SYNTHESIS OPTIMIZATION AND CHARACTERIZATION OF Fe OXIDE NANOPARTICLES

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The paper discusses the results of researches carried out to define the optimal conditions of synthesis of Fe oxide nanoparticles using a simple and low-cost method. In addition, the results derived from XRD and IR spectroscopy analyses conducted for the characterization of the obtained nanoparticles are provided.

Keywords: Fe_2O_3, sensor, sol-gel, nanoparticles.

INTRODUCTION

A large number of researches have been devoted to the study of the electrical and optical properties of nanostructured materials during the last decades. Special optical and electronic properties were defined as compared to those of bulk materials. The study of such materials is interesting not only from a fundamental scientific viewpoint, at the same time they have been applied in a wide range of our lives. This is due to their electronic structure, chemical and mechanical stability, and the sensitivity to the ambient conditions.

Metal-oxide nanoparticles (NPs) are among the most used nanostructured materials (NMs). The metal oxide NPs (ZnO, TiO_2, Ga_2O_3, SnO_2, NiO and CuO, etc.) have been effectively applied as optical and electrical-resistive sensors [3-6]), catalysts [7] due to their strong absorption properties, large surface area, high porosity and good permeability [8,9].

Transition metal oxides RuO_2, MnO_2, CuO, NiO, and Fe_2O_3 [10-15], which have excellent redox reversibility, high theoretical specific capacitance [10] were successfully applied in supercapacitors as electrodes [11,12], potential anode materials for Li-ion batteries, mainly due to their excellent stability and safety features [13]. From this end of view, NPs of NiO and Fe_2O_3 have attracted considerable attention. Fe_2O_3 is also attractive for its low cost, abundance, nontoxicity, and eco-friendliness [14,15].

There are different ways of the synthesis of Fe_2O_3 and NiO NPs, such as spray pyrolysis [16,17], hydrothermal [18], Sol-gel [19] methods. Both the synthesis methods and costs of the starting materials significantly influence the end material cost, which are important in the developing and commercialization of the fabricated device.

In this work, we presented the results of the researches carried out to develop a simple and cost-effective method for the synthesis of Fe oxide NPs. The resulting powdery nanomaterial was characterized by XRD, Raman, and IR spectroscopy.

EXPERIMENTAL

Nanosize particles of iron oxide (Fe_2O_3) are synthesized by a simple Sol-gel method. 0.1 M $FeCl_3$:$6H_2O$ was dissolved in 100 ml deionized water (Solution 1) and 5M sodium hydroxide (NaOH) was dissolved in 16 ml DI water (Solution 2). First, the solution 1 is stirred in 1500/min speed with heating. When the temperature of the Solution 1 was increased to 90 ºC, the Solution 2 was added to it dropwise under constant magnetic stirring during 1 h to maintain the pH at 7 and the obtained solution was stirred constantly for 1 h until the temperature was decreased to 25ºC. The obtained precipitate was recovered by centrifugation, washed several times with DI water to remove chloride and other unreacted ions, and then dried in the open air at 150 ºC. Finally, the samples were annealed at 300 and 500ºC for 3 h in the open air to obtain dark reddish colored powders Fe_2O_3 nanoparticles from $Fe(OH)_3$ according to the reaction:

$$2Fe(OH)_3 \xrightarrow{t} Fe_2O_3+3H_2O.$$

All the samples are characterized by X-Ray diffraction method for determination crystallinity and formatted phases and the particle size using D2 Phaser diffractometer from Bruker with CuKα radiation of wavelength 1.5406 Å. IR spectroscopy methods were used for using Irrafinity 1 FRIR spectrometer from Shimadzu Japan in 2,5 – 25 mkm of wavelength range

RESULTS

X-Ray diffraction analysis has been performed to identify the phase formation and crystallinity of the obtained powder like Fe-O compound by two calcination temperatures. Fig.1 shows that better crystallinity is obtained at 500ºC. The strong diffraction peaks are compared with Standard JCPDS card no.86-0550, which confirm the crystallization in rhombohedral hexagonal phase α-Fe_2O_3. The extra small peaks are explained with remained impurities. The average crystallite size was calculated using Sherrer's formula ($L= k*\lambda/\beta \cos\theta$, *where* β– is full width at half maximum), which is 10 nm.

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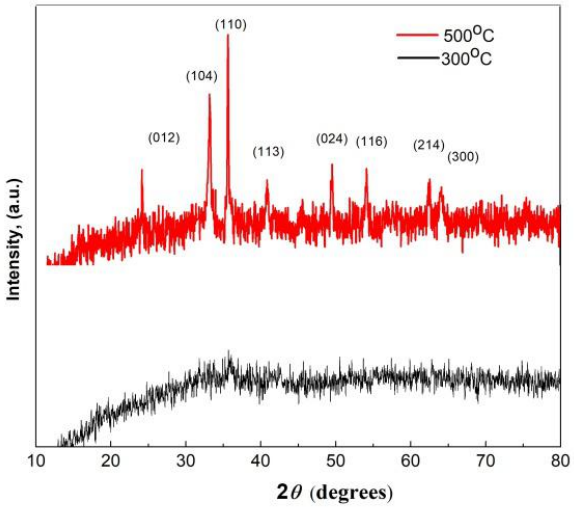

Fig.1. X-ray pattern of a- Fe_2O_3 NPs at different calcination temperatures.

IR SPECTROSCOPY ANALYSIS

Fig 2 shows IR spectra of Fe oxide in three different (300, 500 and 700^{0}C) calcination temperatures. The bands near 3409 cm^{-1}, 1624cm^{-1}, 1401 cm^{-1} and 600 cm^{-1} are observed in all spectra. The bands at about 540 cm^{-1} and 465-455 cm^{-1} are observed only in the samples calcinated at 500 and 700^{0}C.

The stretching vibration of the O-H group characterizes by wide band at 3409 cm^{-1}, and 1624cm^{-1} may be attributed to the benching vibration of the H-O-H group, and both due to physisorbed water molecules on the surface. The band at 1401 cm^{-1} corresponds to C–O–H stretching. The formation of α-Fe_2O_3 is proved by presence of the bands at about 540 cm^{-1} and 460 cm^{-1} in the calcined samples [20,21]. The band at 600 cm^{-1}, which is attributed Fe–O vibration shows presence of γ-Fe_2O_3 phase, while its characterized line (at 430 cm^{-1}) is not observed on the graphs, which require future analysis.

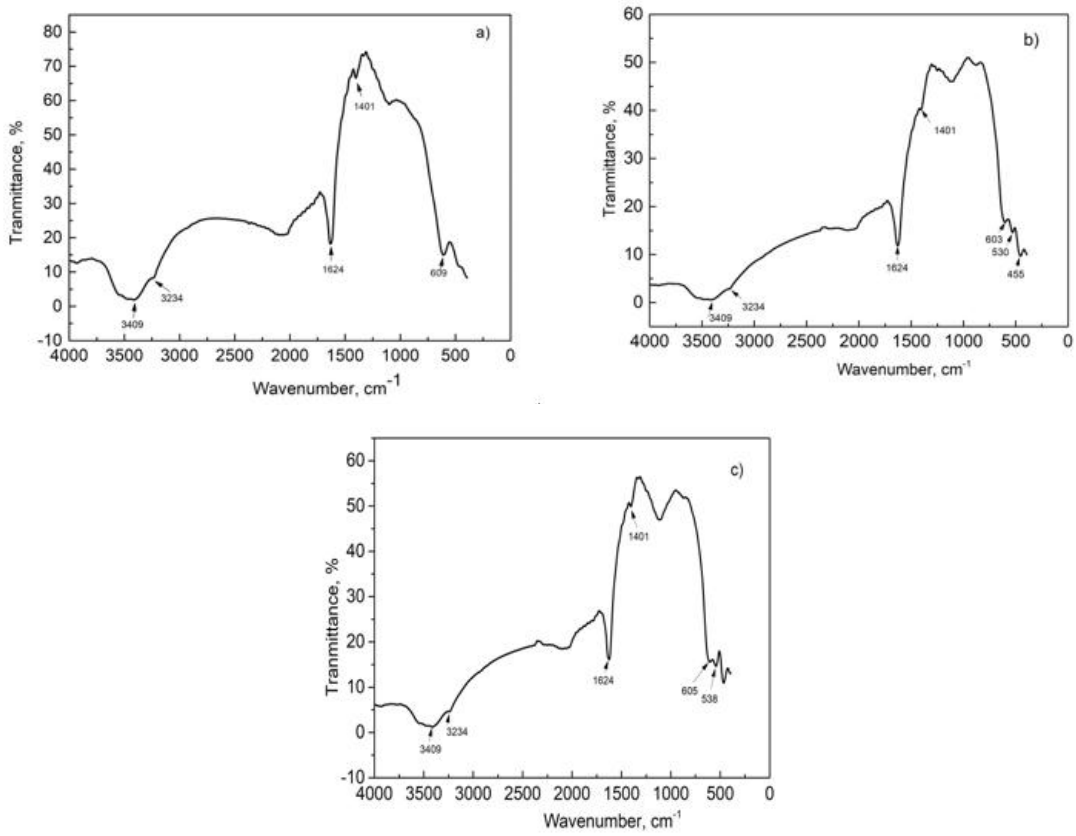

Fig.2. FT-IR spectra of Fe oxide nanoparticles calcined in different temperatures: (a) 300^{0}C; (b) 500^{0}C; (c) 700^{0}C .

CONCLUSION

Optimal and cost-effective method to synthesize of Fe oxide nanoparticles have been developed. Both analyzing X-ray diffraction and IR spectroscopy methods showed that α-Fe_2O_3 is formed, which is crystallization in rhombohedral hexagonal phase.

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