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Influence of Amount of Iron Oxide and Temperature of Synthesis on Their Particle Size in Composites with Bentonite

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Composites structured of iron oxide nanoparticles and clay mineral matrices are synthesized and studied because of their interesting properties [1-4]. The clay-iron oxides magnetic materials could be used as adsorbents [5-6] and they are also tested as a perspective MRI contrast agent [7].

Composites of iron oxide particles on the surface of monomineral fraction of natural bentonite from Slovak province *Jelšový potok* have been prepared by the method of precipitation from the solution of ferrous and ferric salts in the presence of powdered mineral. Three different weight ratios bentonite to iron oxide were chosen: 1:1, 4:1 and 5:1 and two temperatures of synthesis in a water bath were selected: 20 and 85 °C.

The composite materials have been studied by XRD, Mössbauer spectroscopy, FE-TEM and SQU-ID. Influence of the conditions of composites preparation on the size of the iron oxide particles has been proved. The prepared composite materials differ in size, morphology and phase of the iron oxide particles. Synthesis at higher temperature (85 °C) allowed to obtain only the magnetic phase, γ -Fe₂O₃ (maghemite), while in materials prepared at 20 °C, except the maghemite, presence of oxyhydroxide phase – goethite (α -FeOOH) was observed. Comparison of broadenings of hyperfine splittings of maghemite in octahedral position (or goethite hyperfine splittings, Fig. 1) in composites prepared at the same temperature showed, that the iron oxide particles are the smallest in the samples with the lowest iron oxide content. This feature was more expressive for composites prepared at 20 °C where superparamagnetic properties of the sample with a weight ratio of 5:1 appeared.

In this sample the biggest amount of very fine particles was observed FE-TEM. Except for the spherical shape of maghemite particles, needle forms, probably belonging to α -FeOOH, were detected too.

Composite samples exhibited also good magnetic properties. In case of samples synthesized at 20 °C the mass magnetization was partially decreased due to the presence of goethite but they could be

still utilized, *e.g.*, for magnetic separation of composites used as sorbents from the medium after the sorption process.



Fig. 1 Comparison of hyperfine splittings of goethite in composite samples with weight ratio 1:1 (1) and 4:1 (2) synthesized at 20 $^{\circ}$ C.

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