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INVESTIGATION OF THERMAL DECOMPOSITION OF HYDRATED ALUMINIUM OXIDE

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The composition of a hydrated aluminium oxide was determined and the course of its thermal decomposition was investigated. The stages of the dissociation were established and all the solid and gaseous intermediates of the decomposition were identified. A correlation was observed between the peak temperatures for boehmite and the particle size and degree of crystallinity.

Hydrated aluminium oxides occur as intermediates in the production of metallurgical aluminium oxide from Polish clays by the modified acidic Bretsznajder method [1].

They are obtained during the additional hydrolysis of basic aluminium ammonium sulphate in thr presence of ammonia. The application of such additional hydrolysis seems to be a very important element in the modified conception of the Bretsznajder method. The thermal decomposition of hydrated aluminium oxide appears to be a relatively simple process, as distinct from the thermal dissociation of basic aluminium ammonium sulphate, for the only gaseous decomposition product is water.

Depending on the conditions of the hydrolysis process, there are various possibilities for the preparation of hydrated aluminium oxides differing from each other in composition and properties.

Experimental and results

The aim of the present work was to study the thermal decomposition of hydrated aluminium oxide, obtained by washing out sulphates from basic aluminium ammonium sulphate with aqueous ammonia solution at 343 K.

The course of decomposition of this hydrated aluminium oxide was compared with those for the commonly known aluminium hydroxides, the main components of bauxites, which are the fundamental materials in Bayer's method of producing metallurgical aluminium oxide.

The studies were carried out by complex thermal analysis (DTA, TG, DTG, MS) supplemented with X-ray phase analysis.

Both freshly-prepared and aged hydrated aluminium oxide samples were used in the studies. Figures 1–3 present thermal curves of the starting hydrated aluminium oxide and samples obtained by roasting this compound at 630 K and 800 K. The results of X-ray phase analysis of the starting hydrated aluminium oxide and partly decomposed samples are presented in Table 1.

Analysis of the results of these investigations and cumulative data concerning natural and synthetic aluminium hydroxides [2, 3] showed that freshly-prepared hydrated aluminium oxide appeared to be aluminium gel, which after a few days crystallized to boehmite (γ-AlOOH) and bayerite (α-Al(OH_3)).

The thermogravimetric curves indicate that the thermal dissociation of the hydrated aluminium oxide proceeds in two stages, with characteristic peaks at 573 K and 733 K, corresponding to dehydration of the compound. By means of the X-ray diffraction data, a solid intermediate in the decomposition of hydrated aluminium oxide was identified as boehmite (γ-AlOOH). The final product of the decomposition is α-Al_2O_3.

In contrast with the peak temperatures for the dehydration of bayerite, the peak temperatures for the dehydration of boehmite differ from each other in the different samples.

The results seem to suggest that the peak temperature for boehmite-dehydration is very dependent on such factors as the particle size and degree of crystallinity, which differed in the examined hydrated aluminium oxide samples, depending on the preparation conditions.

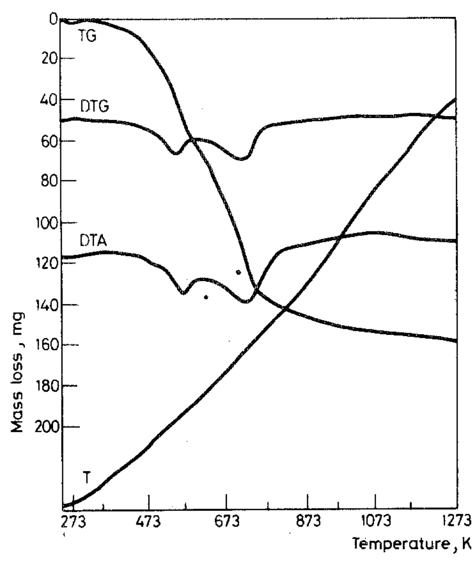

Fig. 1 TG, DTG and DTA curves of starting hydrated aluminium oxide; heating rate 10 degree/min

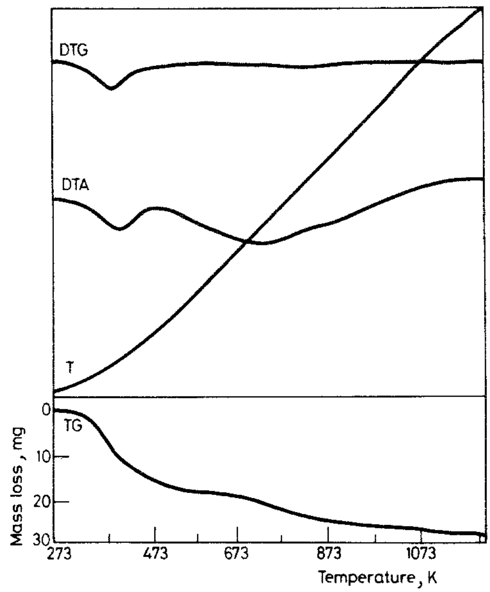

Fig. 2 TG, DTG and DTA curves of a partly decomposed product obtained by roasting at 630 K; heating rate 10 degree/min

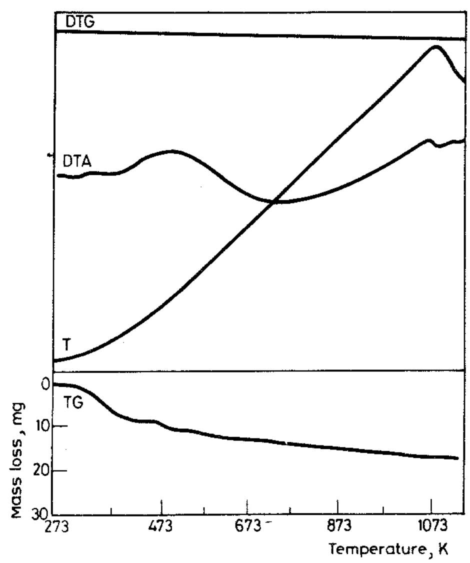

Fig. 3 TG, DTG and DTA curves of a partly decomposed product obtained by roasting at 800 K; heating rate 10 degree/min

Table 1 X-ray diffraction analysis of starting hydrated aluminium oxide and partly decomposed products

Starting hydrated aluminium oxide	Bayerite standard [4]		Boehmite standard [4]		Partly decomposed hydrated aluminium oxide at temperatures:				γ-Al_2O_3 [4]		α-Al_2O_3 [4]	
					553 K	633 K	803 K	1273 K				
d, Å	d, Å	I	d, Å	I	d, Å	d, Å	d, Å	d, Å	d, Å	I	d, Å	I
4.72	4.72	100	6.11	100	6.37	6.45		3.50	2.41	6	3.48	74
4.36	4.36	79	3.16	65	3.16	2.40	2.39	2.82	1.98	10	2.55	92
3.20	3.19	25	2.34	53	1.85	1.98	1.98	2.54	1.95	6	2.37	42
3.16	3.08	1	1.98	2	1.43	1.39	1.39	2.36	1.39	10	2.16	1
2.34	2.69	3	1.86	32				2.30	1.14	3	2.08	100
2.21	2.45	3	1.85	27				2.15			1.74	43
1.85	2.34	6	1.77	6				2.08			1.60	81
1.72	2.21	67	1.68	13				1.95			1.51	7
1.43	1.71	26	1.43	9				1.74			1.40	32
1.39	1.45	7	1.39	6				1.59			1.37	48
								1.50			1.29	16
								1.37			1.04	13
								1.04				

Correlations between peak temperatures, particle size and degree of crystallinity were confirmed by other authors investigations on the thermal decomposition of a great number of synthetic boehmites.

For instance, a synthetic boehmite sample with a small particle size and possibly a low degree of crystallinity gives a peak at about 723 K, compared with about 843 K for a sample with a large particle size and a high degree of crystallinity [2].

References

1 S. Bretsznajder, Przem. Chem., 42 (1963) 677.

2 R. C. Mackenzie, The Differential Thermal Investigation of Clays, Mineralogical Society, London, 1957.

3 B. C. Lippens, Structure and Texture of Aluminas, Delft University of Technology, The Netherlands, 1961.

4 Powder diffraction file search manual. Alphabetical listing inorg. 1973 Joint Committee on Powder Standards, Pennsylv., USA.

Zusammenfassung — Die Zusammensetzung eines hydratisierten Aluminiumoxids wurde bestimmt und der Verlauf der thermischen Zersetzung dieses Oxids untersucht. Die Dissoziationsstufen wurden ermittelt und alle gasförmigen und festen Zwischenprodukte der Zersetzung identifiziert. Es wurde eine Korrelation zwischen den Peak-Temperaturen für Böhmit und der Partikelgröße und dem Kristallisationsgrad beobachtet.

Резюме — Определен состав гидратированной окиси алюминия и изучен процесс ее термического разложения. Установлены стадии диссоциации и идентифицированы все твердые и газообразные промежуточные продукты реакций разложения. В случае боэмита наблюдалась корреляция между температурными пиками и размером частиц, а также и степенью кристалличности.