The size dependence and defect induced room temperature ferromagnetism of ZnO and Zn 1-x-y AlxZyO (Z=Mg/Ni) nanocomposites

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In the current research, Al-Mg-ZnO and Al-Ni-ZnO nanocomposites were synthesized by using simple soft chemical route. Prepared nanocomposites of Al-Mg-ZnO and Al-Ni-ZnO with capsule shape distributed magnetite nanostructured were carried out under the calcinated at 120 C for 12 h in furnace. The structural morphology and characterization analysis of as prepared nanocomposites was analyzed by XRD, UV–vis. FTIR, PL, TEM, VSM studies. XRD analysis confirmed the highly pure crystallized cubic phases. The XRD peaks show that the crystalline sizes are of the order of 22 nm, 19 nm, and 11nm. Magnetic property of the prepared nanocomposites was discussed in room temperature using VSM measurement. In spite of a number of researchers reporting the effect of co-doping ZnO nanoparticles (NPs) with two different metals was modify the properties of the entire system such as enrich in room-temperature ferromagnetism. In this research we tried to by experimentally observed the magnetic properties of a series of soft chemical synthesized Zn1-x-yAlxZyO (Z=Mg/Ni) nanocomposites. Interestingly, it was found that in comparison to un-doped ZnO NPs and co-doped with two different metals. The ferromagnetic signal changes in a co-doped system in which one of the Mg/Ni ions increases the concentration of defects mechanism when Mg/Ni ions exhibited only one oxidation state. The potential role of charge transfer ferromagnetism is involving Mg2+ and Ni2+ ions substituted into ZnO lattice, The origin of magnetism in the nanocomposites is due to exchange interaction between local spin polarized electrons and the conduction electrons. The potential role of charge transfer ferromagnetism involving mixed valence ions and effects defect mechanism was used to explain the room temperature ferromagnetism.

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1. Introduction

Recent research, metal oxide diluted magnetic semiconductor based on TiO_2, SnO_2 and ZnO, and have great attention and novelty application due to the possibility of inducing room temperature ferromagnetism. Increased thrust for search of new novel materials due to increase the demand in nanotechnology, magneto electronics, spintronics, half-metallic magnets, etc. have been at the centre of the technical movement [1-8]. Zinc oxide is a member of transparent conductor for most promising candidate in scientific and technical applications [8-14]. These applications are possible in nanosized ZnO as due to combination of exclusive physical properties such as high excitation binding energy (60 MeV), and wide direct band-gap (3.3 eV). In the last decade, doped and co-doped semiconductors based on ZnO have been reported such as ZnS, $Sn0_2$, TiO_2, etc. [15-22].

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Recently, most of the researcher has tried to dope other metal ions into ZnO to obtain two metal ions co-doped ZnO in order to change their structural, magnetic, electrical and optical properties by introducing additional carriers [21-29]. During current research, various works have been reported on room temperature ferromagnetism (RTFM) in transition metal (TM) ions (Mn^{2+}, Co^{2+}, Ni^{2+}, etc.) doped ZnO systems [30-40]. The replace the interaction between free delocalized carriers and the localized d spins of Ni and Cu ions was considered nanoparticles [40-46]. So far of our knowledge, most of the researchers reported to magnetic properties of transition metal doped ZnO but the magnetic properties of Al-Mg co-doped ZnO and Al-Ni co-doped ZnO has not yet been reported.

In the present investigation, optical and magnetic properties of un-doped ZnO, Al-Mg co-doped ZnO and Al-Ni co-doped ZnO prepared by soft chemical method have been reported. The substitution of Mg, Ni and Al into ZnO lattice is used to alter its properties such as structural, optical, and room temperature ferromagnetism. The origin of the ferromagnetic ordering at room temperature (RT) and the intrinsic defect in the prepared samples of co-doped ZnO nanoparticles was also discussed. Based on the reports, this material is suitable for many electromagnetic device applications.

2. Materials and methods

In the experiment, the co-doped samples were synthesized by the soft chemical method. The zinc nitrate hexahydrate ($Zn(NO_3)_26H_20$), magnesium nitrate hexahydrate (Mg(NO3)26H20), aluminium nitrate nonahydrate ($Al(NO_3)_36H_20$), nickel nitrate hexahydrate ($Ni(NO_3)_2.6H_20$) and sodium hydroxide (NaOH) were used as the starting materials without further purification. The aluminium and magnesium co-doped ZnO nanoparticles $Zn_{0.90}$ $Al_{0.05}Mg_{0.05}O$ were prepared. The atomic % of the dopants precursor was changed in the precursor solution. Finally a white coloured colloidal solution was obtained and it was washed many times with de-ionized water. Then the powder was dried in air. The powder was calcinated at 120°C for 12 hours. Finally the products were ground for 15 minutes to obtain fine aluminium and magnesium co-doped ZnO nanoparticles (NPs). The same procedure was repeated for $Zn_{0.90}$ $Al_{0.05}Mg_{0.05}O$ nanocomposites. The prepared undoped ZnO, $Zn_{0.90}$ $Al_{0.05}Mg_{0.05}O$ and $Zn_{0.90}$ $Al_{0.05}Ni_{0.05}O$ composites were hereafter termed as A, B and C, respectively.

2.1. Characterization of the prepared nanocomposites

Techniques such as X-ray diffraction (XRD), ultra violet (UV) diffuse reflectance spectroscopy (DRS), Photoluminescence (PL), Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), and vibrating sample magnetometer (VSM) measurements were performed to characterize the prepared nanocomposites (A, B & C).

The X-ray diffractometer (X'Pert PRO; PANalytical, the Netherlands) was used to identify the structural phase and the crystallite size of the prepared nanocomposites. CuK, radiation (X=1.5406A) was operated at voltage and current ratings of 40 kV and 30 mA, respectively, in the 20 scan position from 10° to 80°. The optical properties of the prepared nanocomposites were determined using a UV-visible DRS (UV-2450, Shimadzu, USA) and PL (PL-8031, Cary Eclipse, Agilent Technologies, Singapore). The UV-DRS spectra and PL were recorded the wavelength-dependent absorption of the prepared nanocomposites from 200 to 1200 nm.

The functional groups and chemical bonds existing in the prepared nanocomposites were identified using FTIR spectra (Perkin Elmer Spectrometer, Spectrum 100, USA) in the wavenumber range of 4000-400 cm^{-1} at room temperature. A pellet was fashioned of 95% KBr with 5% prepared nanocomposites for FTIR spectral analysis. The surface morphology and elemental composition of the prepared nanocomposites were characterized using TEM (Philips CM 200) and XRF analysis spectrum (EDX-720; Shimadzu, Japan). Magnetic measurements of the prepared nanocomposites were performed using a VSM (7410, Lake Shore, USA) at room temperature with an applied magnetic field of -20 to +20 kOe.

3. Result and discussions

3.1. XRD structural analysis

Fig. 1 shows the XRD patterns of A, B and C nanocomposites. The diffraction peaks can be indexed to a hexagonal wurtzite structured ZnO, which coincides with the standard JCPDS cards (No. 36-1451) [35-37, 47-54]. The average size is calculated using Scherrer formula, which can be described as follows:

$$D = \frac{0.89\,\lambda}{\beta\,Cos\,\theta}\ (m) \tag{1}$$

where, is the wavelength of the x-ray, 0 is the Bragg's diffraction angle and β is the FWHM [55-60]. The lattice constant (a) was calculated using the standard equation [39]:

$$a = d\sqrt{h^2 + k^2 + l^2} \tag{2}$$

where d is the inter-planner spacing and hkl is the Miller indices of the XRD diffraction plane.

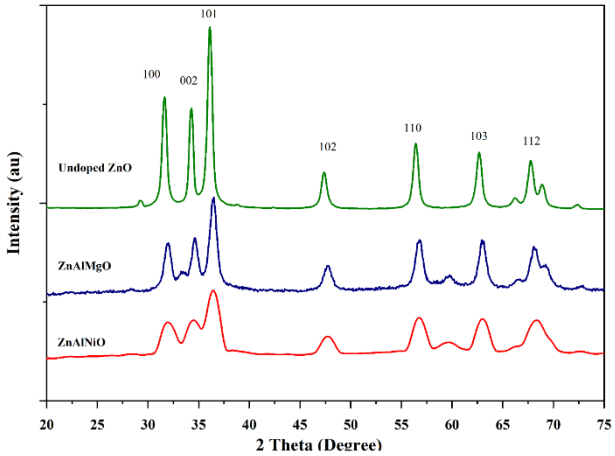

Fig. 1. XRD patterns of the prepared nanocomposites.

The lattice parameters of these samples are obtained by fitting the XRD data with the least square method, and the results can be seen in Table 1. It is found that the lattice parameters of co-doped ZnO are a little larger than that of un-doped one. The increased lattice parameters indicate that the co-doped ions of Mg^{2+}, Ni^{2+} and Al^{2+} may be co-doped into the ZnO crystal lattice via the substitution of the Zn^{2+} sites because of the large difference in ionic radius between the Zn^{2+} and co-dopants ions. Structural parameters such as average crystallite size, lattice constant, volume of unit cell and density are presented in Table 1.

Table 1. Crystallite size, lattice constant, volume of unit cell and density of the prepared nanocomposites.

Parameter	A	B	C
Crystallite size (nm)	25	19	11
Lattice constant (a) (Å)	5.2310	5.2912	5.3756
Volume of unit cell $(Å)^3$	143.13	148.13	155.33
Density (ρ_{xrd}) (g/cm^3)	7.7075	6.9408	6.7673

3.2. Surface morphology analysis

The TEM micrographs of the prepared nanocomposites (A, B and C) are shown in Fig. 2. It clearly indicates the particle size of the prepared nanocomposites is decreased. The TEM image of the sample A, B and C exhibits the rounded particles with average size 22, 15, 11 nm, respectively. Compared to the sample A, the change in co-doping ions (Mg^{2+}, Ni^{2+} and Al^{2+}) concentrations does not induce obvious change in morphology of the nanoparticles. But the average particle size of ZnO samples decreases from 22 to 11 nm as changing the doping concentrations of (Al^{2+}, Mg^{2+} and Ni^{2+}) which exhibits a good agreement with the XRD results (The average particle size of these samples decreases as change in co-doping ions (Al^{2+}, Mg^{2+} and Ni^{2+}) concentrations which could be attributed to the interference of the co-doing ions to the ZnO crystal growth (36, 41, 59, 60), The stoichiometry ratio of the elements present in the prepared nanocomposites (A to C) traced by XRF analysis is shown in Table 2. The XRF analysis confirmed that the prepared nanocomposites were composed of Zn, Al, Mg, Ni and O. Therefore, the obtained values of the atomic percentage of the constitute elements from XRF are close to the starting composition of the prepared nanocomposites.

Table 2. Elemental composition of the prepared nanocomposites.

Elements	A	B	C
Zn	43.92	43.53	43.72
O	56.08	55.71	55.50
Al	-	0.39	0.37
Mg	-	0.37	-
Ni	-	-	0.41
Total	100	100	100

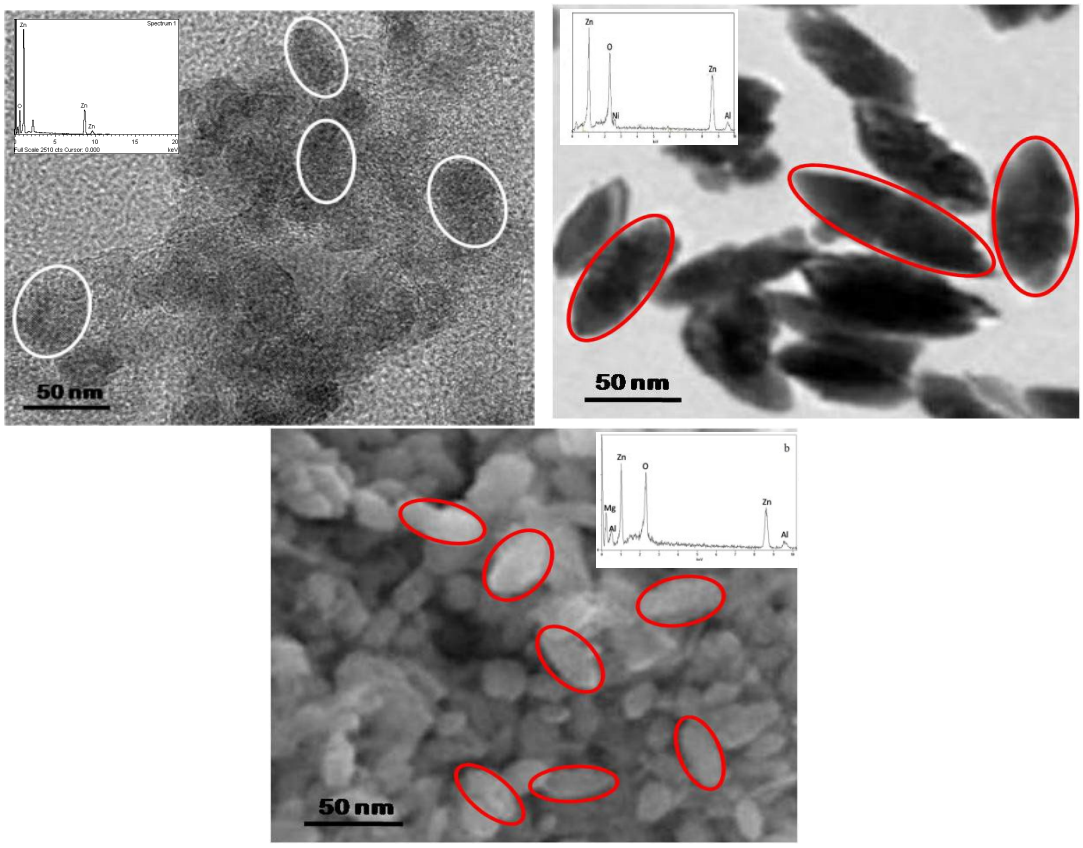

Fig. 2. TEM image of the prepared nanocomposites.

3.3. UV and PL analysis

Fig. 3(a) shows the LTV spectrum of the prepared nanocomposites. It is clear that the major absorption band of the prepared nanocomposites was observed at 325 nm represents the near-band-edge emission of pure ZnO (un-doped ZnO) NPs and shows the direct recombination of excitons. PL is one of the popular techniques for optical analysis of the prepared nanocomposites. PL analysis is mainly used for purity measuring emission properties such as surface defects, vacancies state of the samples.

Fig. 3(b) shows emission spectrum of pure ZnO NPs (un-doped ZnO) and it has shown ultraviolet and green region emission bands at 413 nm and 534 nm, respectively. Similarly, Fig. 3(c) shows emission spectrum of $Zn_{0.90}$ $Al_{0.05}Mg_{0.05}O$ NPs and it has shown ultraviolet, indigo, blue and green region. The emission bands were observed at 361 and 384 (ultraviolet), 421 (indigo), 468 (blue), and 535 nm (green). Further, Fig. 3(d) shows emission spectrum of $Zn_{0.90}$ $Al_{0.05}Ni_{0.05}O$ NPs and it has shown ultraviolet, blue and green region. The emission bands were observed at 379 and 403 (ultraviolet), 442 (blue), and 491 nm and 561 nm (green). The green emission peaks found photo-generated hole with an electron occupying the oxygen vacancy (55-58, 61, 62). Broad peak shows the presence of less intrinsic defects of Pure ZnO NPs.

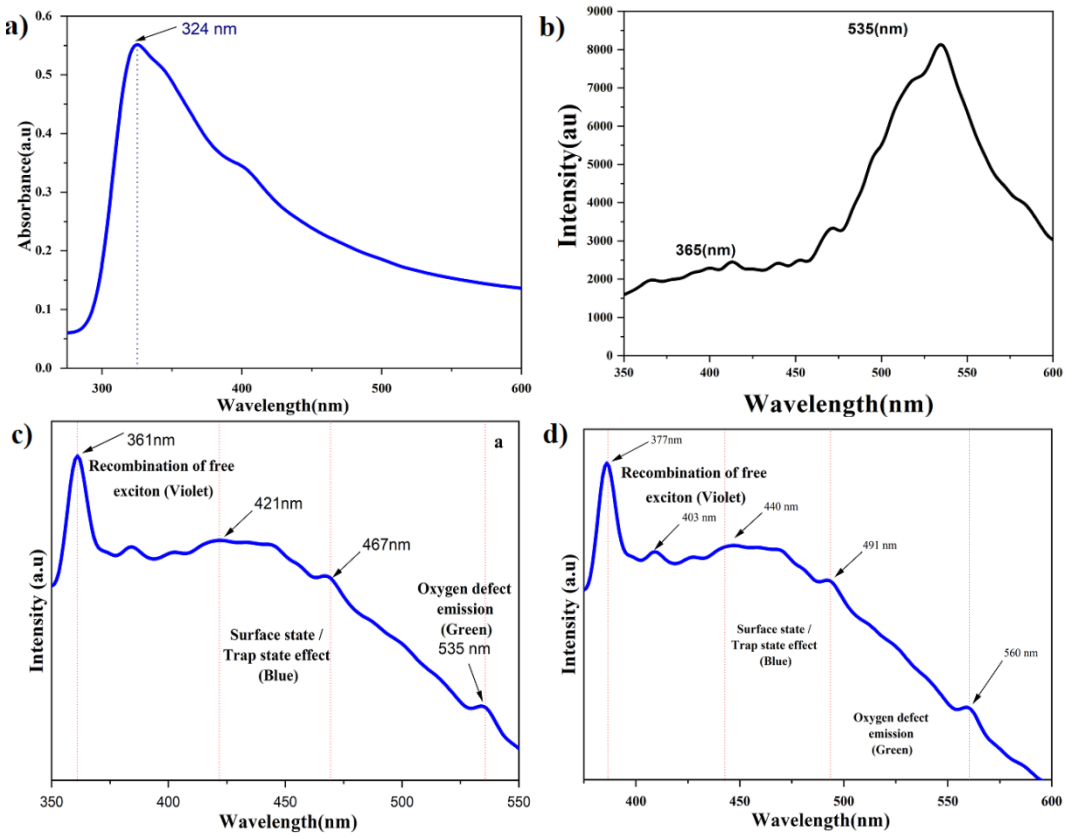

Fig. 3. UV-Visible spectra and PL of the prepared nanocomposites.

3.4. FT-IR analysis

FTIR is a powerful technique used to analyze the elemental constituents present and chemical bonding the in the prepared nanocomposites. In general, the chemical compositions of the material establish the absorption peaks and peak position of the spectra (34, 37). Fig. 4 represents the characteristic peaks of sample A, B and C between 500 cm^{1} and 3500 cm^{-1} using KBr method at room temperature. The peaks at 3386 cm^{-1}, 3492 cm^{-1} and 3520 cm^{-1} are attributed to O-H stretching vibration in the nanocrystalline of ZnO. The absorption peak at 2386 cm^{-1} is because of an existence of CO_2 molecule present in the atmosphere (34, 43).

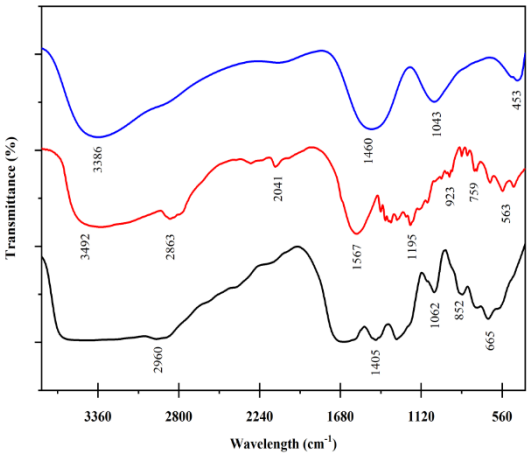

Fig. 4. UV-Visible spectra and PL of the prepared nanocomposites.

FTIR spectra below 1000 cm^{-1} describes the presence or absence of Zn-O/Cu-O/Ni-O bonds and their functional groups. The absorption bands between 780^{-1} and 830 cm^{-1} represents the micro structural formation by Cu-doping. The change in peak positions and peak intensity around 800 cm^{-1} correspond to the density of defects state surrounding Ni/Cu ions in ZnO network where Cu = 5% doped sample has sharp and high intensity absorption peak. The additional weak absorption peak cantered at 657 cm^{-1} corresponding to Cu-O phase is found to be dominant at Cu = 5% doped sample. The absorption peaks at 512 cm^{-1} and 406 cm^{-1} are assigned to stretching mode of Zn-O bonding in pure ZnO. In the present case, the absorption peaks varies from 521 cm^{-1} to 596 cm' where Cu = 1% doped sample has broad absorption peak around this region due to the distortion produced by initial doping of Cu into Zn-Ni-O. Further doping of Cu shift the absorption peaks towards the lower wave number side which is due to the change in the bond length by the replacement of Cu/Ni ions instead of Zn ions (44, 48). The peak at 3386 cm^{-1}, 3492 cm^{-1} and 2960 cm^{-1} represents the presence of hydrogen bonded O-H stretching mode for un-doped and co-doped ZnO NPs peak at 1460 cm^{-1}, 2041 cm^{-1} and 1405 cm^{-1}corresponds to the C=O stretching bending vibrations. The peak bond around 453 cm^{-1}, 563 cm^{-1} and 665 cm^{-1} illustrates the stretching vibration mode of ZnO (44, 53). The determined FTIR frequencies along with the vibrational assignments which are responsible for A, B and C at room temperature are given in Table 3.

Table 3. FTIR frequencies along with the vibrational assignments of the prepared nanocomposites.

Band Peaks	A	B	C
ZnO Stretching	453 cm^{-1}	665 cm^{-1}	563 cm^{-1}
AlO Stretching		852 cm^{-1}	759 cm^{-1}
MgO Stretching			923 cm^{-1}
NiO Stretching		1062 cm^{-1}	
C-O Stretching	1043 cm^{-1}		1195 cm^{-1}
C=O Stretching	1460 cm^{-1}	1405 cm^{-1}	2041 cm^{-1}
OH Stretching	3386 cm^{-1}	3520 cm^{-1}	3492 cm^{-1}

3.5. VSM analysis

Fig. 5 shows the M-H hysteresis loops of A, B and C nanocomposites measured at room temperature. In Figure the prepared nanocomposites exhibit obvious ferromagnetic behavior at room temperature. The saturation magnetization values are 10.033 emu/g, 15.049 emu/ g and 20.066 emu/ g and coercive force values are 255.45 Oe, 247.34 and 234.86 Oe for prepared nanocomposites A, B and C, respectively. The saturation magnetization of nanocomposites increase with the increasing of ZnO doping concentration compared with other co-doped samples (63-66).

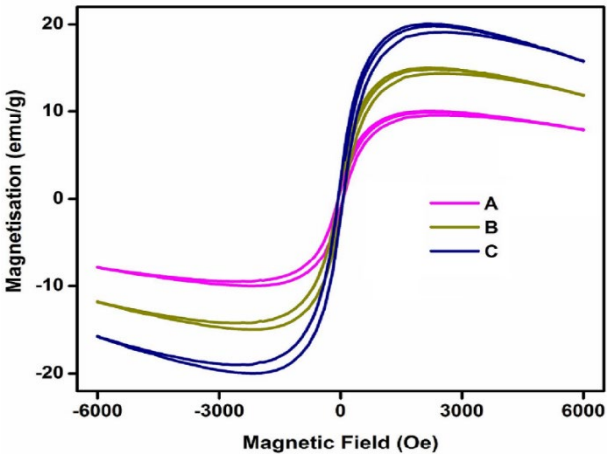

Fig. 5. VSM hysteresis behavior of the prepared nanocomposites.

At room temperature ferromagnetism of NPs can be explain by the two kind of mechanism, one is intrinsic magnetism are arise due to exchange interactions and other is extrinsic magnetism includes the formation of secondary phase elements. From XRD analysis clearly shows that no other peaks of impurity or secondary phases. EDAX study is also confirm the no other element present in prepared samples. Thus, from above results, the room temperature occurs only due to formation of clusters of transition elements or there is no evidence for secondary phase elements in prepared samples. Hence, the obtained room temperature ferromagnetism is an intrinsic magnetic property of un-doped ZnO (A), $Zn_{0.90}$ $Al_{0.05}Mg_{0.05}O$ (B) and $Zn_{0.90}$ $Al_{0.05}Ni_{0.05}O$ (C) composites. It is evident from the XRD results that the ZnO lattice is incorporated with Al, Mg and Ni. The view of Ni^{2+} ions substituted into ZnO lattice, the origin of magnetism in the nanocomposites is due to exchange interaction between local spin polarized electrons and the conductive electrons. The ferromagnetic ordering in the ZnO based materials is carrier mediated ferromagnetism, which depends on the type and carrier density of the magnetic depends. The measured magnetic values of prepared nanocomposites are given in the Table 4.

Table 4. Magnetic parameters of the prepared nanocomposites.

Properties	**A**	**B**	**C**
Coercivity (Oe)	134	73	130
Saturation Magnetization ($x10^{-3}$ emu/g)	14.926	7.876	11.670
Remanent Magnetization ($x10^{-3}$ emu/g)	4.43	1.49	1.87
Percentage remanence (%)	30%	19%	16%

4. Conclusions

Un-doped ZnO, Mg-Al co-doped ZnO and Ni-Al co-doped ZnO nanocomposites were synthesized by novel soft chemical method. XRD studies revealed that synthesized nanocomposites showed hexagonal wurzite structure. XRD studies suggest the formation of nanocomposites with average size of 22 nm, 15 nm and 11 nm respectively. FM & studies confirm the functional groups of the synthesized nanocomposites. The surface morphology of synthesized nanocomposites was observed from TEM image show elongated spherical shaped morphology and capsules-like shaped. The observed hysteresis loop shows soft ferromagnetic nature at room temperature. Un-doped, Mg-Al co-doped and Al-Ni co-doped nanocomposites of the present study having low magnetization can form diluted magnetic semiconductor for spintronics applications.

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