Rev. Adv. Mater. Sci. 28 (2011) 40-43

CHEMICAL SYNTHESIS OF ALUMINUM CHLORIDE ($AlCl_3$) BY COST-EFFECTIVE REDUCTION PROCESS

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Received: February 17, 2011

Abstract. Aluminum chloride ($AlCl_3$) powder was synthesized using a novel method that is feasible and cost-effective reduction process. Gas mixture of $AlCl_3$ and $FeCl_3$ was formed by the reaction of aluminum (Al) and iron chloride ($FeCl_3$) at 450 °C for 5 hours under ultra-high purity argon atmosphere. The powder mixtures of $AlCl_3$ and $FeCl_3$ were obtained once they were cooled at room temperature. The $AlCl_3$ powder was only evaporated and then separated from the mixture at 150 °C 5 hours owing to different evaporating temperature, while $FeCl_3$ powder was left behind as a solid phase. The $AlCl_3$ gas was condensed into the fine powder and gathered in cooling chamber under ultra-high purity argon atmosphere. The $AlCl_3$ powder was examined and identified by a X-ray diffraction (XRD) and an Energy Dispersive X-ray Spectroscopy (EDX).

1. INTRODUCTION

Aluminum chloride ($AlCl_3$) has been used in producing aluminum metal powder, which is a raw material for manufacturing a solar cell and electromagnetic components [1-3]. Moreover, it is widely applied as acid catalyst, isomerization of hydrocarbon, catalyst of aldehyde synthesis, and catalyst of cation polymerization. In case of an anhydride, it is also used for cracking of oil, and catalyst of Friedel-Crafts reaction in an organic synthesis [4,5].

Many methods for fabrication of anhydrous aluminum chloride have been suggested for the past decades, but methods in traditional production process of aluminum chloride used to have injecting chlorine gas (Cl), which is known to be environmentally harmful, into the aluminum melting bath at high temperatures.

In this study, aluminum chloride can be produced by using iron chloride extracted from wasted solution, and aluminum scraps achieved from aluminum can and foil. This novel process newly proposed is helpful to recycle wasted resources and protect an environment, and also can reduce production cost by lowering cost for purchasing raw materials. Aluminum chloride was achieved through a method of distillation and condensation by the use of different vaporization temperature of iron chloride and aluminum chloride, and high purity of aluminum chloride produced was successfully identified by an Energy Dispersive X-ray Spectroscopy (EDX) and a X-ray diffraction (XRD).

2. EXPERIMENTAL PROCEDURES

Iron chloride ($FeCl_3$) and aluminum metal (Al) used in this study were recycled from wasted materials. The iron chloride was used by extracting it from wasted solution obtained from a pickling liquid for processing the surface of steel board, and the aluminum chloride was recycled from wasted aluminum scraps such as aluminum can or foil.

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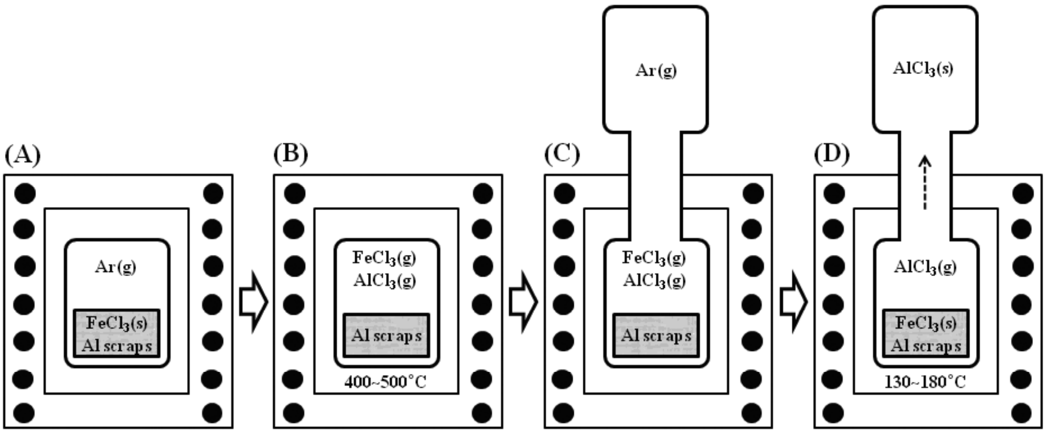

Fig. 1. Schematic diagram of experimental apparatus and procedure.

Schematic diagram of experimental apparatus and procedure to fabricated aluminum chloride ($AlCl_3$) is shown in Fig. 1. In detail; (A) Iron chloride (50 g) and solid scraps of aluminum (20 g of aluminum foil pieces) were put into a sealing container under UHP argon atmosphere to avoid oxidation of Fe and Al. They were heated at desired temperature once placed in electric furnace. (B) As heated between 300~500 °C, iron chloride and aluminum chloride were vaporized by heating, but aluminum scraps still existed in a solid state. The sealing container was taken out of furnace to cool it down at room temperature after reaction was completed. (C) When cooling was completed, the sealing chamber containing aluminum scrap, aluminum chloride, and iron chloride were put back to the electric furnace under UHP argon atmosphere. The container was built with another empty container connected with on top of the sealing container inside the furnace. (D) Temperature was increased at 130~180 °C leading to produce aluminum chloride as a gas phase flowing to another container sealed. Once the gaseous $AlCl_3$ moved the sealing container located outside at room temperature, it will be naturally cooled down to become fine solid powders and their phases were checked by a X-ray diffraction (XRD) and an Energy Dispersive X-ray Spectroscopy (EDX).

3. RESULTS AND DISCUSSION

Fabrication of fine powders of aluminum chloride was carried out through three main processes. First step was to encourage iron chloride to react with aluminum to produce gaseous $FeCl_3$ and $AlCl_3$ together remaining Al scraps behind at 450 °C for 5 hours. The chemical reaction is stated below at temperature range from 25 to 500 °C.

$$FeCl_3 + Al \rightarrow AlCl_3 + Fe,$$

$$\Delta G = -290 \sim -300 kJ/mol.$$

This reaction clearly demonstrates that iron chloride reacted with solid aluminum can produce aluminum chloride that is created as the reaction occurs from the surface of solid aluminum. Second step was to cool the gasified mixture of iron chloride and aluminum chloride to transform them as solid phases. Last step was to separate aluminum chloride from the mixture of iron chloride and aluminum chloride at 150 °C for 5 hours using separated sealing container that is maintained under UHP argon atmosphere.

Stability diagram of aluminum chloride and iron chloride was obtained using *HSC Chemistry*[5.11] equilibrium software as a function of temperature and vapor pressure to expect evaporation temperatures of each material shown in Fig. 2. At 760 mm Hg (1 atm) in vapor pressure in experimental conditions, $AlCl_3$ is expected to be vaporized at about 450K and $FeCl_3$ at about 555K. From the results $AlCl_3$ as a gas phase can be separated from $FeCl_3$ remaining as a solid phase once heated at temperature below a vaporization temperature of $FeCl_3$.

Weight change of aluminum chloride ($AlCl_3$) and iron chloride ($FeCl_3$) was measured using thermogravimetric analyzer (TA Instrument, SDT Q600) as a function of temperature to find out exact points of evaporation temperature of each material. The change in weight with time was measured with elevation speed of 10 °C/minute in UHP argon at-

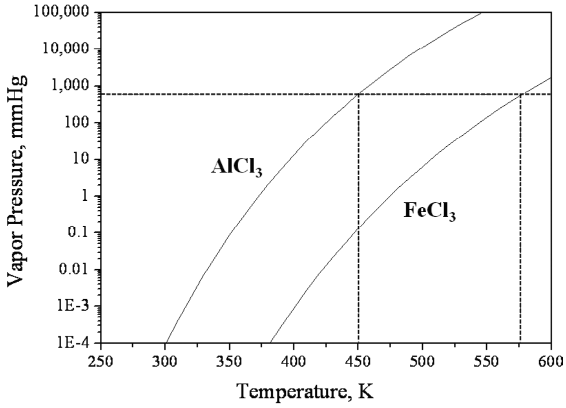

Fig. 2. Phase stability diagram of $AlCl_3$ and $FeCl_3$ as a function of temperature and vapor pressure.

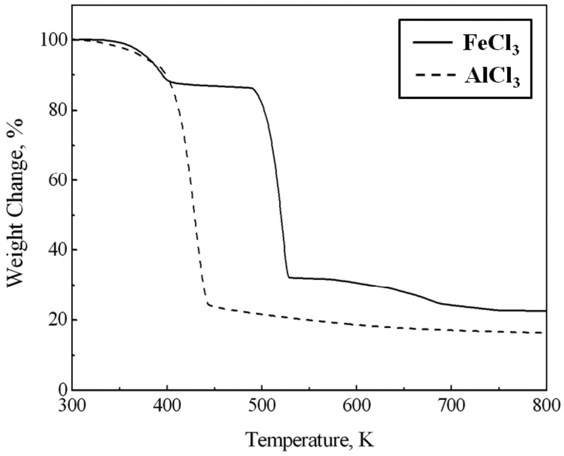

Fig. 3. Weight change of $FeCl_3$ and $AlCl_3$ with temperature.

mosphere. According to Fig. 3, the weight change of aluminum chloride was rapidly decreased at 423K meaning that the aluminum chloride charged as a solid phase was transformed to gas phase as being weight lost. Therefore, the evaporation temperature of aluminum chloride at argon atmosphere is found to be 423K. In case of iron chloride, it was seen that a minor change in weight was observed to be decreased at around 373K and the weight change was maintained until it rapidly decreased at 523K. It can be explained that the evaporation temperature of iron chloride was about 523K. Meanwhile, the fact that weight loss of the iron chloride was observed around 373K could be explained by evaporation effect of moisture contained in aluminum chloride.

X-ray diffraction (XRD) analysis of $AlCl_3$ powders synthesized at 150 °C for 5 hours was conducted to find out if the aluminum chloride has been formed well, and the results are shown in Fig. 4. It was

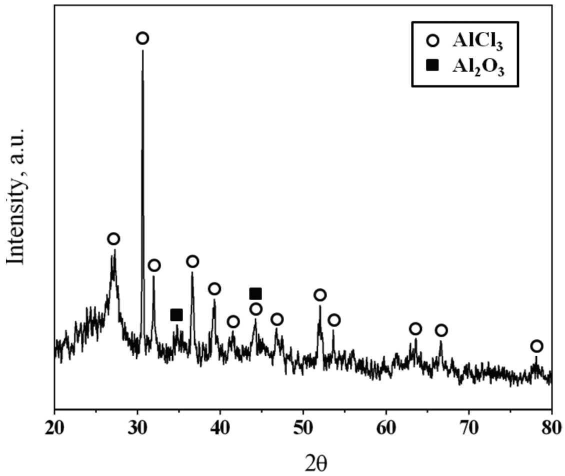

Fig. 4. XRD patterns of $AlCl_3$ powders synthesized at 150 °C for 5 hours.

seen that $AlCl_3$ peaks were mainly observed in XRD patterns indicating that $AlCl_3$ powder was successfully obtained, while a few of Al_2O_3 peaks were also detected. It might be explained that XRD measurement was conducted in air and $AlCl_3$ powders could be therefore oxidized easily at that moment.

Chemical composition of $AlCl_3$ powder was also analyzed using Energy Dispersive X-ray Spectroscopy (EDX), and the results are shown in Fig. 5. It showed that Al and Cl were detected with some amounts of O and Pt. It may be explained that O is oxygen exposed to air and Pt is the result of Pt coating that is used for measuring EDX. In the long run, the data examined by XRD and EDX would clearly support that the synthesis of the fine powder of $AlCl_3$ was completed and not contaminated from iron sources using cost-effective reduction process newly proposed in this study.

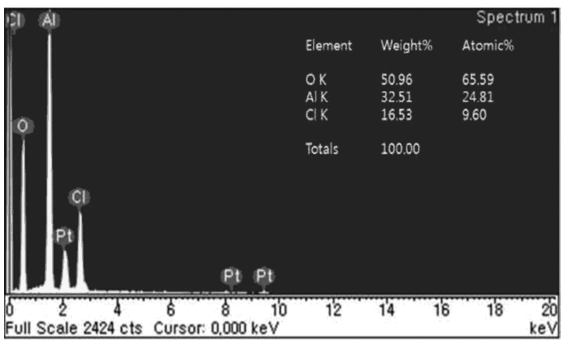

Fig. 5. Chemical composition of $AlCl_3$ powder analyzed by EDX.

4. CONCLUSION

Fine powders of aluminum chloride were fabricated with iron chloride and aluminum scrap as source materials in cost-effective reduction process.

1. The results from TGA analysis showed that weight loss was clearly observed at 150 °C for $AlCl_3$ and 250 °C for $FeCl_3$.
2. Aluminum chloride was successfully separated from the gas mixture of iron chloride at 150 °C for 5 hrs in consecutive processes of evaporation and condensation.
3. The $AlCl_3$ powders obtained were confirmed by XRD analysis, and chemical composition conducted by EDX verified that they were mainly consisted of Al and Cl.

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