Structure Transformation of Gibbsite by Calcination

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Gibbsite, which is aluminum oxide, was calcined in the temperature range from 200 to 1150°C, and the relation between the calcination temperature and various properties of gibbsite has been examined. It is speculated based on the results of TG-DTA experiment that the structure of gibbsite changed greatly at a calcination temperature of 282.7°C. Specific surface area of gibbsite kept on increasing as the calcination temperature is elevated up to 400°C, where it reached a maximum value. In the range where the calcination temperature is higher than 400°C, specific surface area decreased as the temperature was elevated further. In addition, it became evident from the results of XRD measurement that in the calcination temperature range lower than 200°C, the crystal kept gibbsite structure, but it changed to the structure of aluminum oxide of transition type above 300°C, and that it changed further to the structure of stable aluminum oxide when the calcination temperature was elevated over 1000°C. From these results, it is verified that by making use of the fact that the structure of gibbsite changes greatly in the calcination temperature range from 300 to 400°C, a novel adsorbent having boehmite structure can be produced.

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I. INTRODUCTION

Aluminum is a perfectly recyclable metal. Gibbsite as well as boehmite is components of bauxite, and it is well known that gibbsite is cheaper than boehmite. It is reported that boehmite had been utilized as a substance to adsorb and desorb phosphate ion which causes eutrophication occurring in the territories such as closed coastal seas, and also utilized as an adsorbent to remove heavy metal dissolved in drinking water [1]. It is reported that boehmite has adsorptive capacity. It is well known that specific surface area of boehmite decreases as the calcination temperature is elevated. And by calcined boehmite and suspending the powder in purified water, the amount of hydroxyl group on the surface increases. It reaches a maximum value when the calcination temperature is 400°C. Therefore, it is expected that calcined boehmite is a much more effective adsorbent to recover phosphate ion as compared with untreated boehmite.

However, due to a demerit of high production cost, its practical application is in a harsh situation. On the other hand, naturally-occurring gibbsite is very cheap, and the relation between the calcination temperature and various properties of gibbsite has been evaluated in terms of TG-DTA, XRD and specific surface area measurements.

In this study, gibbsite was calcined in the temperature range from 200 to 1150°C, and various physical properties of gibbsite were evaluated in terms of TG-DTA, XRD and specific surface area measurements.

II. EXPERIMENTAL

A. Materials

Higilite H-42M (Showa Denko, Japan) was used as a gibbsite specimen. The calcination procedure was as follows; 20 g of gibbsite was taken in a porcelain crucible, and it was heated in a muffle furnace up to a series of specified temperatures between 200 and 1150°C in 2 hrs, and the specimen was kept at that temperature for 2 hrs.

B. Characteristics of gibbsite

Differential thermal analysis of gibbsite was carried out using the Automatic Differential Thermal / Thermogravimetric Simultaneous Measuring Equipment DTG-60AH (Shimazu, Japan). X-ray diffractometeric analysis was conducted using RIN2100V (Rigaku, Japan), XRD conditions are 40 kV, 20 mA and Cu-Kα, and specific surface area was measured using Flow SorbII2300 (Micrometrix, USA).
III. RESULTS AND DISCUSSION

Figure 1 shows a thermogram of Differential Thermal Analysis for a gibbsite specimen. It is clearly shown that gibbsite loses most of the structural water above 282.7°C by thermal dehydration. In addition, TG-DTA curve gradually changes as temperature is elevated, which means that the structure of gibbsite continuously changes. Results of XRD for calcined gibbsite are shown in Fig. 2. Then, it became evident that when the calcination temperature is 200°C, the structure of gibbsite for calcined gibbsite at 200°C is the same as that of virgin gibbsite, but when the calcination temperature was higher than 300°C, structure of gibbsite was destroyed, and when the calcination temperature was 400°C, boehmite structure appeared and amorphous aluminum oxide was produced. There is a report [11] that the structure of the substance changes from gibbsite structure to boehmite structure, and in this study, it is reconfirmed that similar structural change happens when the substance is calcined at around 400°C. Furthermore, it must be pointed out that when the calcination temperature is higher than 1000°C, gibbsite became aluminum oxide with crystal structure which is a very stable substance. On the other hand, it is shown in a report that if the calcination condition is different, micro-pore sizes and the other physical properties are different, and also the transition state [12] and structure [13]...
of the produced aluminum oxide are also different. The highest calcination temperature in this study was 1150°C. But there is a report where gibbsite was calcined at much higher temperatures, namely 1200 and 1400°C [4], and so there is a possibility that the functional effect of gibbsite is clarified further more.

Figure 3 shows the relation between the calcination temperature and specific surface area of gibbsite. It is evident that specific surface area becomes maximum when the calcination temperature is 400°C. There is a report [7] that specific surface area becomes maximum when the calcination temperature is 300°C, but in this study the calcination temperature of about 400°C gave the maximum value of specific surface area. And it is recognized that the specific surface area decreased as the calcination temperature is raised in the range over 400°C. It is reported that specific surface area of pseudo-boehmite by calcining gibbsite at 500°C is 265 m²/g [14], and in the calcining experiment of gibbsite in this study, specific surface area at the calcination temperature of 500°C was 227 m²/g. The two values are very close with each other.

IV. CONCLUSIONS

It is clarified by this study that structure of gibbsite changes greatly at around 282.7°C, and when the calcination temperature is elevated further, the structure keeps on changing gradually. And specific surface area of gibbsite increases rapidly when the calcination temperature is about 300°C. Maximum value was obtained at the calcination temperature of 400°C. It was clarified that specific surface area decreases when the calcination temperature is elevated in the region over 400°C. It became evident that by calcining gibbsite, it changes to amorphous aluminum oxide, and then to aluminum oxide with stable crystal structure. By calcining gibbsite at 400°C, boehmite was obtained.

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