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SOME POSSIBILITIES OF THERMOANALYTICAL METHODS FOR TECHNOLOGICAL CONTROL OF ALUMINA PRODUCTION

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ABSTRACT

Some intermediates formed during alumina production have been examined using a complex thermal analysis. Based on this, the criteria for an optimal performance of the stages of alumina production, such as milling of alumina hydrate and washing of the precipitate from admixture salts, have been formulated.

INTRODUCTION

Alumina is widely used in different industries as a support catalyst, sorbent, and drying agent. The traditional way of its production is precipitation from solutions of salts. A technical-grade alumina hydrate (hydrargillite) is dissolved in acid or alkali, and then Al(III) hydroxides are precipitated from solutions by adding respectively alkali or acid. The resulted precipitates are filtered, washed from salts plasticized with an acid, formed into granules of the desired form, dried and calcined. The knowledge of the state of semiproducts and possibility to vary their properties allow intentional control of the quality of the produced alumina. In this communication the possibilities of the complex thermal analysis for express technological control of semiproducts during alumina production are discussed.

EXPERIMENTAL

The complex thermal analysis was performed in air at a heating velocity of 10 degree/min using an OD-103 derivatograph in platinum crucibles. The weight of each analyzed sample was 0.2 g. The X-ray phase analysis was carried out on a DRON-20 diffractometer using a CuK_{α} -irradiation and Ni-filter.

RESULTS AND DISCUSSION

The starting stage of alumina production, -- dissolution of hydrargillite in alkali or acid, -- requires high temperatures

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(up to 413 K) and prolonged time. Hydrargillite is dispersed to accelerate dissolution. In this work, hydrargillite, dispersed in a desintegrator was examined.

Two endothermic effects are observed on the thermal curves of the starting hydrargillite (see Fig. 1-1). The endothermic effect I in the temperature range 473-673 K is attributed to dehydration of hydrargillite and has a pre-effect. The endothermic effect II in the temperature range 723-853 K is due to dehydration of boehmite, produced in the course of hydrargillite dehydration. As known /1/, with decreasing the size of hydrargillite particles, the pre-effect of effect I and the related effect II diminish on its thermal curves. The thermal curves (see Fig. 1-2) of hydrargillite treated in the desintegrator reveal a considerable decrease in both the pre-effect of effect I and effect II. This occurs due to the diminishing of the size of hydrargillite particles during its treatment in the desintegrator and is in good agreement with our previous data /1/. Hence, a decrease or absence of the pre-effect of effect I on the thermal curves of hydrargillite may serve as a criterion for an optimal dispersion of hydrargillite.

Thermal analysis also provides the possibility to control the state of semiproducts at the stages of formation of aluminium hydroxide precipitate and its washing from the salts. Fig. 2 shows thermal curves of the initial aluminium hydroxide and of aluminium hydroxides that are produced by precipitating from the Al(III) nitrate solution with ammonia and are washed with different amounts of water. The thermal curves (see Fig. 2-1) of the freshly precipitated Al(III) hydroxide are characterized (i) by the presence of an endothermic effect at 373-503 K, caused by the removal of coordinatively-bound water and, partly, of structurally-bound water; and (ii) by an exothermic effect at 533-553 K, which results from the decomposition of ammonium nitrate and is accompanied by a high evolution of nitrogen oxides. The heat evolved in the case of the exothermic effect is quite sufficient to activate the thermal crystallization of amorphous alumina. The latter leads to the decrease in the specific surface area and in the porosity of Al(III) oxide. Alumina obtained from such semiproduct has a low mechanical strength.

To avoid these phenomena, Al(III) hydroxides are washed until no NO_3^- ions are present in the precipitates, water consumption being 200 m^3 per 1 ton of alumina.

We conventionally divide the process of washing into 3 stages: I stage -- an initial unwashed Al(III) hydroxide; II stage -- the precipitate has almost unwashed NO_3^- ions; III stage -- Al(III) hydroxide has no NO_3^- ions. Upon washing, as the amount of easily washed aluminium nitrate decreases samples, the exothermic effect observed on the thermal curves at 533-553 K diminishes down to its complete disappearance (see Fig. 2-2). However, according to the chemical analysis data, in such samples of Al(III) hydroxide there are also rather large amounts of almost unwashed NO_3^- ions. Besides, there appears an endothermic effect at 593-773 K (see Fig. 2-2), which arises from the dehydration of pseudoboehmite. Water consumption for this extent of washing is 3-4 times less than that for the complete washing of NO_3^- ion from the precipitates.

Further washing (stage III) leads to the complete removal of NO_3^- ions from the precipitates. In this case, on the thermal curves there appears also an endothermic effect at 543-603 K (see Fig. 2-3) caused by the formation of bayerite crystals in the precipitates. The change in the phase composition of the samples during washing and appearance of the bayerite phase in them result in a dramatic decrease of the mechanical strength of alumina granules.

Hence, the absence on the thermal curves of both: the exothermic effect, caused by the decomposition of ammonium nitrate, and the endothermic effect, resulted from the dehydration of bayerite, may serve as a criterion for the sufficient washing of the samples from salts.

Thus, based on the above samples we have shown that the complex thermal analysis provides the possibility to control the properties of semiproducts and their effect on the quality of alumina obtained by precipitation.

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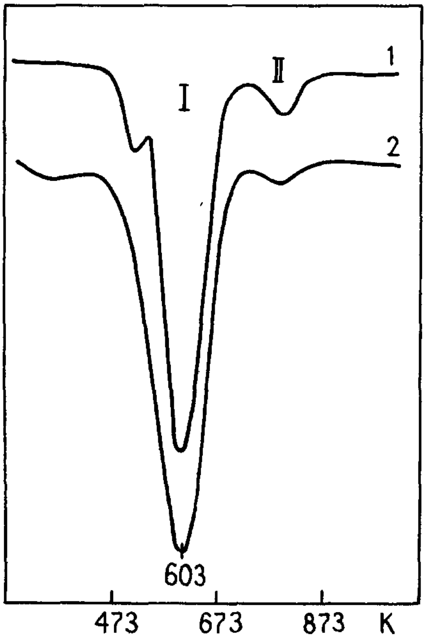

Fig. 1. DTA curves of the initial hydrargillite 1, the desintegrated hydrargillite 2.

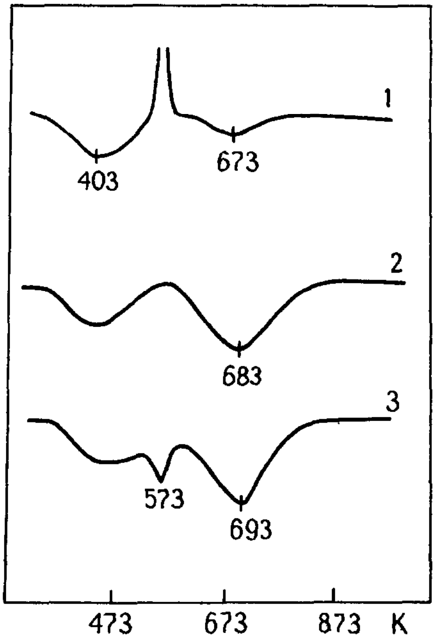

Fig. 2. DTA curves of the freshly precipitated unwashed Al(III) hydroxide -- 1, after the IInd stage of washing -- 2, and the IIIrd stage -- 3.