

## Research Article

# Synthesis, Characterization, and Magnetic Studies of $\alpha$ - $\text{Fe}_2\text{O}_3$ Nanoparticles

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Very fine nanosized metal oxide, namely, iron oxide ( $\alpha$ - $\text{Fe}_2\text{O}_3$ ) has been synthesized by precipitation method using ammonia as precipitating agent and characterized by using XRD (X-ray diffraction), TGA/DTA, surface area measurement, transmission electron microscopy (TEM), and magnetic measurements techniques. XRD studies show that iron oxide was formed as  $\alpha$ - $\text{Fe}_2\text{O}_3$  instead of the commonly formed magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) or a mixture of magnetite ( $\text{Fe}_3\text{O}_4$ ) and maghemite ( $\gamma$ - $\text{Fe}_2\text{O}_3$ , cubic), and it has rhombohedral structure. Magnetic measurements showed that iron oxide has five unpaired electrons and is ferromagnetic in nature,  $M_s$  value being 1.7 emu/g. The particle size of the synthesized iron oxide was determined by TEM. TEM images show that the size of particles of  $\text{Fe}_2\text{O}_3$  varied from 15 nm to 49 nm with average crystallite size 35 nm.

## 1. Introduction

Transition metal oxides have many applications as catalysts [1–5], sensors [6–9], superconductors [10, 11], and adsorbents [12, 13]. Metal oxides constitute an important class of materials that are involved in environmental science, electrochemistry, biology, chemical sensors, magnetism, and other fields. One of their most important applications is heterogeneous catalysis. Iron oxides belong to the most abundant minerals and occur with a large variety of stoichiometries, structures, and properties. The more important ones are FeO (wustite),  $\lambda$ - $\text{Fe}_2\text{O}_3$  (maghemite),  $\alpha$ - $\text{Fe}_2\text{O}_3$  (hematite), and  $\text{Fe}_3\text{O}_4$  (magnetite) with rock-salt, vacancy rich inverse spinel, corundum, and inverse spinel structures, respectively; the two former ones being thermodynamically less favorable and  $\alpha$ - $\text{Fe}_2\text{O}_3$  being the most oxidized one. Iron oxides are widely used in industry as catalysts or catalyst supports. Nanosized iron oxide particles within various ordered mesoporous silicas (SBA-15, SBA-16, Fm3m, and Ia3d) have been prepared from the corresponding nitrate and acetylacetonate precursors and studied for their catalytic behavior in methanol decomposition [14]. Nanosized catalyst (iron oxide) into the pores of

a mesoporous material (titania) has been deposited using ultrasound radiation and the resulting catalyst is used for the oxidation of cyclohexane under mild conditions [15]. Nanosized iron and mixed iron-cobalt oxides supported on activated carbon materials and their bulk analogues have been synthesized and their catalytic behavior in methanol decomposition to  $\text{H}_2$ , CO, and methane is tested [16]. A series of nanosized gold/iron-oxide catalysts has been prepared and tested for CO oxidation [17]. Catalytic oxidation of PCDD/Fs (polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans) with ozone (catalytic ozonation) over nanosized iron oxides (denoted as  $\text{Fe}_x\text{O}_y$ ) has been carried out at temperature of 120–180°C [18]. Iron oxide nanoparticles (IONPs) as solid catalyst were prepared using a biotic method, that is, biomineralization, and abiotic methods, that is, thermal decomposition and electrochemical methods, for use as solid catalysts in the heterogeneous catalytic ozonation of *para*-chlorobenzoic acid (*p*CBA) [19]. Nanosized iron oxide has applications in waste water treatment and as sensors. Role of nanosized colloidal iron oxides in microbial iron reduction has been studied [20]. Implications of heat treatment on the

properties of a magnetic iron oxide-titanium dioxide photocatalyst have been studied [21]. Nanosized iron oxide particles were intercalated into the interlayer of layered compounds  $\text{HTiNb}(\text{Ta})\text{O}_5$  by a successive ion-exchange reaction and studied for photocatalytic water splitting [22]. Adsorption and desorption properties of arsenate  $[\text{As}(\text{V})]$  on nano-sized iron-oxide-coated quartz (IOCQ) through batch experiments were conducted to investigate the coating of nano-sized iron oxide on the quartz surface using the heat treatment process which aimed to utilize the adsorption properties of the nano-sized iron oxide and the filtration properties of the quartz [23]. Removal of phosphate from solution using nanosized  $\text{FeOOH}$ -modified anion resin was studied in fixed bed column. Effect of bed height and flow rate on the breakthrough curves was investigated [24]. Nanosized particles of  $\alpha\text{-Fe}_2\text{O}_3$  in the range of 17–64 nm were synthesized and were used for LPG sensing [25]. A simple and reproducible method to obtain  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  mixed oxide thin films by reactive RF sputtering has been presented and investigated for the gas sensing properties toward CO [26]. Different methods have been used for the synthesis of nanosized iron oxide nanoparticles. The raw material Lauha churra (iron filings) that has been taken as raw material for the synthesis of nanosized iron oxide and phase transformation from  $\alpha$ - to  $\gamma$ -phase has been studied [27]. Highly crystallized iron oxide nanorods have been fabricated by hydrothermal synthesis in the cavity of carbon-coated nanochannels with a diameter of 25 nm [28]. Nanosized iron oxide powder with average crystallite sizes 35, 100 and 150 nm was prepared by thermal evaporation and coprecipitation techniques and tested as catalyst for the photocatalytic decomposition of Congo red dye [29]. Nanosized iron oxides have considerable attention due to their unique magnetic properties (superparamagnetism, high coercivity, low curie temperature, high magnetic susceptibility, nontoxicity, biocompatibility, and low cost of production), which allow their usage in various nanotechnology applications in a broad range of disciplines. Magnetic nanoparticles are also important in biomedical applications, for example, magnetic bioseparation [30], magnetic target drug delivery [31], hyperthermia [32], magnetic resonance imaging [33], and magnetofection [34]. In the present paper we have synthesized  $\alpha\text{-Fe}_2\text{O}_3$  nanoparticles by simple aqueous precipitation using ammonia as precipitating agent and their magnetic properties have been studied. This method involves a simple, cheap, and one-step process for synthesis of  $\text{Fe}_2\text{O}_3$  nanoparticles. Iron oxide was formed as  $\alpha\text{-Fe}_2\text{O}_3$  instead of the commonly formed magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) or a mixture of magnetite ( $\text{Fe}_3\text{O}_4$ ) and maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ , cubic). The obtained particles of  $\text{Fe}_2\text{O}_3$  have size from 15 to 42 nm. The synthesized nanoparticles were characterized by XRD, TGA/DTA, magnetic susceptibility, and TEM.

## 2. Methods and Materials

**2.1. Chemicals.** All chemicals used in the experiment are analytic reagent grade. Ferric nitrate,  $\text{Fe}(\text{NO}_3)_3$ , was purchased from Merck, India. Ammonium hydroxide (liquor

ammonia) was purchased from SRL. Deionized water was used throughout the experiment.

**2.2. Synthesis of Iron Oxide.** 500 mL of 0.1 M solution of  $\text{Fe}(\text{NO}_3)_3$  was taken and aqueous ammonia was added dropwise with constant stirring until the pH of the solution reached 10. The precipitates thus obtained were filtered by Buckner funnel and were washed several times with distilled water. The precipitates were dried in oven at  $70^\circ\text{C}$  for 24 hrs and were calcined at  $500^\circ\text{C}$  in a muffle furnace for 5 hrs. Obtained material was ground and sieved through 100 mesh size sieve.

**2.3. Characterization Techniques.** The microstructure of the particles was characterized by X-ray diffraction (XRD), Philips PW 11/90 diffractometer using nickel filtered  $\text{CuK}\alpha$  ( $l = 1.5405 \text{ \AA}$ ) radiations. The average diameter ( $D$ ) of the iron oxide nanocrystals has been calculated from the broadening of the XRD peak intensity after  $K\alpha_2$  corrections using the Debye-Scherrer equation. Transmission electron microscopy (TEM) measurements of the sample were taken on Hitachi H7500 with a 70 kV accelerating voltage. The dispersions of nanoparticles in water were placed on carbon-coated 400 mesh copper grids, allowed to dry at room temperature before taking measurement. The obtained micrographs were then examined for particle size and shape. The magnetic property of the solid was measured at 300 K using a Vibrating sample Magnetometer Model 155. TGA/DTA studies were carried out using Perkin Elmer Pyris Diamond. The BET surface area of the samples was measured by nitrogen adsorption isotherms on micromeritics ASAP 2010 (UK).

## 3. Results and Discussions

**3.1. X-Ray Studies.** X-ray diffraction of synthesized oxide is shown in Figure 1. X-ray diffraction pattern of pure iron oxide indicated that iron oxide was in the form of  $\alpha\text{-Fe}_2\text{O}_3$  (Figure 1). The X-ray diffraction plot, shown in Figure 1, shows peaks only due to  $\alpha\text{-Fe}_2\text{O}_3$  and no peak is detected due to any other material or phase indicating a high degree of purity of the as-synthesized sample. The broadening of the X-ray diffraction lines, as seen in the figure, reflects the nanoparticle nature of the sample. In X-ray diffraction, some prominent peaks were considered and corresponding  $d$ -values were compared with the standard [JCPDS file no. 85-0987] (Table 1). X-ray diffraction shows that metal oxide is pure  $\alpha\text{-Fe}_2\text{O}_3$  having rhombohedral structure.

Sharpness of the peaks shows good crystal growth of the oxide particles. Average particle size ( $t$ ) of the particles has been calculated from high intensity peak using the Debye-Scherrer equation

$$t = \frac{K\lambda}{B \cos \theta}, \quad (1)$$

where  $t$  is the average crystallite size of the phase under investigation,  $K$  is the Scherrer constant (0.89),  $\lambda$  is the wave length of X-ray beam used,  $B$  is the full width at half

TABLE 1: X-ray diffraction data for iron oxide ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>).

S. No.	$d$ (Å) (observed)	$d$ (Å) (reported)	$I/I_0 \times 100\%$ (observed)	$I/I_0 \times 100\%$ (reported)
(1)	3.6806	3.6775	35.78	58.7
(2)	2.6980	2.6959	100.00	100
(3)	2.5155	2.5135	83.14	63.1
(4)	2.2033	2.2015	24.03	3.4
(5)	1.8394	1.8379	36.98	6.1
(6)	1.6949	1.6936	43.26	18.0
(7)	1.4852	1.4840	26.88	18.1
(8)	1.4511	1.4512	26.77	9.7

85-0987		Wavelength= 1.54060					C				
Fe <sub>2</sub> O <sub>3</sub>		2 $\theta$	Int	h	k	l	2 $\theta$	Int	h	k	l
Iron Oxide		24.201	587	1	1	0	84.578	5	4	4	4
		33.212	999*	1	2	1	85.191	57	3	2	1
		35.729	631	1	1	0	88.440	4	1	4	0
		39.320	334	2	2	2	88.808	57	2	4	0
		40.957	34	1	2	0					
		43.622	3	0	2	0					
		49.575	61	2	2	0					
		54.168	180	1	3	2					
		56.319	7	1	2	0					
		57.609	27	1	2	1					
		57.609		3	3	2					
		62.598	181	1	3	0					
		64.191	97	2	1	1					
		66.194	3	2	3	0					
		69.720	27	2	4	2					
		72.035	75	3	4	3					
		75.361	3	2	4	1					
		75.690	76	2	2	0					
		77.936	56	3	3	0					
		79.014	1	1	3	1					
		79.738	2	2	2	1					
		80.835	18	1	3	0					
		80.835		3	4	1					
		83.094	18	2	4	4					

Rad.: CuK $\alpha$ 1	$\lambda$ : 1.54060	Filter:	d-sp: Calculated
Cut off: 17.7	Int.: Calculated	I/Icor.: 4.00	
Ref: Calculated from ICSD using POWD-12++ (1997)			
Ref: Zachariassen, W.H., Skr. Nor. Vidensk.-Akad., Kl. 1: Mat.-Naturvidensk. Kl., 1928, 1 (1928)			
Sys.: Rhombohedral	S.G.: R $\bar{3}c$ (167)		
a: 5.42(2)	b:	c:	A: C: 1.0000
$\alpha$ : 55.200(1)	$\beta$ :	$\gamma$ :	Z: 2 mp:
Ref: Ibid.			
Dx: 5.302	Dm:	ICSD # : 033643	

Peak height intensity. R-factor: 0.081. Al<sub>2</sub>O<sub>3</sub> type. C.D.  
 Cell: a=5.022, c=13.738, c/a=2.7354, S.G.=R-3c(167). PSC:  
 hR10. Mwt: 159.69. Volume[CD]: 300.07.



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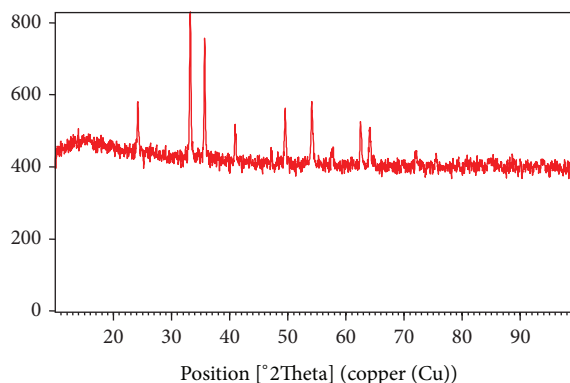


FIGURE 1: -XRD spectra of synthesized iron oxide.

maximum (FWHM) of diffraction (in radians), and  $\theta$  is the Bragg's angle.

The average crystallite size calculated is 35 nm which is in close agreement with the TEM results.

**3.2. Magnetic Measurements.** The magnetic moment for iron oxide was carried out at room temperature and was observed as 5.68 B.M. This value of magnetic moment supports the

fact that the synthesized iron oxide is in the form of Fe<sub>2</sub>O<sub>3</sub> with actual magnetic moment 5.92 B.M. This indicates the presence of 5 unpaired electrons in Fe<sub>2</sub>O<sub>3</sub>. Magnetic measurements were also carried out at temperatures ranging from 300 K to 100 K to determine the temperature of Morin transition. The results are shown in Figure 2(a) and have been reported in Table 2. VSM studies were carried out at 300 K to show hysteresis behavior of nanosized particles and it has

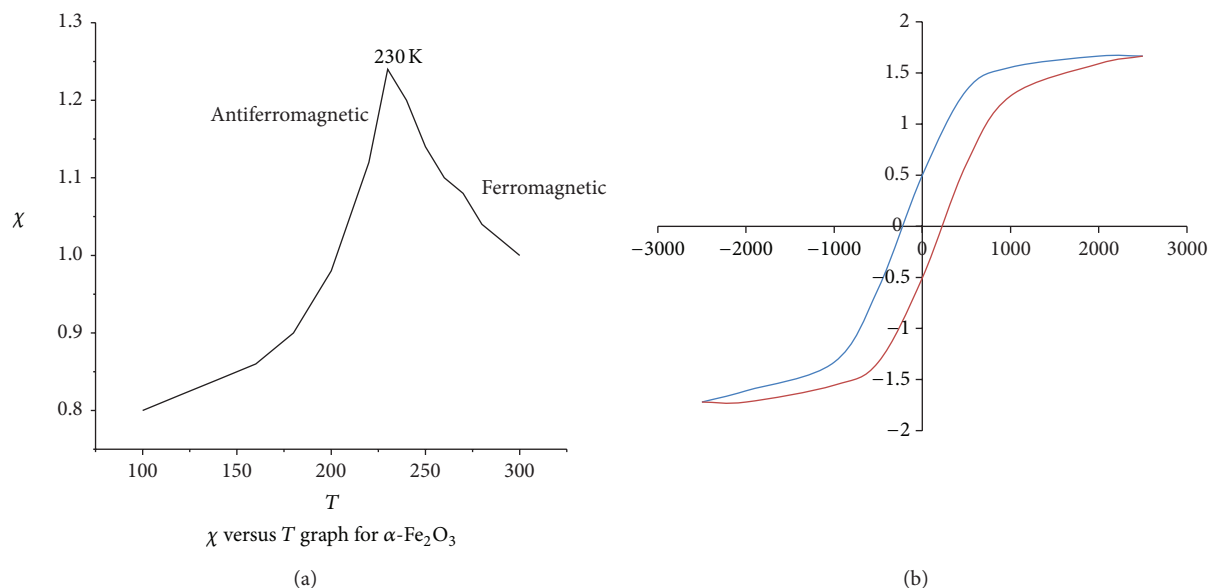


FIGURE 2: (a) Morin transition curve for synthesized iron oxide nanoparticles. (b) VSM studies of synthesized iron oxide.

TABLE 2: Magnetic susceptibility data of iron oxide.

Temperature (K)	Volt (mV)	Magnetic moment (emu.)
300	5.75	0.0050
290	5.58	0.0051
280	5.38	0.0052
270	5.17	0.0054
260	4.96	0.0055
250	4.75	0.0057
240	4.54	0.0060
230	4.33	0.0062
220	4.13	0.0056
200	3.71	0.0049
180	3.29	0.0045
160	2.88	0.0043
140	2.46	0.0042
120	2.05	0.0041
100	1.63	0.0040

been observed that  $\text{Fe}_2\text{O}_3$  show ferromagnetic behavior in nanocrystalline form,  $M_s$  value being 1.7 emu/g (Figure 2(b)).

**3.3. TGA/DTA Studies.** TGA/DTA transition shows an endothermic peak at  $364^\circ\text{C}$  (Figure 3). It simply indicates that when  $\text{FeO}(\text{OH})$  is heated, it takes an amount of energy and 1.5 water molecules are removed. So, for the formation of iron oxide temperature above  $364^\circ\text{C}$  is required.

**3.4. Surface Area Measurement.** The BET surface area of the samples was measured by nitrogen adsorption isotherms. Surface area of the metal oxide was  $27 \text{ m}^2/\text{g}$ . Samples were activated at 473 K for 4 h prior to the measurement.

**3.5. TEM Studies.** TEM studies were carried out to find out exact particle size of synthesized  $\text{Fe}_2\text{O}_3$ . Figure 4 shows the

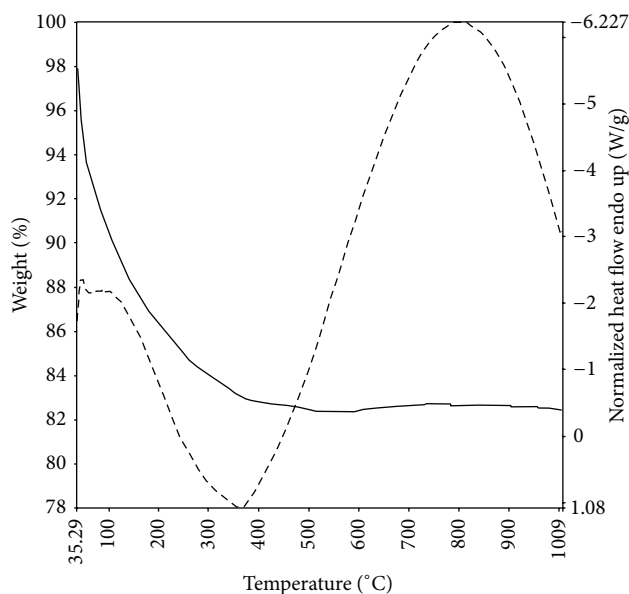
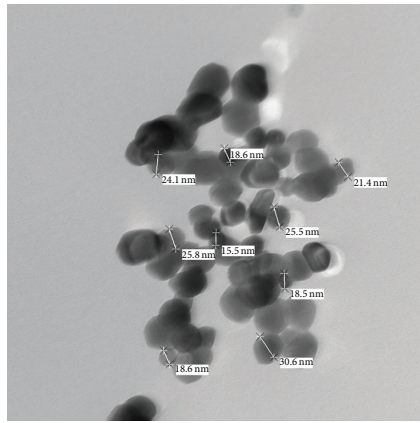


FIGURE 3: TGA/DTA curve of iron oxide heated at  $70^\circ\text{C}$ .

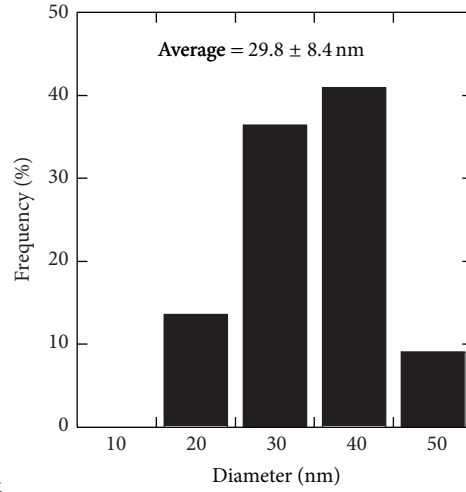
TEM image of the synthesized  $\text{Fe}_2\text{O}_3$  nanoparticles. TEM images show that  $\text{Fe}_2\text{O}_3$  nanoparticles are having particle size in the range of 15 nm–49 nm (Figure 4). The size distribution histograms for nanoparticles provided their respective sizes as  $29.8 \pm 8.4 \text{ nm}$  (Figure 4(a)),  $30.6 \pm 7.0 \text{ nm}$  (Figure 4(b)),  $26.4 \pm 4.7 \text{ nm}$  (Figure 4(c)), and  $32.4 \pm 6.6 \text{ nm}$  (Figure 4(d)), respectively.

## 4. Conclusion

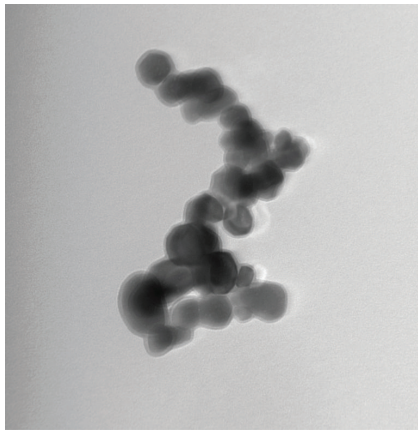
$\alpha\text{-Fe}_2\text{O}_3$  nanoparticles with rhombohedral structure are synthesized successfully by aqueous precipitation method using ammonia as precipitating agent. From TEM study, it is found



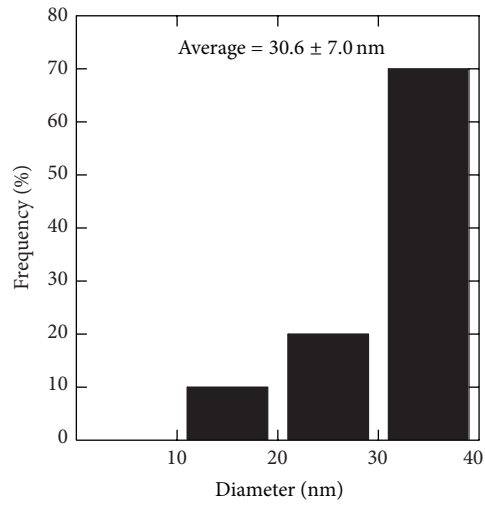
100 nm  
 HV = 90 kV  
 Direct mag: 200000x  
 X: 456.4 Y: 297.9 T:0.4



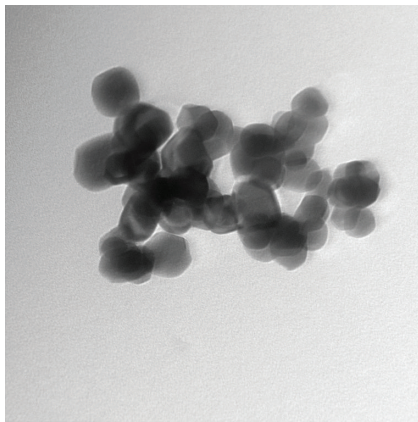
(a)



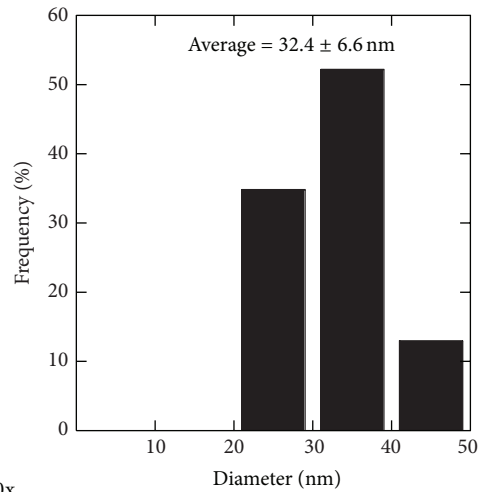
100 nm  
 HV = 90 kV  
 Direct mag: 200000x  
 X: 474.3 Y: 416.8 T: 0.4



(b)



20 nm  
 HV = 90 kV  
 Direct mag: 300000x  
 X: 476.1 Y: 325.8 T: 0.4



(c)

FIGURE 4: Continued.

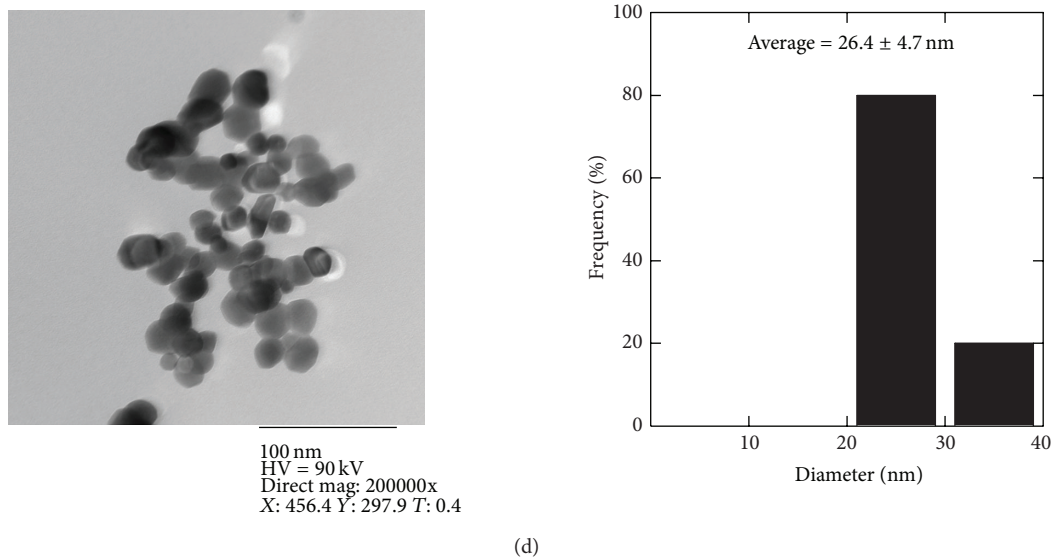


FIGURE 4: TEM images of iron oxide particles.

that particles are with average size of 15–49 nm. Magnetic measurements show that  $\text{Fe}_2\text{O}_3$  has five unpaired electrons. VSM studies show ferromagnetic behavior of synthesized oxides. XRD studies show that iron oxide was formed as  $\alpha\text{-Fe}_2\text{O}_3$  instead of the commonly formed magnetite nanoparticles  $\text{Fe}_3\text{O}_4$  or a mixture of magnetite and maghemite.

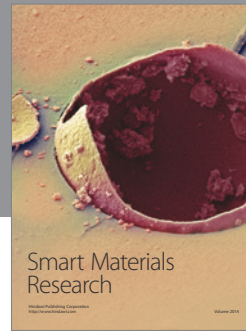
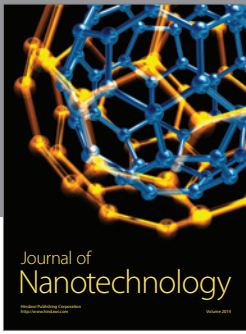
### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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