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## Synthesis Features of Iron Oxide Nanopowders with High Magnetic and Sorption Properties

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**Abstract.** The magnetic particles of iron oxides are promising materials for the purification of water from ions of heavy metals and radionuclides. Their advantage compared to other sorbents is the ability to extract by applied magnetic field, which greatly simplifies the task of extraction, separation and processing in cleaning technologies. The aim of this work is investigation of temperature and concentration of iron in the solution effect on the phase composition, nanoparticle size and their magnetization.

Phase magnetite in the sample increases with increasing temperature and the magnetization decreases slightly with increasing the initial concentration of iron in solution. We found that regardless of the conditions of deposition formed spherical particles whose average size ranges from 7 to 15 nm. The sorptive capacity of the particles is virtually independent of the phase composition and for cobalt is about 18 mg/g. For sorption-based material magnetic particles Fe<sub>3</sub>O<sub>4</sub> recommended to carry out the deposition process at a temperature not lower than 80°C. The concentration of iron in solution must be within 0,15–0,3M. The particles obtained contain in their composition at least 90 wt.% of magnetite phase and are characterized by a magnetization in the range of 65–70 A·m<sup>2</sup>/kg. Also in the paper is comparing efficiency of extraction and sorption capacity for cobalt particles by different phase of magnetite and hematite.

### Introduction

Sorption extraction is one of the most effective and simple methods of purification of natural and waste waters from heavy metals and radionuclides [1, 2]. Polymers [3], natural materials, oxides [4] were proposed to use as sorbents. In the latter group of materials iron oxides should be noted as components of effective sorption materials. Their advantage over other compounds consists in low cost of precursors, simplicity of synthesis, and in the fact that they exert no toxic effects on the human body [5].

Typically, the particles of iron oxide are produced by deposition from aqueous solution with a subsequent heat treatment at 200–600°C. [6, 7].

It is well known that the functional properties of materials are largely determined by particle size and shape. For example, increasing the specific surface area of the sorbent leads to an increase in its sorptive capacity. Thus, in case of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> of a specific surface of 130 m<sup>2</sup> g<sup>-1</sup> the sorption capacity (at pH 3) for arsenic(V) and chromium(VI) was 51 and 30 mg g<sup>-1</sup>, respectively. A decrease in the specific surface area of up to 2 m<sup>2</sup> g<sup>-1</sup> led to a decrease in the sorption capacity of up to 0.30 and 0.37 mg g<sup>-1</sup>, respectively [8].

Thus, an issue of the effect of the conditions of particles producing on their morphological and functional characteristics is a topical. The nature of the anions is one of the factors exerting a significant effect on the formation of particles in deposition from aqueous solutions [9-12].

## Experimental Procedure

### *Synthesis of Fe<sub>2</sub>O<sub>3</sub> particles*

Synthesis of Fe<sub>2</sub>O<sub>3</sub> particles as follows: 100 ml of a 0.2M precursor salts of iron (III) an aqueous solution of ammonia or ammonium bicarbonate to a predetermined pH ranging from 7 to 12 and stirred on a magnetic stirrer for 30 min. The resulting precipitate was filtered, washed several times with distilled water and dried at room temperature for 24 h, then calcined at 450°C for 1 hour.

### *Synthesis of Fe<sub>3</sub>O<sub>4</sub> particles*

Synthesis of Fe<sub>3</sub>O<sub>4</sub> particles was performed by precipitation from aqueous solution: 5.99 g FeSO<sub>4</sub>•7H<sub>2</sub>O and 10.79 g of FeCl<sub>3</sub>•6H<sub>2</sub>O was dissolved in 40 ml of distilled water. The resulting solution was heated to 60°C and 10 ml of 25% aqueous ammonia solution under constant magnetic stirring. The resulting precipitate was filtered and washed with distilled water. Then dried in air for 24 hours.

### *The sorption properties*

Sorption is carried out in monocomponent heavy metal solution at pH 6.5 with concentration of 10–250 mg/l. In a 100 ml beaker the 50 ml of model solution was adjusted by addition of nitric acid or aqueous ammonia solution to a predetermined pH 6.5. Solution pH was measured by the "150 pH" pH-meter with glass electrode ESL-63-07 and the auxiliary silver chloride electrode EVL1MZ. Then was added 0.1 g of the adsorbent and kept under stirring for 40 minutes. The process of sorption was performed every 10 minutes with pH control in the case of deviations exceeding 0.1 pH units, respectively adjusted to predetermined values. The sorbent was filtered after sorption and the content of metals in the filtrate was determined.

## Results and Discussion

X-ray analysis of the sample Fe<sub>2</sub>O<sub>3</sub> showed that after annealing at 450°C formed *α*-Fe<sub>2</sub>O<sub>3</sub> (hematite). Unlike samples Fe<sub>2</sub>O<sub>3</sub>, for Fe<sub>3</sub>O<sub>4</sub> samples X-ray data is multiphase and contain magnetite (Fe<sub>3</sub>O<sub>4</sub>) – 70%, maghemite (*γ*-Fe<sub>2</sub>O<sub>3</sub>) – 25% and goethite (FeOOH) – 5%. The presence of impurity phases goethite and maghemite Fe<sub>3</sub>O<sub>4</sub> samples associated with aging and is characteristic of the powders obtained from aqueous solutions.

Fig. 1 shows the photomicrographs of particles (a) Fe<sub>2</sub>O<sub>3</sub> and (b) Fe<sub>3</sub>O<sub>4</sub>, obtained from aqueous solution and annealed at 450°C.

It is established that from nitrate solutions formed shapeless large agglomerates of 50 – 200 microns (Fig. 1a), consisting of spherical particles of Fe<sub>2</sub>O<sub>3</sub> (specific surface S<sub>sp</sub> = 150 m<sup>2</sup>/g). Introduction of chloride ions leads to the formation of solid particles of cubic and oval size of about 0.12 – 0.20 microns (S<sub>sp</sub> = 30 m<sup>2</sup>/g). Sodium ion has no significant effect on the formation of particles Fe<sub>2</sub>O<sub>3</sub>.

Unlike Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub> is not observed in the formation of large agglomerates and the powder is composed of spherical with a size of from 0.5 μm to several microns. The specific surface area of Fe<sub>3</sub>O<sub>4</sub> was 130 m<sup>2</sup>/g.

Fig. 2 shows the degree of extraction of metals from the pH of the particles Fe<sub>2</sub>O<sub>3</sub> (Fig. 2(a) and (c)) and Fe<sub>3</sub>O<sub>4</sub> (Fig. 2(b) and (d)).

As can be seen from Fig. 2, iron oxide particles effectively recovered europium (97%), Ce (97%) and copper (92%). However, in an acidic medium using Fe<sub>2</sub>O<sub>3</sub> particles as sorbents can achieve high degrees of recovery of these metals as compared with Fe<sub>3</sub>O<sub>4</sub> particles.

The maximum extraction for Fe<sub>2</sub>O<sub>3</sub> is observed at pH 5.5, and for Fe<sub>3</sub>O<sub>4</sub> is optimal pH 6.5. Efficiency of extraction of cobalt and strontium increases with increasing pH and reached maximum values at pH 9.

The highest efficiency of extraction occurs on the particles of Fe<sub>3</sub>O<sub>4</sub> and 95% to 68% cobalt, and strontium.

Furthermore, for strontium in the pH range 4 – 7, depending observed additional peak recoveries in the range of pH 5.2 – 5.7, which is more pronounced on Fe<sub>2</sub>O<sub>3</sub> particles, see Fig. 2(a).

The results of calculation by Langmuir's method shown in Table 1. As can be seen from the data, the size of the sorption capacity Fe<sub>2</sub>O<sub>3</sub> particles with 150 m<sup>2</sup>/g specific surface for all investigated metals are higher than the sorbents based on Fe<sub>3</sub>O<sub>4</sub> with 130 m<sup>2</sup>/g specific surface.

Table 1 Parameters of sorption isotherms of europium, cerium and copper particles Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>

Metal	Fe <sub>2</sub> O <sub>3</sub>			Fe <sub>3</sub> O <sub>4</sub>		
	K	A <sub>∞</sub> , [mg/g]	R <sup>2</sup> , [%]	K	A <sub>∞</sub> , [mg/g]	R <sup>2</sup> , [%]
Eu	0.007	21.3	89.85	0.024	19.7	92.97
Ce	0.346	9.2	94.83	0.162	7.5	90.52
Cu	0.029	15.7	94.12	0.039	11.66	93.57

The highest sorption capacity is observed for the europium – 21.3 mg/g and 19.7 mg/g on the particles of Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>, respectively, and the lowest - cerium on the particles of Fe<sub>2</sub>O<sub>3</sub> (9,2 mg/g) and Fe<sub>3</sub>O<sub>4</sub> particles (7.5 mg/g).

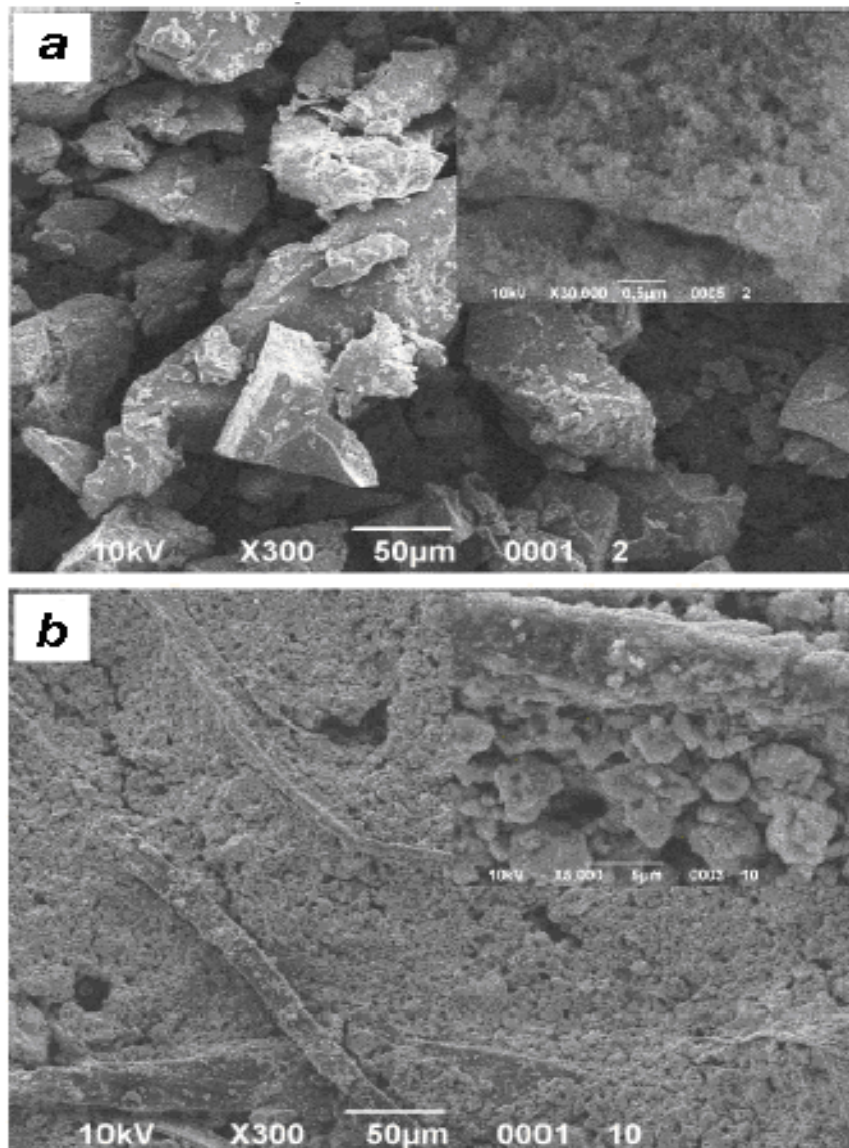


Fig. 1 Micrographs of particles (a) Fe<sub>2</sub>O<sub>3</sub>, (b) Fe<sub>3</sub>O<sub>4</sub> annealed at 450°C

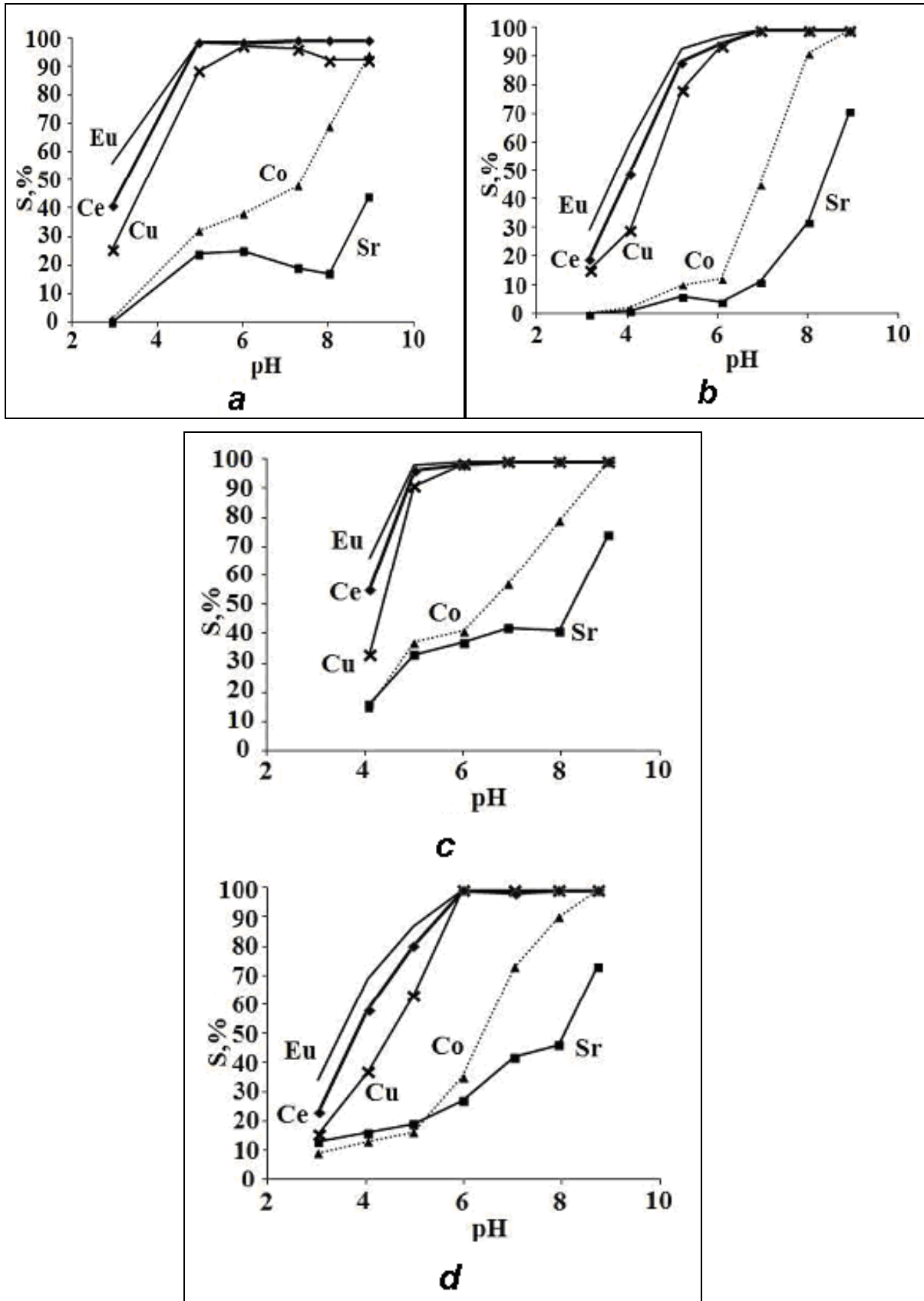


Fig. 2 Dependence of the degree of extraction of metals from the pH of the particles: (a) and (c)  $Fe_2O_3$ ; (b) and (d)  $Fe_3O_4$

## Summary

(a) The effect of the deposition conditions (temperature, concentration of iron in solution) on the phase composition, particle size and magnetization. A comparison of the efficiency of extraction and sorption capacity of heavy metals in phase magnetite and hematite.

(b) It was found that the formation of magnetite phase is significantly affected by the deposition temperature. The higher the temperature, the greater the amount of magnetite phase in the sample. Carrying out the synthesis at 90°C in a 0.15M solution promotes formation of iron powder containing phase magnetite (Fe<sub>3</sub>O<sub>4</sub>) 100 wt.%

(c) Regardless of the deposition conditions form spherical particles having an average size ranging from 7 to 15 nm.

(d) Raising the deposition temperature and reducing the concentration of iron in the solution contributes to obtaining a powder with larger particles that are characterized by higher value of magnetization.

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