Synthesis and characterization of composites of mixed oxides of iron and neodymium in polymer matrix of aniline–formaldehyde

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Abstract. Nanocomposites of mixed oxides of iron and neodymium in polymer matrix of aniline–formaldehyde are reported. The composites have been obtained by treating the aqueous solution of aniline, hydrochloric acid and formaldehyde with halide of iron and neodymium oxide. The infra-red spectra show broad peaks at $\sim 590\text{ cm}^{-1}$ and at $\sim 610\text{ cm}^{-1}$ due to the presence of oxides of both iron and neodymium. In heated samples, the absorption peaks due to metal oxides are better resolved. A broad and strong peak in XRD spectra at 2θ value of 35.69920 corresponds to spinel $\gamma\text{-Fe}_2\text{O}_3$. ^{57}Fe Mössbauer spectrum for unheated sample gives Mössbauer parameters, i.e. isomer shift (δ), quadrupole splitting (ΔE) and effective magnetic field (H_{eff}). Transmission electron microscopy (TEM) micrographs reveal well dispersed particles at different magnifications. Vibrating sample magnetometry (VSM) studies indicate that the ferrite nanoparticles exhibit characteristics of ferromagnetism.

Keywords. Nanocomposites; polymer matrix; neodymium oxide; spinel ferrites; quadrupole splitting; Scherrer equation.

1. Introduction

In the past few years, nanocomposite materials have become one of the most extensively studied materials all over the world as they have shown several applications such as in quantum electronic devices, magnetic recording materials, sensors, etc (Anderson *et al* 1994; Sinfelt 1977).

Moreover, nanocomposite materials composed of oxides and conducting polymers have brought out more fields of applications such as smart windows, toners in photocopying, conductive paints, drug delivery, rechargeable batteries, etc (Maeda and Armes 1994; Butterworth *et al* 1995; Jarjays *et al* 1995; Suri *et al* 2001). Many of the attributes that influence structure-property relationships for a polymer such as molecular weight, chain length, architecture, ordering and functional groups have been explored. The size of polymer makes them convenient for use as nanostructured materials, and they are also useful as a scaffold to orient and arrange other nanomaterials to produce nanocomposites with enhanced properties.

The nanocomposites formed by combining organic polymer and inorganic nanoparticles (*viz.* oxides) possess all the good properties of both the constituents and an enhanced utility thereof and hence, constitute a relatively new and unique area in material science. By combining at

the molecular level inorganic and organic polymer material, a blending of unique physical properties can be achieved. Magnetic ferrites having hexagonal/cubical symmetry belong to the magneto-plumbite group of oxides and find application in high density recording media, microwave devices and in many magnetically operated devices such as magnetic levitations, telephone ringers and receivers, etc (Viswanathan 1990; Bahadur 1992; Kladnig and Zenger 1992). An interesting chemical route (Pramanik 1995) has been reported to prepare nanoparticles of inorganic oxides by using polymeric materials.

The present work reports the *in situ* direct synthesis of nanocomposites by employing mixed oxides of iron and neodymium in co-polymer matrix of aniline–formaldehyde at room temperature.

2. Experimental

In the present work, we report the synthesis of a series of nanocomposites of neodymium ferrites in the co-polymer matrix of aniline–formaldehyde.

2.1 Synthesis of nanocomposites in different stoichiometric ratio

Nanocomposites of neodymium ferrites in a co-polymer matrix were synthesized by using the direct room tempe-

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ature chemical method (Vadera *et al* 1997b). Five different nanocomposites were synthesized by taking ferric chloride and neodymium oxide in different stoichiometric ratios given in table 1 along with the monomers to form the copolymer. In a typical preparation, sample ND1 was synthesized by mixing the aqueous solutions of aniline (0.027 mol), hydrochloric acid (0.1 mol) and formaldehyde (0.09 mol) and the chloride of iron and neodymium oxide taken according to the stoichiometry. The resulting solution was stirred thoroughly and added to a 10% solution of sodium hydroxide. The precipitated composite was filtered and washed repeatedly with distilled water till the filtrate was free of alkali and then dried in air. Similarly, the samples ND2–ND5 were synthesized by taking Nd and Fe in different stoichiometric ratios.

The samples ND1–ND5 were heated to 4500°C and 7000°C in a furnace under a restricted supply of air to obtain samples ND6–ND10 and ND11–ND15 respectively.

2.2 Synthesis of nanocomposites in presence of different oxidants

The nanocomposites of neodymium ferrites were synthesized in a co-polymer matrix of aniline–formaldehyde using a synthetic route (Sharma *et al* 2003). As a typical preparation, sample NT1 was synthesized by mixing the aqueous solution of aniline (0.027 mol), hydrochloric acid (0.1 mol), formaldehyde (0.09 mol) and ferric chloride and neodymium oxide taken in a fixed molar ratio. To this quinolinium chlorochromate (0.008 mol) was added as an oxidant. The resulting solution was stirred thoroughly and added to a 10% solution of sodium hydroxide. The precipitated composite was filtered and washed repeatedly with distilled water till the filtrate was free of alkali (pH 7) and then dried in air.

Similarly, the samples NT2–NT7 were synthesized following the similar procedure by using different oxidants (potassium permanganate, morpholinium chlorochromate, pyridinium chlorochromate, potassium dichromate, isoquinolinium chlorochromate, piperidinium chlorochromate). The compositional details of the samples NT1 to NT7 are given in table 2. The samples NT1 to NT7 were heated in a furnace under a restricted supply of air to 4500°C and 7000°C to obtain samples NT8 to NT14 and NT15 to NT21, respectively.

Table 1. Reaction conditions for the synthesis of as-prepared samples ND1–ND5.

Sample	Conc. of FeCl ₃ (mol)	Conc. of Nd ₂ O ₃ (mol)
ND1	0.01	0.005
ND2	0.015	0.005
ND3	0.02	0.005
ND4	0.025	0.005
ND5	0.03	0.005

3. Characterization of nanocomposites

The nanocomposites were characterized by FTIR spectroscopy, X-ray diffraction, ⁵⁷Fe Mössbauer spectroscopy, scanning electron microscopy, energy dispersive X-ray analysis (EDAX), transmission electron microscopy (TEM) and vibrating sample magnetometry (VSM) studies.

3.1 Infrared spectra

The characteristic IR spectral bands of the nanocomposites ND1–ND5 in the frequency range 2000–400 cm⁻¹ are presented in table 3. The absorption peak due to polymer part of the samples (Mathur *et al* 1999a) are observed in the frequency range 2000–800 cm⁻¹ while the absorption peak around 578 and 427 cm⁻¹ can be attributed to the presence of neodymium ferrite (Li *et al* 1998). Vadera *et al* (1997a) have reported a similar IR spectrum of nanocomposite of spinel α -Fe₂O₃ in a co-polymer matrix. Further, the comparison of IR spectra of the samples ND1–ND5 shows that the peak due to neodymium ferrite broadens with increasing concentration of Fe from samples ND1–ND5 which may be attributed to decrease in size of magnetic particles (Mathur *et al* 1999b). In the IR spectra of the samples ND6–ND10 (heated to 4500°C) (table 3), the absorption peak at 575 cm⁻¹ is observed due to neodymium ferrite phase. The peaks due to polymer part disappear as most of the organic part burns away at 4500°C. The shifting of the peak position $\sim$ 575 cm⁻¹ due to neodymium ferrite in samples ND6–ND10 may be attributed to the increase in their particle size. IR bands of samples ND11–ND15 heated to 7000°C are given in table 3. The IR spectrum of sample ND12 shows peak at 560 cm⁻¹ thereby indicating that both α -Fe₂O₃ and neodymium ferrite coexist in the sample (Mathur *et al* 1999b).

The IR spectra of the nanocomposites NT1–NT7 (table 4) show strong absorption around 580 and 428 cm⁻¹ due to neodymium ferrite (Sharma *et al* 2003). The possibility of α -FeOOH is ruled out due to the absence of its characteristic absorption at 800 cm⁻¹ and 890 cm⁻¹ (Koch *et al* 1986). The absorption bands observed at 815 and 510 cm⁻¹ are due to presence of the co-polymer (Mathur *et al* 1998). It is evident that for the as-prepared nanocomposites NT1–NT7, absorption peak around 600 cm⁻¹ is due to tetrahedrally coordinated metal ion in spinel ferrites (Josyulu and Sobhanadri 1981). This peak may be attributed to the presence of oxides of both iron and neodymium in these samples (Li *et al* 1998). In case of the samples heated to 4500°C (table 4), the polymer part is burnt and only the absorption peaks due to metal oxides are found. In the samples NT8–NT14, containing iron and neodymium, absorption peaks at around 590–570 cm⁻¹ and 495–470 cm⁻¹ are due to γ -Fe₂O₃ and neodymium

Table 2. Reaction conditions for the synthesis of as-prepared samples NT1–NT7.

Sample	Conc. of FeCl ₃ (mol)	Conc. of Nd ₂ O ₃ (mol)	Oxidant used	Conc. of the oxidant
NT1	0.15	0.005	Potassium dichromate	0.009
NT2	0.15	0.005	Potassium permanganate	0.019
NT3	0.15	0.005	Morpholinium chlorochromate	0.010
NT4	0.15	0.005	Quinolinium chlorochromate	0.008
NT5	0.15	0.005	Isoquinolinium chlorochromate	0.008
NT6	0.15	0.005	Pyridinium chlorochromate	0.013
NT7	0.15	0.005	Piperidinium chlorochromate	0.010

Table 3. IR bands (cm⁻¹) of nanocomposites prepared in different stoichiometric ratios.

Sample	Peaks of as prepared samples	Sample	Peaks of samples heated to 450°C	Sample	Peaks of samples heated to 700°C
ND1	1634.6, 1517.4, 1462.3, 745.1	ND6	867.1, 689.6, 536.1	ND11	534.0, 485.2
ND2	1635.8, 1576.5, 1555.0, 816.3	ND7	856.8, 565.8, 445.5	ND12	590.1, 474.0
ND3	1685.6, 1572.9, 743.3, 668.7	ND8	604.1, 585.5, 549.7	ND13	556.8, 467.0
ND4	1675.6, 1666.3, 624.3, 556.4	ND9	723.1, 638.4, 554.5	ND14	545.3, 463.0
ND5	1637.4, 1543.3, 625.2, 423.0	ND10	765.3, 556.5, 424.4	ND15	556.0, 472.3

Figure 1. XRD spectra of sample ND7.

ferrite phase respectively (Li *et al* 1998). Further, variations in absorption peak due to iron oxide, in samples

NT8–NT14, may be attributed to varying degrees of interactions between Fe and Nd ions which in turn might be a consequence of the changes in polymer backbone by different oxidative environment, in the initial samples. From the IR spectra of samples heated to 7000°C, further broadening of the metal oxide peaks is observed in samples NT15–NT21. This behaviour may arise because of the increased solubility of neodymium oxide in γ -Fe₂O₃ due to their small size effects (Johnson and Alexander 1986). A very broad peak at 555 cm⁻¹ indicates that both α -Fe₂O₃ and neodymium ferrite coexist in the samples (Mathur *et al* 1999b).

3.2 X-ray diffraction

X-ray diffraction spectra (XRD) of sample ND7 heated to 4500°C is shown in figure 1. It is seen that the lines for the heated samples are narrow and intense which indicates that on heating the nanocomposites, the

Table 4. IR bands (cm^{-1}) of nanocomposites prepared in presence of different oxidants.

Sample	Peaks of as prepared samples	Sample	Peaks of samples heated to 450°C	Sample	Peaks of samples heated to 700°C
NT1	1712.1, 1523.4, 1423.3, 815.1	NT8	723.3, 578.0	NT15	523.1, 444.2
NT2	1665.5, 1592.5, 1565.0, 877.3, 487.8	NT9	1566.8, 1458.5, 765.6, 569.0, 526.6	NT16	543.5, 445.2
NT3	1764.6, 1556.9, 871.3, 669.7	NT10	1663.1, 635.5, 565.8, 465.6	NT17	562.8, 456.2
NT4	1666.6, 1622.3, 1612.3, 512.4	NT11	1532.1, 882.4, 569.5, 479.3	NT18	568.3, 445.0
NT5	1633.4, 1534.3, 862.2, 412.0	NT12	1766.3, 571.2, 775.2, 423.2	NT19	561.2, 437.1
NT6	1563.1, 1463.0, 689.2, 542.3	NT13	1657.2, 1475.2, 764.3, 554.8	NT20	576.8, 438.1
NT7	1676.2, 1575.4, 1578.9, 539.1	NT14	1607.5, 1577.4, 813.6, 774.3	NT21	557.1, 442.5

Figure 2. XRD spectra of sample ND13.

inorganic component still remains unchanged in terms of its structure, i.e. it is still neodymium ferrite but with an increase in particle size. A very broad peak in case of samples ND7 and ND8 at 2θ value of 35.69920 corre-

sponds to the strongest diffraction peak due to spinel $\gamma\text{-Fe}_2\text{O}_3$ and neodymium ferrite. The estimated particle size calculated by using Scherrer equation (Suryanarayana and Norton 1996) lies in the range 10–16 nm as given in table 5.

$$B = K\lambda / L \cos\theta,$$

where β is the broadening of the line in terms of angle (2θ), K is the constant (taken as 1), L is the average length of the crystallite, λ is the wavelength of the X-rays and θ is the angle of diffraction.

The XRD spectra of the samples ND11–ND15 heated to 7000°C show very interesting behaviour. As can be seen from figure 2, in case of ND13, the diffraction peak at 2θ value of about 25.69930 corresponds to $\alpha\text{-Fe}_2\text{O}_3$ phase (Mathur *et al* 1999b) while in sample ND13, increase in intensity of diffraction pattern due to Nd-Fe/ $\alpha\text{-Fe}_2\text{O}_3$ phase is observed. The calculations suggest that the average size in these samples range from 14–27 nm as given in table 5.

From the XRD studies, it is seen that the metal oxides in unheated samples NT1–NT7 were amorphous while on

heating at temperatures $> 3000^{\circ}\text{C}$, the samples were crystalline in nature. The sample on heating to 4500°C in air for one hour mostly contain inorganic part as almost all the organic part has burnt away at this temperature. The observed diffraction peaks were indexed to identify different phases present in the samples. In the XRD spectrum of sample NT11, a broad peak at 2θ value of 35.77550 corresponds to the strongest diffraction peak due to $\gamma\text{-Fe}_2\text{O}_3$ and Nd-Fe phase respectively (figure 3). The estimated particle size as calculated by using Scherrer equation is 17 nm as shown in table 6.

The XRD spectrum of the sample NT18 heated to 7000°C is shown in figure 4. From the figure, a strong diffraction peak at 2θ value of 33.22340 corresponds to $\alpha\text{-Fe}_2\text{O}_3$ phase. A prominent peak corresponding to Nd-Fe is also identified at $2\theta = 54.24380$ with slight shift in

case of sample NT15 (Li *et al* 1998; Mathur *et al* 1999b). The calculations suggest that the average particle size in the samples is 31 nm as given in table 6.

The above studies show that in unheated samples, the formation of $\gamma\text{-Fe}_2\text{O}_3$, takes place at room temperature which on heating to 7000°C transforms into $\alpha\text{-Fe}_2\text{O}_3$. The studies also show that the extent of reduction in the intensity of diffraction due to Nd-Fe phase is greatly influenced by varying the nature of the oxidative environments present during the preparation of initial samples and its solubility behaviour.

3.3 ^{57}Fe Mössbauer spectroscopy

Room temperature ^{57}Fe Mössbauer spectrum was taken for unheated sample ND5 (figure 5). The values of the computed Mössbauer parameters for the sample ND5 are given in table 7. Fitting of the spectral data, by assuming the lines to be lorentzian in shape, given in figure, shows the presence of two sextets corresponding to magnetically

Table 5. Crystallite size of nanocomposites in different stoichiometric ratios.

Sample	Crystallite size (nm)	Sample	Crystallite size (nm)
ND6	14	ND11	17
ND8	12.6	ND13	19
ND10	9	ND14	21.9

Table 6. Crystallite size of nanocomposites in presence of different oxidants.

Sample	Crystallite size (nm)	Sample	Crystallite size (nm)
NT9	15	NT16	16.30
NT11	11.4	NT18	22.4
NT13	10.9	NT21	26.3

Figure 3. XRD spectra of sample NT11.

Table 7. Mössbauer parameters for the magnetic particles of iron/neodymium oxides in copolymer matrix of aniline-formaldehyde.

Sample	Hhf (kOe)	QS (mm/s)	IS (mm/s)	WV (mm/s)
ND5				
Sextet I	362	-0.08	0.19	0.60
Sextet II	483	-0.39	0.25	0.10
Doublet	—	0.77	0.24	0.30

Isomeric shift values are with respect to natural iron

Table 8. Mössbauer parameters for the magnetic particles of iron/neodymium oxides in copolymer matrix of aniline-formaldehyde in presence of oxidant potassium dichromate.

Sample	Hhf (kOe)	QS (mm/s)	IS (mm/s)	WV (mm/s)
NT1	—	0.56	0.22	0.42

Isomeric shift values are with respect to natural iron

Figure 4. XRD spectra of sample NT18.

ordered phase. The six-line pattern could be resolved into two sextets with H_{eff} values which correspond to the presence of $\alpha\text{-Fe}_2\text{O}_3$ (Vadera *et al* 1997a). However, the relaxed nature of the spectrum indicates that the size of the magnetic particles is in the critical nanometer range.

Room temperature ^{57}Fe Mössbauer spectrum was recorded for unheated sample NT1 (figure 6). The Mössbauer parameters calculated from the computer fitted spectral data for the sample NT1 are given in table 8. A single quadrupole doublet due to Fe^{3+} ions is observed. The higher values of QS as compared to bulk iron are a clear indication of the particles being in nanometer range (Greenwood and Gibb 1971). The presence of quadrupole doublet indicates the presence of paramagnetic/superparamagnetic phase for which H_{eff} value is zero which shows the presence of very small sized particles of neodymium ferrite (Matutes- Aquino *et al* 2000).

3.4 Scanning electron microscopy and energy dispersive X-ray analysis (EDAX)

Figure 7 shows SEM micrograph of the unheated sample ND3. SEM micrograph of neodymium ferrite shows well

Figure 5. Room temperature Mossbauer spectra of sample ND5.

Figure 6. Room temperature Mössbauer spectra of sample NT1.

dispersed particles at different magnifications (Li *et al* 1998). In some portions of the image, agglomerated spherical particles are seen perhaps due to their magnetic nature. The particles vary in size and dimensions showing composite nature which is observed in X-ray diffraction.

Figure 8 shows the SEM micrograph of the sample NT5. It is seen that particles vary in size and

Figure 7. SEM image of sample ND3.

Figure 8. SEM image of sample NT5.

Table 9. EDAX results: concentration of different elements in prepared sample ND3.

Element	Wt%	At%
CK	67.38	79.06
NK	09.44	09.50
OK	10.51	09.26
CIK	00.69	00.27
FeK	04.78	01.21
NdL	07.20	00.70
Matrix	Correction	ZAF

dimensions showing composite nature which is observed in the X-ray diffraction. They are surrounded by an oxidant layer which separates them from each other and prevents aggregation and exhibits rough surface of composites.

Figures 9 and 10 show the corresponding EDAX spectra of samples ND3 and NT5, which indicate the presence of only iron, neodymium, chlorine and oxygen. It

is observed that the atomic percentage of neodymium is less than that of iron and oxygen as shown in tables 9 and 10. The sample NT5 shows the presence of chromium as an impurity from the oxidant.

3.5 Transmission electron microscopy

TEM micrograph of the sample NT5 is shown in figure 11. From the micrograph it is evident that the mixed

Figure 9. EDAX image of sample ND3.

Figure 10. EDAX image of sample NT5.

Table 10. EDAX results: concentration of different elements in prepared samples NT5.

Element	Wt%	At%
CK	66.34	79.00
NK	8.04	09.50
OK	9.51	08.36
ClK	00.69	00.27
CrK	03.40	01.16
FeK	04.28	01.25
NdL	07.35	00.73
Matrix	Correction	ZAF

Figure 11. TEM Image of sample NT5.

Figure 12. TEM image of sample NT19.

Figure 13. Magnetic hysteresis loop of sample ND3.

Figure 14. Magnetic hysteresis loop of sample NT7.

oxide particles are finely dispersed in the polymer matrix and are approximately spherical in shape within a narrow size range. However, in case of heating sample NT5 to 7000°C (NT19) (figure 12), the micrograph shows spherical particles with a particle size distribution of approximately 9–50 nm. The residual part of the decomposed polymer can also be seen in the micrograph.

3.6 Vibrating sample magnetometry studies

The ferrites are solid-phase materials that constitute a combination of Fe_2O_3 with the oxides of other metals. They have special ferromagnetic, dielectric and semiconductor properties. Magnetic parameters were determined for samples ND3 and NT7 using vibrating sample magne-

tometer (VSM) at room temperature. Figures 13 and 14 show the presence of a hysteresis loop at room temperature of the resultant nanocomposite of neodymium ferrite. The magnetization curve shows increase in magnetization with increasing field.

The values for coercivity (H_c) and saturation magnetization (M_s) obtained from the hysteresis loop are 23.91 Oe and 3.01 emu/g and 22.42 Oe and 2.99 emu/g respectively. The magnetization curve for the as-prepared nanocomposite exhibits ferromagnetic curve. The values for both H_c and M_s are lower as compared to reported values for bulk sample (Mishra *et al* 2004). The low values of H_c and M_s observed in nanocomposites is due to the existence of non-magnetic phase and non-magnetic medium because of dipole-dipole interactions, which contribute to magnetic anisotropy and consequently

change the magnetic properties of nanoparticles (Li *et al* 1998). Also, the small size of nanoparticles is responsible for low value of coercivity which gives rise to the formation of monodomain structure.

4. Conclusions

The chemical route using the copolymer matrix of aniline-formaldehyde has been very effective in the synthesis of nanocomposites of neodymium ferrites. The X-ray diffraction, ^{57}Fe Mössbauer and scanning electron microscopy show the formation of nanosized particle of neodymium ferrites in the polymer matrix. These studies further show the formation of solid solution of iron and neodymium oxide on heating the samples at temperatures ranging from 4000–7000°C. From Mössbauer and X-ray diffraction studies, it is seen that $\gamma\text{-Fe}_2\text{O}_3$ which normally transforms into $\alpha\text{-Fe}_2\text{O}_3$ on heating up to 7000°C, still persists in the samples containing neodymium ions. VSM studies indicate the superparamagnetic nature of the composites. Further, the infrared studies indicate that the polymeric backbone is strongly influenced by different reaction conditions and lead to variable magnetic characters in the heated samples.

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