Utjecaj dodatka biljnog materijala u procesu mikrovalne sinteze željezovih oksida

Influence of the addition of plant material in the microwave-assisted synthesis of iron oxides

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Introduction

Considering the increasing awareness of environmental protection, there is a growing interest in ecological synthesis methods that reduce the use of harmful chemicals and replace them with natural substances. The following results demonstrate a new green approach for the synthesis of nanoparticles, using plant extracts. Although magnetite is usually synthesized from ferrous and ferric cations in a ratio of 1:2, the use of plant materials enabled magnetite synthesis from ferric cations only. The influence of the addition of extracts of *Lavandula spica L*. and *Spartium junceum L*., as well as the influence of different experimental conditions (synthesis at 200 °C for 20 min and 260 °C for 5 min) on the final iron oxide product phases were studied. The ecological effect is achieved not only by reducing chemicals and adding plant material, but also by using water as a solvent and a microwave oven, which reduces the synthesis time from several hours to a few minutes. All the obtained products were analyzed by Fourier infrared spectroscopy (FTIR), powder X-ray diffraction (PXRD) and field emission scanning electron microscope (FE-SEM).

Experimental

The process of synthesis and the influence of the added plant material extract began with the preparation of the extracts of *Lavandula spica L*. and *Spartium junceum L*.. The plant material was grind and 20 g were added to 200 mL of water and heated to 60 °C, keeping the temperature constant for 20 minutes. After filtration and centrifugation, the obtained extract was placed in a Teflon vessel together with the iron precursor in highly alkaline media under continuous microwave emission of 850 W at different conditions listed in the Table 1.

Table 1. Experimental data of magnetitte nanoparticles synthesis methods

Sample	1 M $FeCl_3$ /mL	8 M NaOH /mL	H_2O /mL	Lavandula spica L. /mL	Spartium junceum L./mL	T /°C	t /min
RS1	4	4	32	/	/	200	20
S1	4	4	/	32	/	200	20
S4	4	4	/	/	32	200	20
RS2	4	4	32	/	/	260	5
S2	4	4	/	32	/	260	5
S3	4	4	/	/	32	260	5

Results for the samples synthesized at 200 °C for 20 min.

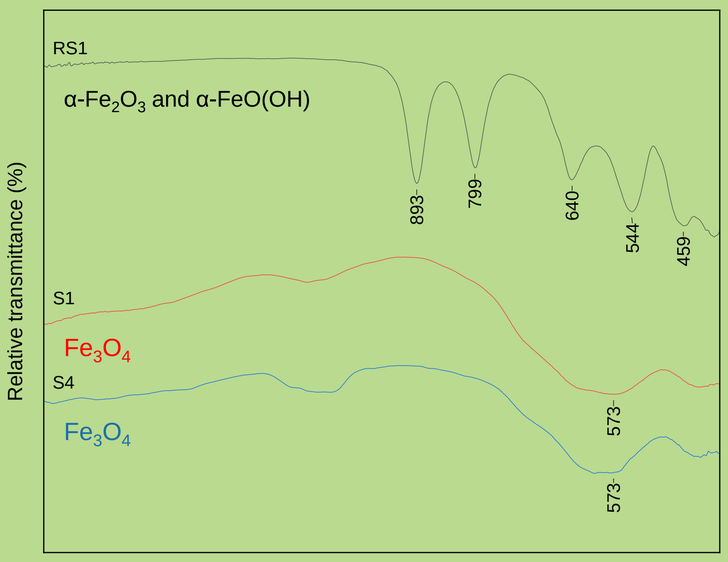

Figure 1. FTIR spectra for the samples synthesized at a temperature of 200 °C for 20 min.

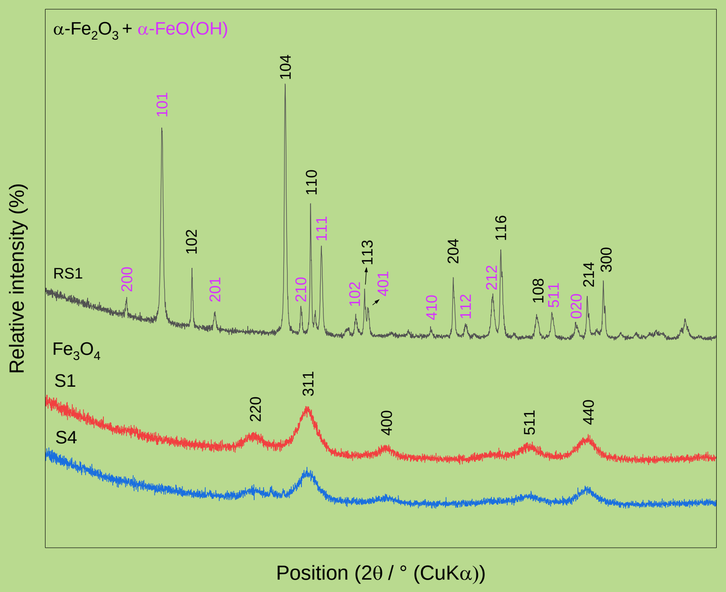

Figure 3. PXRD patterns for the samples synthesized at a temperature of 200 °C for 20 min

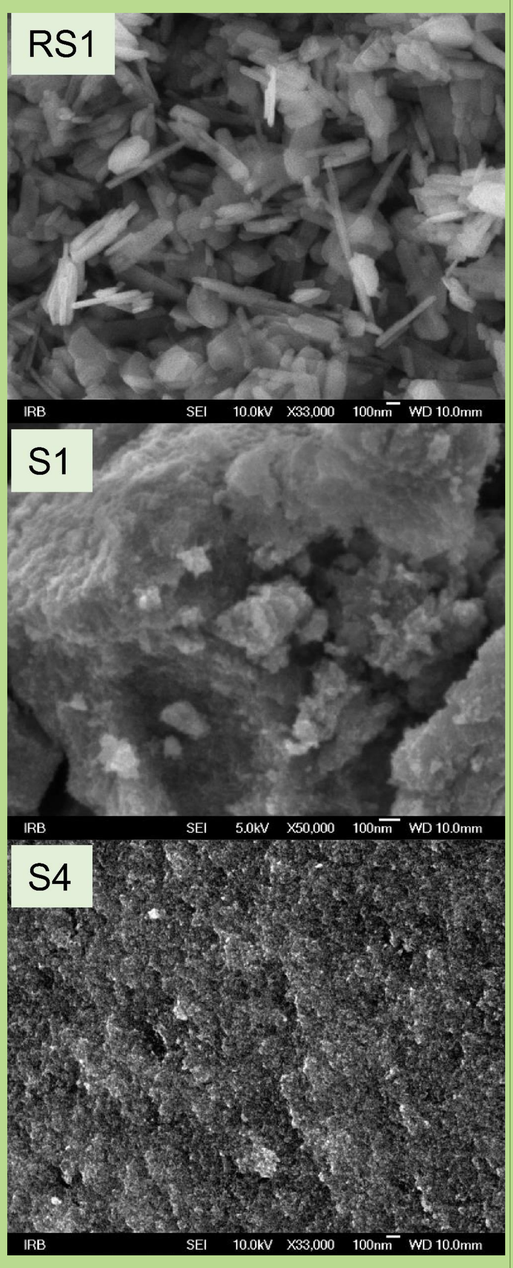

Figure 4. FE-SEM images for the samples synthesized at a temperature of 200 °C for 20 min.

Results for the samples synthesized at 260 °C for 5 min.

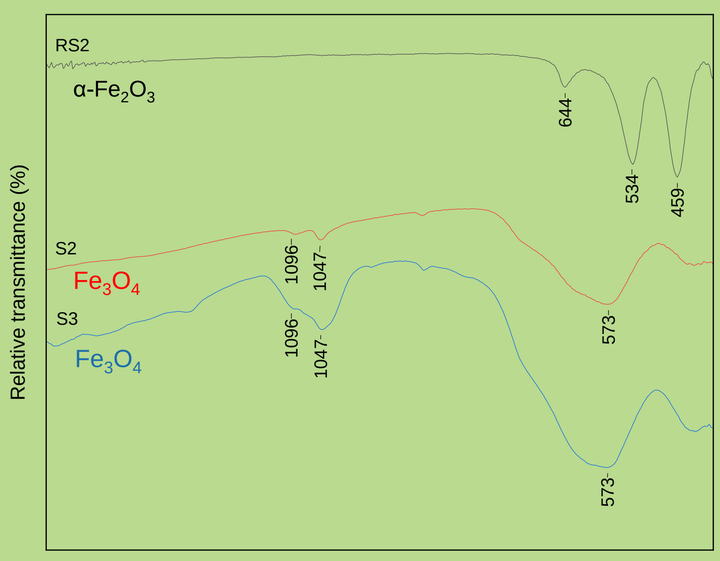

Figure 2. FTIR spectra for the samples synthesized at a temperature of 260 °C for 5 min.

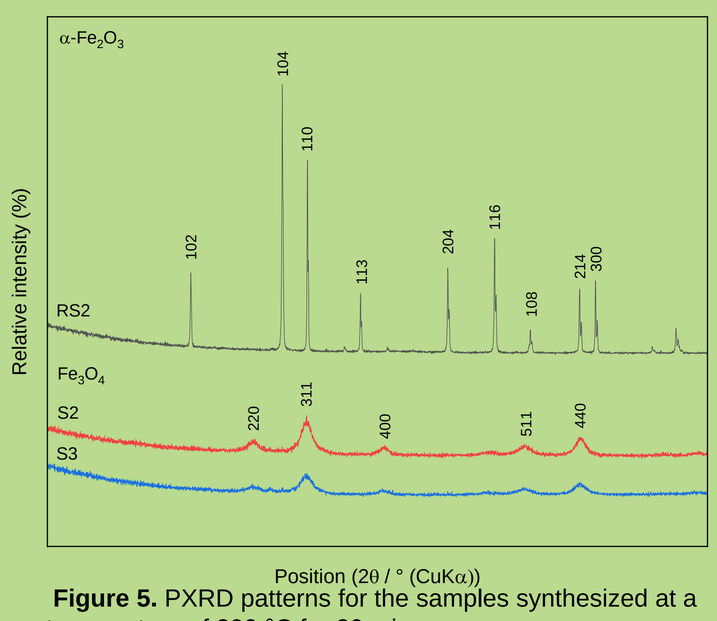

Figure 5. PXRD patterns for the samples synthesized at a temperature of 200 °C for 20 min.

Figure 6. FE-SEM images for the samples synthesized under the temperature of 200 °C for 20 min

Based on the FTIR and PXRD spectra of reference samples and the samples synthesized with the extracts of *Lavandula spica L*. and *Spartium junceum L*., their completely different composition was found. The reference sample synthesized at 200 ˚C is a mixture of goethite and hematite phases, while the samples synthesized under the same conditions but with the addition of plant materials have a magnetite phase. The same effect of plant material is obtained when the synthesis under higher temperature, while the reference sample synthesized under 260 °C was pure hematite. The FE-SEM images of the RS1 sample showed the presence of hematite plates and rod-shaped goethite, while in the RS2 sample only hematite plates are seen, and samples with extract addition show irregularly shaped magnetite particles.

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