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Hydrothermal synthesis of Fe-Mn magnetic nanoparticles supported by Al_2O_3 and study the effect of calcination conditions

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Abstract
The purpose of this study was to explore the impacts of different calcination conditions as a parameter on the structures of Iron-Manganese oxide nanoparticles, which based on Al_2O_3 by using hydrothermal method. In present research different terms of times and high temperatures of calcination used to prepare the particles. The synthesized Nano-scale powder investigated by field-emission Scanning Electron Microscope (FESEM), X-Ray Diffraction (XRD) and Energy Depressive X-Ray (EDX or EDS). The observation showed the existence of various compound in samples such as Fe_2O_3, Fe_3O_4, $MnFe_2O_4$, Mn_2O_3, Mn_3O_4 and the crystals size was range between 20-30 nanometers with spherical morphology. Subsequently the results of surveys showed that the suitable time and temperature of calcination process to obtain the best structures of Fe-Mn magnetic nanoparticles in this research was in 1 hour and 700 □C.

Keywords: Al_2O_3, Iron-Manganese, Hydrothermal, Calcination

Introduction

Nano science is one of the most important research in modern science. Nanotechnology is beginning to allow scientists, engineers, chemists, and physicians to work at the molecular and cellular levels to produce important advances in the life sciences and health care[1-3]. Numerous chemical methods can be used to synthesize magnetic nanoparticles for medical imaging applications such as micro-emulsions, sol-gel syntheses, hydrothermal reactions, flow injection syntheses, and electrospray syntheses. In present research we used hydrothermal method to obtain the Fe-Mn magnetic nanoparticles. Hydrothermal synthesis can be defined as a method of synthesis of single crystals that depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in an apparatus consisting of a steel pressure vessel called an autoclave, in which a nutrient is supplied along with water. The use of nanoparticle [NP] materials offers major advantages due to their unique size and physicochemical properties. Because of the widespread applications of magnetic nanoparticles [MNPs] in biotechnology, biomedical, material science, engineering, and environmental areas, much attention has been paid to the synthesis of different kinds of MNPs[4-5]. Industrial applications of magnetic nanoparticles cover a broad spectrum of magnetic recording media and biomedical applications, for example, magnetic resonance contrast media and therapeutic agents in cancer treatment. Magnetic nanoparticles are a class of nanoparticle that can be manipulated using magnetic fields. Such particles commonly consist of two components, a magnetic material, often iron, nickel , manganese, cobalt, and a chemical component that has functionality. The magnetic nanoparticles have been the focus of much research recently because they possess attractive properties which could see potential use in catalysis including nanomaterial-based catalysts,[6] biomedicine [7] and tissue specific targeting,[8] magnetically tunable colloidal photonic crystals,[9] microfluidics,[10] magnetic resonance imaging,[11] magnetic particle imaging,[12] data storage,[13] environmental remediation,[14] Nano fluids,[15] and optical filters,[16] defect sensor and cation sensors[17].

Material and method

The two precursors used in this experiment were $Fe(NO_3)_3.9H_2O$, $Mn(NO_3)_2.9H_2O$, Na_2CO_3, and Nano Al_2O_3 from Merck company. First of all the salt of metals $Fe(NO_3)_3.9H_2O$ and $Mn(NO_3)_2.4H_2O$ solved an combined in hot distilled water on the stirrer and Nano alumina slightly added to solution contained the metals. After dissolving the reactants the titration with Na_2CO_3 began and the pH of solution controlled by pH meter. Na_2CO_3 is a precipitating agent and used to obtain the basic pH upper than 9 to procurement of nano scale particles. The reaction time (aging time) of precursors was about 1 hour in 80˚C and occurred in the autoclave area in 90˚C. After that the material dried in the oven at about 90 minutes and 120˚C to eliminate the exist water in the sample. Finally, the calcination process administered and samples calcined in various calcination times and temperatures. Table 1 determines the experiment conditions applied to preparation of these magnetic nanoparticles. The table provides the different conditions of calcination time and calcination temperatures, the particles size and the peak position of phases.

Table1. experiment conditions

samples	Particle size		Calcination Conditions		Peak pos.	B structure
	crystals XRD	nanoparticles FESEM	temperature	time		
A	36 nm	40nm	600◦C	1h	33.36	0.236
B	37 nm	42nm	600◦C	2h	33.58	0.177
C	38 nm	41nm	600◦C	3h	33.46	0.236
D	36 nm	44nm	600◦C	4h	33.49	0.236
E	24 nm	49nm	500◦C	1h	36.21	0.236
F	51 nm	46nm	550◦C	1h	35.90	0.236
G	41 nm	38nm	650◦C	1h	35.55	0.236
H	37 nm	21nm	700◦C	1h	36.03	0.177

Results and discussion

Different instruments and analysis used to characterize and investigate the properties (structure, size, morphology and phases) of prepared particles. The XRD patterns of all samples determined the existence of various phases such as Fe_2O_3, Fe_3O_4, $MnFe_2O_4$, Mn_2O_3, Mn_3O_4 and the average crystalline size of about 40 nm was obtained using Debye-scherrer formula. Also the structures of samples indicated in cubic and tetragonal form. Figure 1 shows the XRD pattern in one sample that denotes the high peak belongs to Fe_2O_3 phase in all samples.

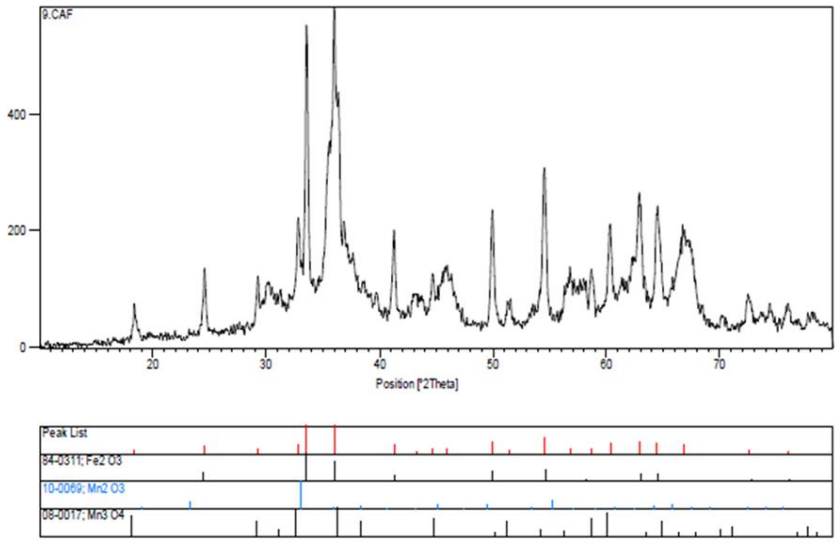


Fig1. XRD pattern of sample H

Figure 2 pertains to EDS analysis of sample H that indicates the existence of expecteble elements as Fe, Mn, O and Al in different molcular weights with no impurities. FESEM analysis in figure 3 and figure 4 display the image of nanoparticles in two different conditions actully in constant time and constant temperature of calcination and uphold the forming of nano scale particles in the samples.

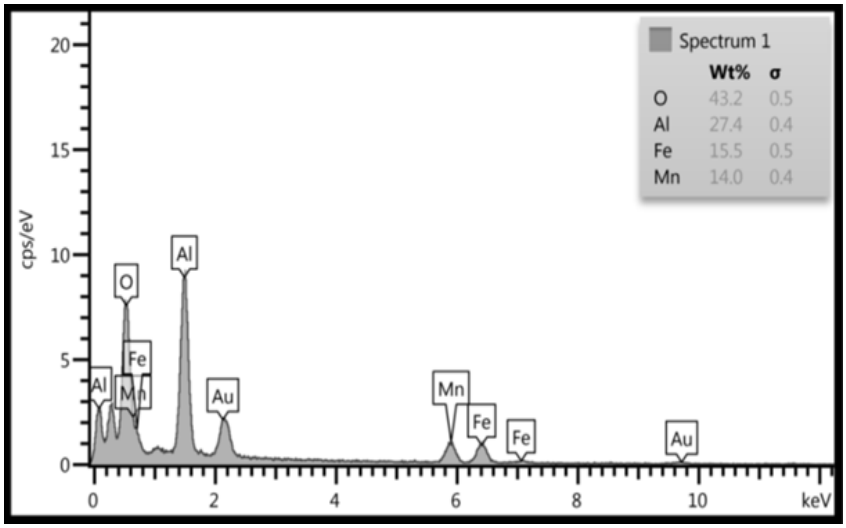


Fig2 EDS analysis of sample H

Fig3. FESEM analysis of sample C

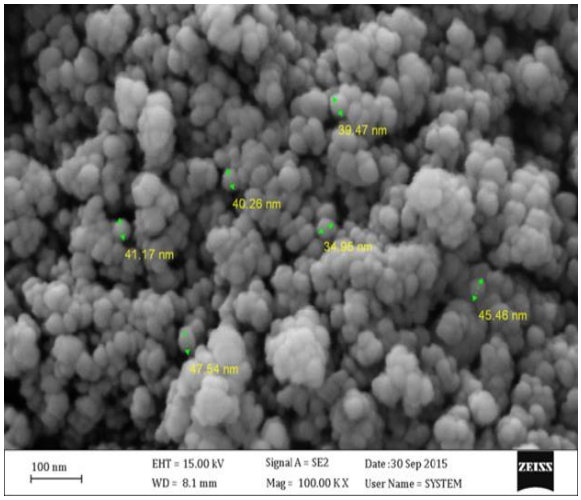
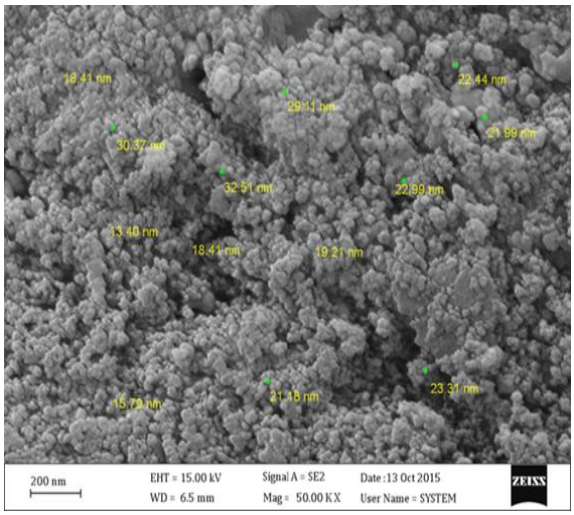


Fig4. FESEM analysis of sample H



Conclusion

Iron-Manganese oxide magnetic nanoparticles which based on Al_2O_3 support were successfully synthesized with hydrothermal method in different times and temperatures of calcination conditions in the stove area to heat in high temperatures. Various experimental techniques including X-Ray Diffraction (XRD), Field-Emission Scanning Electron Microscope (FESEM) and Energy Dispersive X-Ray (EDX) were used to characterize and investigate the samples. The results indicated that the nanoparticles were completely formed in cubic and tetragonal structures and average crystal size obtained about 40nm by Debye-scherrer formula.

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