubic (FCC), the body-centered cubic (BCC), the hexagonal close-packed (HCP) and other types exist, particularly among minerals. There are seven crystal lattice systems- cubic, tetragonal, orthorhombic, hexagonal, trigonal, triclinic and monoclinic [2].

Crystallization is the (natural or artificial) process for the formation of solid crystals from a uniform solution and the crystal growth is the subsequent growth of the nuclei that succeed in achieving the critical cluster size[3]. Nucleation and growth continue to occur simultaneously while the supersaturation exists.

The objectives of the study are:

1. To familiarize with slow evaporation method of crystal growth.
2. To prepare and compare the crystals of potash alum and potassium dichromate.
3. To familiarize with the need of recycling solid wastes particularly scrap metals, like aluminum.

2. METHODS OF CRYSTAL GROWTH

Growth of crystal ranges from a small inexpensive technique to a complex sophisticated expensive process and crystallization time ranges from minutes to months. Crystal growth may be classified into three categories as follows:
Solid Growth - Solid-to-Solid phase transformation
Liquid Growth - Liquid to Solid phase transformation
Vapor Growth - Vapor to Solid phase transformation

2.1 Hydrothermal growth

Hydrothermal implies conditions of high pressure as well as high temperature in aqueous solution. Substances like calcite, quartz is considered to be insoluble in water but at high temperature and pressure, these substances are soluble. Temperatures are typically in the range of 400° C to 600° C and the pressure involved is large (hundreds or thousands of atmospheres).

2.2 Slow cooling technique

It is the best way to grow single crystals by solution technique with the main limitation of using a range of temperature. The possible range of temperature is usually so small that much of the solute remains in the solution at the end of the run. To compensate this effect, large volumes of solution are required. The temperature at which such crystallization can begin is usually within the range 45 °C - 75 °C and the lower limit of cooling is the room temperature.

2.3 Slow evaporation method

This method is similar to the slow cooling method in view of the apparatus requirements. The temperature is fixed and provision is made for evaporation. With non-toxic solvents like water, it is permissible to allow evaporation into the atmosphere. Typical growth conditions involve temperature stabilization to about ± 0.005°C and rates of evaporation of a few ml /hr.

2.4 Temperature gradient method

This method involves the transport of the materials from a hot region containing the source material to be grown to a cooler region where the solution is supersaturated. Changes in the small temperature differences between the source and the crystal zones have a large effect on the growth rate.

2.5 Gel growth

Gel is a two-component system of a semisolid rich in liquid and inert in nature. The material, which decomposes before melting, can be grown in this medium by counter diffusing two suitable reactants.

2.6 Growth from melt

All materials can be grown in the form of single crystal from the melt provided that they melt congruently without decomposition at the melting point and do not undergo any phase transformation between the melting point and room temperature.

2.7 Growth from vapor

Crystals of high purity can be grown from vapor phase by sublimation, condensation and sputtering of elemental materials. To obtain single crystals of high melting point materials this method is used [4].

3. PREPARATION AND PURIFICATION OF THE POTASH ALUM CRYSTAL

Aluminum beverage cans generally have a thin coating of plastic on the inside that protects the aluminum from the corrosive action of the chemicals in the beverage. The outside usually has a thin coating of paint. These coatings must be removed before any chemical reactions with the metal can be carried out. The coatings may be effectively scraped off with a metal pan cleaner. A cleaned piece of metal is then dissolved in a potassium hydroxide solution according to the following complete, balanced equation:

$2\ Al(s) + 2\ KOH(aq) + 6\ H_2O(liq) \rightarrow 2\ KAl(OH)_4(aq) + 3\ H_2(g)$

or the net ionic equation

$2\ Al(s) + 2\ OH^-(aq) + 6\ H_2O(liq) \rightarrow 2\ Al(OH)_4^-(aq) + 3\ H_2(g)$

The dissolution of Al(s) in aqueous KOH is an example of an oxidation-reduction or redox reaction. [The Al metal is oxidized to aluminum with an oxidation number of +3 and the hydrogen in KOH or in water is reduced from an oxidation number of +1 to zero in hydrogen gas.] The $Al(OH)_4^-$ ion is a complex ion called “aluminate." These reactions illustrate the reason that alkaline products (such as detergents, cleaners, shampoos, and so on) are never stored in an aluminum container. The aluminum would slowly dissolve! After filtration to remove residual plastic and paint decomposition products, the alkaline solution of $Al(OH)_4^-$ is clear and colorless. The H_2 is evolved as a gas and mixes with the atmosphere. The chemical species in solution are potassium ions (K^+) and aluminate ions [$Al(OH)_4^-$] ions (plus any unreacted KOH). Sulfuric acid is now added and two sequential reactions occur. Initially, before the addition of all the acid, the complete reaction is

$2\ KAl(OH)_4(aq) + H_2SO_4(aq) \rightarrow 2\ Al(OH)_3(s) + 2\ H_2O(liq) + K_2SO_4(aq)$

or the net ionic equation

$Al(OH)_4^-(aq) + H^+(aq) \rightarrow Al(OH)_3(s) + H_2O(liq)$

The reaction above is an acid-base reaction in which the H+ ions from the sulfuric acid neutralize the base $Al(OH)_4^-$ to give a thick, white, gelatinous precipitate of aluminum hydroxide, $Al(OH)_3$. As more sulfuric acid is added, the precipitate of $Al(OH)_3$ dissolves.

$2\ Al(OH)_3(s) + 3\ H_2SO_4(aq) \rightarrow Al_2(SO4)_3(aq) + 6\ H_2O(liq)$

or the net ionic equation is

$Al(OH)_3(s) + 3\ H^+(aq) \rightarrow Al^{3+}(aq) + 3\ H_2O(liq)$

To give aluminum ion Al^{3+}, in solution. The solution at this point contains Al^{3+} ions, K^+ ions (from potassium hydroxide), and SO_4^{2-} ions (from sulfuric acid). On cooling, crystals of hydrated potassium aluminum sulfate, $KAl(SO_4)_2$•12 H_2O (or alum) are very slowly deposited. In the experiment the crystallization process is speeded up by providing a small “seed crystal" of alum for the newly forming crystals to grow on. Cooling is needed because alum crystals are soluble in water at room temperature. The complete equation is

$Al_2(SO_4)_3(aq) + K_2SO_4(aq) + 24\ H_2O(liq) \rightarrow 2\ KAl(SO_4)_2\bullet 12\ H_2O$

and the net ionic equation is

$K^+(aq) + Al^{3+}(aq) + 2\ SO_4^{2-}(aq) + 12\ H_2O(liq) \rightarrow KAl(SO4)_2\bullet 12\ H_2O(s)$

Finally, the crystals of alum are removed from the solution by vacuum filtration and washed with an alcohol/water mixture. This wash liquid removes any contamination from the crystals but does not dissolve them. It also helps to dry the crystals quickly, because alcohol is more volatile than water [5].

4. Preparation of solution and crystal growth

Can was pierced at the lower end and cut around the sides. The rectangular pieces of aluminum (the sides of the can) were placed on the bench and both sides were scrubbed keeping an area of 2" x 2(1/4)" on both sides. The metal was cleaned with a paper towel and weighed by an analytical balance. The weight of the piece was kept not more than 1 g. The weighed piece was cut into smaller pieces and placed in a clean 250 mL beaker. After that 50 mL of 1.4 M KOH was added. The beaker was placed on the gauze and heated on a low flame and the aluminum will be dissolved within 20 minutes. When the aluminum was dissolved (as evidenced by the lack of bubbles of H_2 gas given off), solution was gravity filtered

Allow the flask to cool and wash the funnel and beaker with lots of tap water to remove any trace of potassium hydroxide. Then 18 mL of 10 M H_2SO_4 (with a graduated cylinder) was added quickly and placed on the Bunsen burner apparatus and warm it with swirling until all of the solid material has dissolved.

Fig. 1: Seed crystal of Potash Alum

5. PREPARATION OF SEED AND CRYSTAL GROWTH

Seed crystals are best prepared by slow cooling or slow evaporation of a saturated solution in a clean, controlled temperature enclosure kept especially for this purpose.

Seeds of good visual quality as shown in Fig. 1, free from any inclusion and imperfection are chosen for growth provided seed should be suspended in a saturated solution on a smooth support to avoid spurious nucleation. During the crystal formation, crystals grown at the bottom or sides should be removed to reduce the competition in order to have a large crystal. Potassium dichromate seeds were prepared from saturated potassium dichromate solution resulting crystals with bright and orange in color [6].

6. RESULTS AND DISCUSSIONS

Data was collected for seed crystal growth rate and used a piece of crystal as a seed for growing. Potash alum seed crystal was 4.42 mm.

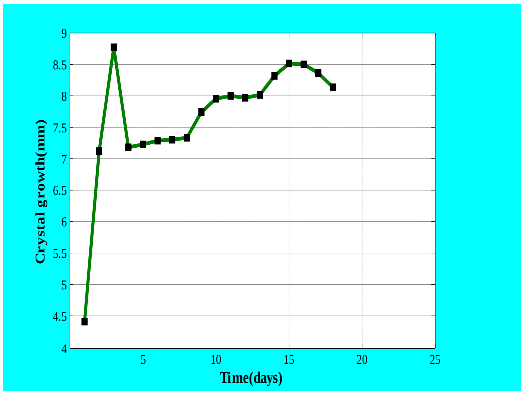

Fig. 2: Growth of potash alum crystal

The Fig. 2 depicts the potassium aluminum sulfate crystal growth rate with time. Initially the crystal size increased for three days and crystal size plummeted next day because of other crystals started to grow. As a result, some crystal from seed goes back to the solution again. After removing the undesirable crystals, the seed crystal began to grow again slowly. There was a significant decrease in the crystal growth rate in the last stages as the solution became unsaturated and some of the seed crystal tried to dissolve in the solution.

7. CONCLUSION

Crystal growth by slow evaporation method proved to be quite successful for both potash alum Focus was given for identifying and analyzing the crystal growth process and recycling aluminum scrap metal. Further variations and improvement in the crystal growth rate could be found if the temperature and evaporation rate was varied.

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