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Hydrothermal Preparation of Haematite from Amorphous Iron(III) Hydroxide

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The hydrothermal preparation of haematite ($\alpha\text{-Fe}_2\text{O}_3$) from amorphous iron(III) hydroxide was studied in acid solutions at 180°C . An investigation by Christensen¹ demonstrated that the pH of the mother liquid is decreased by a hydrothermal treatment of freshly precipitated iron(III) hydroxide when the precipitation of ferric hydroxide is interrupted at pH = 5 or at lower pH values. The decrease in pH is explained by hydrolysis, resulting in a complete precipitation of the iron. In the present investigation the rate of the decrease in pH was investigated.

A solution of 0.1 M ferric nitrate was used in all the experiments. 5 ml of the solution was titrated with a solution of 0.25 M ammonia, as reported in Ref. 1. The titration was interrupted at pH = 4.50

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Table 1. Experimental conditions for the preparation of haematite from amorphous iron(III) hydroxide.

Expt. No.	Time of treatment h	pH of mother liquid after treatment	X-ray investigation of product
1	0.3	4.59	amorphous
2	0.5	3.20	»
3	0.5	3.15	»
4	1	2.70	$\alpha\text{-Fe}_2\text{O}_3$
5	1	2.43	»
6	1.25	2.50	»
7	1.5	2.44	»
8	1.75	2.43	»
9	2	2.40	»
10	2	2.44	»
11	2	2.41	»
12	4	2.41	»
13	4	2.41	»
14	8	2.41	»
15	16	2.38	»

and the precipitated ferric hydroxide with the mother liquid was transferred to a thick-walled pyrex ampoule. The sealed ampoule was heated in a thermostated oven kept at $180 \pm 1.5^\circ\text{C}$. After the hydrothermal experiment the pH of the mother liquid was measured, and the reaction product was washed with water and dried at 25°C . The X-ray powder pattern was obtained with a Philips powder diffractometer. The results are given in Table 1.

The investigation shows that the decrease in the pH of the mother liquid from pH = 4.5 to 2.4 takes approximately 1.5 h. Further treatment does not result in a significant decrease in pH. The product obtained after heating for 1 h is crystalline $\alpha\text{-Fe}_2\text{O}_3$. The diffractometer powder patterns show, that there is no detectable increase in the grain size of $\alpha\text{-Fe}_2\text{O}_3$ for samples treated for a longer period than 2 h.

The hydrothermal formation of crystalline $\alpha\text{-Fe}_2\text{O}_3$ from amorphous iron(III) hydroxides is in the present investigation completed within a period of 1 to 2 h. $\alpha\text{-Fe}_2\text{O}_3$ prepared by the hydrothermal method is possibly a useful material in the preparation of $\gamma\text{-Fe}_2\text{O}_3$ for magnetic tapes.

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