Applied Surface Science 258 (2012) 3679–3688

Contents lists available at SciVerse ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Single step thermal decomposition approach to prepare supported γ-Fe_2O_3 nanoparticles

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ARTICLE INFO

Article history:
Received 21 September 2011
Received in revised form 1 December 2011
Accepted 2 December 2011
Available online 9 December 2011

Keywords:
Supported magnetic nanoparticles
Thermal decomposition method
Superparamagnetism

ABSTRACT

γ-Fe_2O_3 nanoparticles supported on MgO (macro-crystalline and nanocrystalline) were prepared by an easy single step thermal decomposition method. Thermal decomposition of iron acetylacetonate in diphenyl ether, in the presence of the supports followed by calcination, leads to iron oxide nanoparticles supported on MgO. The X-ray diffraction results indicate the stability of γ-Fe_2O_3 phase on MgO (macro-crystalline and nanocrystalline) up to 1150 °C. The scanning electron microscopy images show that the supported iron oxide nanoparticles are agglomerated while the energy dispersive X-ray analysis indicates the presence of iron, magnesium and oxygen in the samples. Transmission electron microscopy images indicate the presence of smaller γ-Fe_2O_3 nanoparticles on nanocrystalline MgO. The magnetic properties of the supported magnetic nanoparticles at various calcination temperatures (350–1150 °C) were studied using a superconducting quantum interference device which indicates superparamagnetic behavior.

1. Introduction

Iron oxide nanoparticles are interesting due to their wide range of applications in catalysis [1], biosensors [2], magnetic resonance imaging [3], bioprobes [4], ferrofluids [5,6], etc. The strong interparticle interactions among the magnetic nanoparticles and their tendency to form aggregates make them difficult for their applications. In order to avoid agglomeration and to stabilize the maghemite phase, non-magnetic supports have been used for the iron oxide nanoparticles which provide mechanical and chemical stability [7]. The supported iron oxide nanoparticles have applications in environmental remediation [8] and catalysis, e.g. growth of carbon nanotubes [9,10], decomposition and reduction of nitrous oxide and nitric oxide, partial oxidation of hydrocarbons and acid transformations of organic substrates [11–14]. Silica is the most commonly used support for the preparation of supported iron oxide nanoparticles [15–18]. Besides silica, other supports such as MgO [19,20], Al_2O_3 [21], CeO_2 [22], carbon [23], and ZrO_2 [24] have also been used for the preparation of supported iron oxide nanoparticles. The supported iron oxide nanoparticles have been prepared by several methods such as impregnation [25–28], co-precipitation [29,30], hydrothermal [31], sol–gel [32–34], thermal decomposition [35], etc.

MgO has been used as the support for the preparation of supported iron oxide nanoparticles due to its low specific weight and it provides high chemical and thermal stability to the magnetic nanoparticles. Some methods have been reported in the literature for the preparation of iron oxide nanoparticles supported on MgO, e.g. spray pyrolysis [19], ball milling [20], impregnation [36], wet chemical [37] and thermal decomposition followed by dispersion [9]. The reported methods often require longer reaction times (2–24 h), special conditions (e.g. H_2 atmosphere, vacuum annealing, etc.) and sometimes expensive equipment (e.g. ultrasonic spray process). The thermal decomposition followed by dispersion method, reported in the literature to prepare the supported iron oxide nanoparticles on MgO, requires higher reflux temperature (286 °C), and N_2 atmosphere [9]. The present study reports a quick thermal decomposition approach for the preparation of iron oxide nanoparticles supported on two different MgO (macro-crystalline and nanocrystalline MgO). The synthesis is carried out at lower temperature (~200 °C) without any need for special conditions. The effect of using macro-crystalline versus nanocrystalline MgO as the support for the stabilization of gamma iron oxide nanoparticles has not been reported so far and this effect has been investigated in the present study.

2. Experimental

2.1. Chemicals

Iron (III) acetylacetonate was received from Alfa Aesar®, macro-crystalline magnesium oxide and diphenyl ether were received

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doi:10.1016/j.apsusc.2011.12.005

Table 1
Crystallite size of supported iron oxide nanoparticles.

Calcination temperature (°C)	Crystallite size of supported iron oxide nanoparticles (nm)	
	On macro-crystalline MgO	On nanocrystalline MgO
900	34.3	18.7
1000	43.4	20.1
1150	63.7	35.9

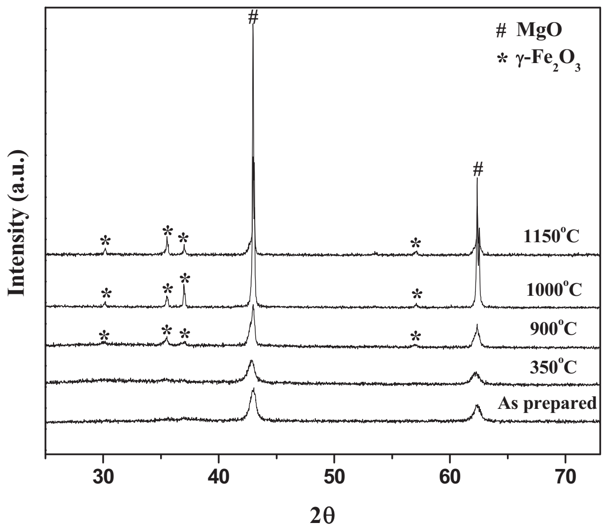

Fig. 1. XRD patterns for the as-prepared as well as calcined samples of iron oxide nanoparticles supported on macro-crystalline MgO.

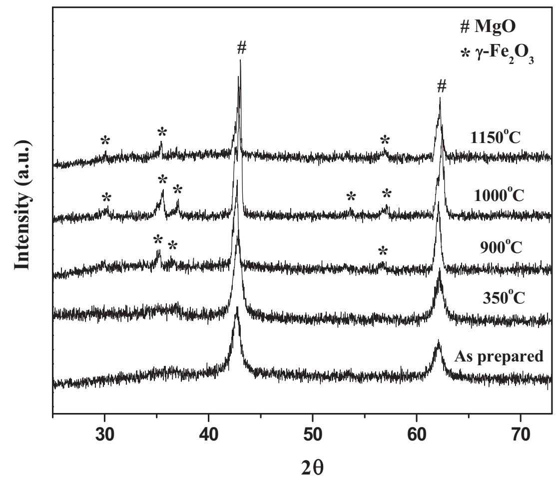

Fig. 2. XRD patterns for the as-prepared as well as calcined samples of iron oxide nanoparticles supported on nanocrystalline MgO.

from Himedia®, and methanol was received from Rankem®. All the chemicals were used without further purification.

2.2. Synthesis

Nanocrystalline MgO (surface area = 157 m^2/g) was synthesized by a previously reported method [38]. About 0.5 g of fresh Mg metal was reacted with 50 ml of methanol with stirring at room temperature to get magnesium methoxide suspension. Then a toluene: ethanol mixture (100 ml:40 ml) was added followed by the slow addition of 1 ml of water. The contents were stirred at room

Table 2
EDXA results on MgO supported iron oxide nanoparticles. The analysis was done on three separate areas for each sample.

Sample	Iron oxide nanoparticles supported on macro-crystalline MgO				Iron oxide nanoparticles supported on nanocrystalline MgO			
	% C	% O	% Mg	% Fe	% C	% O	% Mg	% Fe
	30.1	19.3	43.0	7.6	47.6	17.7	27.4	7.3
As prepared	25.5	16.8	47.8	9.9	33.2	28.6	34.1	4.1
	25.5	23.1	45.0	6.4	45.5	13.2	30.9	10.4
	32.0	26.8	37.2	4.0	12.4	27.2	52.5	7.8
350 °C	39.6	21.4	37.8	5.2	12.7	28.2	51.2	7.9
	35.5	26.5	34.4	3.7	13.8	29.6	50.1	6.5
	–	15.7	61.4	22.9	–	25.4	62.9	11.7
900 °C	–	22.6	60.1	17.3	–	19.7	65.7	14.6
	–	23.6	61.5	14.9	–	14.6	60.5	24.8
	–	28.6	62.6	8.8	–	19.2	63.0	17.8
1000 °C	–	28.2	63.9	7.9	–	22.6	63.6	13.7
	–	27.4	64.2	8.4	–	16.1	63.9	20.0
	–	20.9	66.9	12.2	–	18.3	59.3	22.3
1150 °C	–	21.3	68.5	10.1	–	22.8	61.9	15.3
		19.1	66.4	14.1	–	21.9	63.7	14.4

Table 3
Magnetic parameters for the supported iron oxide nanoparticles.

Support	Parameter	Calcination temperature			
		At 350 °C	At 900 °C	At 1000 °C	At 1150 °C
	M_s at 300 K (emu/g)	2.2	3.3	5.2	3.5
Macro-crystalline MgO	H_c at 300 K (Oe)	35	47.2	106.5	27.7
	M_s at 5 K (emu/g)	4.2	5.6	7.8	5.4
	H_c at 5 K (Oe)	130	204.6	128.2	90.4
	M_s at 300 K (emu/g)	1.0	6.4	8.0	7.5
Nanocrystalline MgO	H_c at 300 K (Oe)	Negligible	82.6	28.4	Negligible
	M_s at 5 K (emu/g)	2.4	8.9	11.3	10.7
	H_c at 5 K (Oe)	126.7	110.5	54.4	Negligible