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EFFECT OF MECHANOCHEMICAL SYNTHESIS CONDITIONS ON OBTAINING NANOSIZED MAGNETITE POWDERS

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The paper investigates the possibility of synthesis of nanosized magnetite powder by mechanochemical activation without the addition of the inert diluent NaCl to the reaction mixture. Physical and chemical properties of the product obtained are also investigated.

Magnetite nanopowder was prepared according to the reaction:

$$FeSO_4 \cdot 7H_2O + 2FeCl_3 + 8NaOH = Fe_3O_4 + Na_2SO_4 + 6NaCl + 11H_2O$$

The synthesis was performed in an APF-3 planetary mill (60 g acceleration) with a steel ball (d=5 mm)-to-powder weight ratio equal to 20:1. Samples were obtained at activation times of 30 s, 1, 2.5 and 5 min. The phase composition, morphology, dispersion, and structure of the synthesized powders were studied by X-ray diffraction analysis, and their specific surface was measured. Intermediate compounds formed on the surface of the particles during the reaction were studied by infrared spectroscopy.

X-ray diffraction analysis of the powders showed the pronounced reflections of the reaction products (sodium chloride and Disodium tetraaquabis(sulfato)iron(II) [$FeNa_2(SO_4)_2(H_2O)_4$]) on the X-ray diffraction pattern after 30 s activation of the reaction mixture [1]. This confirms an exchange reaction in the activator, and one of the reaction products immediately crystallizes. According to the data obtained, the washing of powders after mechanical activation leads to the destruction of Disodium tetraaquabis(sulfato)iron(II) and the formation of magnetite and hematite phases. X-ray spectra show a certain amount of substance in the X-ray amorphous state.

The specific surface of magnetite powders changes nonlinearly with increasing the activation time (Table 1). During the first minute, the specific surface of the powder sharply increases up to 115 m^2/g and then decreases to 103 m^2/g. This fact is likely to be related to the processes of disordering and aggregation of particles in the milling device under intense plastic deformation (impact, friction). Further intense treatment leads to disaggregation of the powder and the increase in the specific surface at 5 min mechanical activation. As shown in [2], the specific surface of magnetite powder synthesized at 30 min activation is 150 m^2/g.

Table 1. Specific surface and average particle size of magnetite powders

$\tau_{activation}$	S_{spec}, m^2/g	D, nm
30 s	115	9.9
1 min	168	6.8
2.5 min	103	11.1
5 min	132	8.7

A comparison of the infrared spectra of magnetite powders synthesized at different activation times shows the presence of goethite (-FeOOH) on the surface, which is indirect evidence of the participation of crystallization water of the initial compound $FeSO_4 \cdot 7H_2O$ in the synthesis reaction.

The results show that a change in the synthesis procedure as compared to that presented in [2], namely, mechanochemical activation in the absence of the inert diluent NaCl, leads to obtaining magnetite nanoparticles with an average particle size of 8 nm with a large specific surface.

REFERENCES

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