

Research Article

Optical and Magnetic Studies on Cu₂O/PANI Nanocomposite Prepared by Chemical Polymerization Method

K. Gopalakrishnan,¹ C. Ramesh,² M. Elango,¹ and M. Thamilselvan¹

¹ Department of Physics, Government College of Technology, Coimbatore, Tamil Nadu 641 013, India

² Department of Chemistry, Kandaswami Kandari's College, Velur, Tamil Nadu, India

Correspondence should be addressed to M. Thamilselvan; mthamil@rediffmail.com

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Polyaniline embedded green copper oxide (Cu₂O/PANI) nanocomposite has been synthesized through in situ chemical polymerization method in acidic medium at room temperature. The structural, optical, and magnetic properties of Cu₂O/PANI nanocomposite were investigated through Fourier transform infrared spectroscopy (FTIR), UV-Vis absorption spectra (UV-Vis spectra), scanning electron microscopy (SEM), photoluminescence spectra (PL), and vibrating sample magnetometer (VSM). FTIR spectra confirmed the formation of Cu₂O/PANI composite through the shifting of vibrational peaks of PANI and green Cu₂O nanoparticles at 825, 1142, 1299, 1499, 1573 cm⁻¹ and 695 cm⁻¹ respectively. SEM analysis revealed that many aggregations of well-separated irregular shape of Cu₂O nanoparticles with diameter about 15–40 nm exist in the composite matrix. Optical absorbance studies further confirmed the formation of composite through the blue shift of absorption peaks of PANI and diminishing intensity peak of Cu₂O. Cu₂O/PANI nanocomposite demonstrates semiconducting as well as diamagnetic behavior like PANI and Cu₂O nanoparticles. The nanocomposite exhibits high relative photoluminescence intensity in blue as well as green-yellow region of visible spectrum. The optical band gap value from absorption coefficient data is found to be 3.23 eV.

1. Introduction

In recent years, polymers with special functional groups are used as specific stabilizers for the synthesis of polymer based nanocomposites with different properties [1, 2]. Polymer and its nanocomposites possess unique characteristics when compared to other bulk materials. The structure of its nanocomposites is improved and controls their size range [3]. The combination of conducting polymers with metal oxide is used as alternate materials for optoelectronic applications. Amongst the family of conducting polymers, polyaniline is the most studied conducting polymer for the past 20 years due to its facile synthesis [4], chemical and environmental stability [5], and excellent electronic properties. Semiconducting nature of polyaniline is used to produce nanocomposite with inorganic metal oxide and such materials facilitate optical measurements. However, the synthesis of monodispersed metal oxide nanoparticles into the polymer matrix of its nanocomposites with adjustable

sizes and protected from photooxidation is a big challenge. In particular, it is difficult to generate highly enhanced luminescence in such polymer and its metal oxide nanocomposites systems. Luminescence from the polymer embedded Cu₂O nanoparticles is greatly enhanced and better stability is achieved from the composite compared to bulk materials. The wavelengths of the emission from the nanocomposites are continuously tuned within the blue range. The formation of nanocomposites is confirmed by photoluminescence (PL) spectroscopy and it is an important technique for finding the energy gap, purity crystalline quality detects, and so forth and is also used to analysis the quantum confinement of the materials [6, 7].

In this paper, optical and magnetic properties of Cu₂O/PANI nanocomposite prepared through the incorporation of Cu₂O nanoparticles in the polyaniline matrix are reported. The appropriate addition of Cu₂O nanoparticles into the polyaniline matrix enhances the optical properties and their performance. This work provides an easygoing and

effective route for the preparation of $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite.

2. Materials and Methods

The chemicals including copper (II) acetate monohydrate ($\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$), glacial acetic acid (CH_3COOH), aniline ($\text{C}_6\text{H}_5\text{NH}_2$), ferrous chloride (FeCl_2), hydrochloric acid (HCl), ammonia (NH_3), and hydrogen peroxide (H_2O_2) of analytical grade were used as received from Merck without further purification. Deionized water was used for all the stages of experiment. Whatman filter paper (number 42) was used for filtration process.

Glacial acetic acid (1 mL) was dissolved in 50 mL deionized water and stirred for 10 minutes. Under stirring, copper (II) acetate monohydrate (3.325 g) was added with the above solution and blue coloured Barfoed's solution was obtained. About 20 g taxonomically authenticated healthy *Tridax procumbens* leaf was cut into fine pieces and boiled with 100 mL double distilled water in Erlenmeyer flask for 8–10 min. The extract was then cooled at room temperature and filtered.

Initially, 10 mL of *Tridax procumbens* leaf extract was added to 10 mL of Barfoed's solution and the colour of the solution was changed from blue to brick red after 10 minutes which indicated the formation of Cu_2O nanoparticles. The solid product was filtered and washed with ethanol and then dried at room temperature for further use.

Initially, the nonaqueous aniline monomer (2.29 g) was dissolved in 300 mL of 1 N hydrochloric acid and mixed with 0.0064 g of ferrous chloride dissolved in 75 mL of water. Under stirring, 68 mL (6 wt.%) of hydrogen peroxide was added drop by drop to this mixture for 30 min in which 0.57 g of synthesized green Cu_2O nanoparticles was added. The reaction was continued for 23 h at constant stirring at room temperature. The green colored product was filtered, washed, and well-dried for further characterization.

3. Characterization

Fourier transform infrared (SHIMADZU) spectrum was recorded in the wavenumber range of 4000–400 cm^{-1} at the scanning rate of 25 scan/min with a resolution of 4 cm^{-1} using KBr pellet. The morphological properties were examined by LEO 1455 VP scanning electron microscope (SEM) operating at 20 kV. The optical absorption spectra were recorded in the range from 190 to 2500 nm using UV-1800 double beam UV-Vis spectrophotometer. The fluorescence spectra were recorded at room temperature on a Horiba Jobin spectrofluoromax 4, USA. The magnetic behavior of the samples were studied using Lakeshore USA model 7404 vibrating sample magnetometer.

4. Result and Discussion

The functional groups of polyaniline, green Cu_2O nanoparticles, and $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite are illustrated in Figure 1. The spectrum of polyaniline (Figure 1(a)) acquires

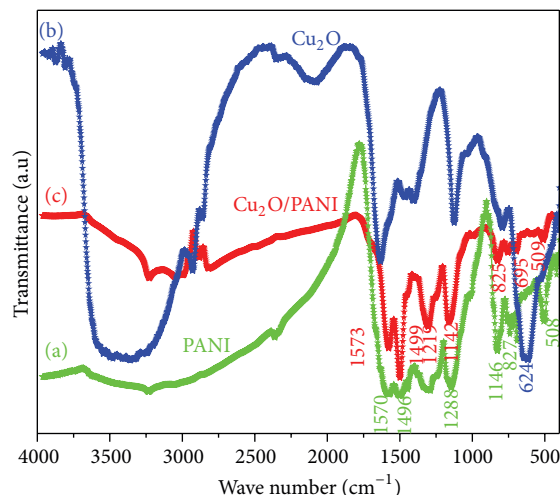


FIGURE 1: FTIR spectra for (a) polyaniline, (b) Cu_2O , and (c) $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite.

the main characteristic peaks at 1570, 1496, 1288, 1146, and 827 cm^{-1} . The bands at 1570, 1496, and 1288 cm^{-1} are assigned to the stretching mode of N=Q=N ring, N-B-N ring, and C-N ($\text{C}_{\text{aromatic}}-\text{N}$) deformation, respectively [8] (where B refers to benzenoid ring and Q refers to quinonoid ring). The observed band at 827 cm^{-1} attributes to the out-of-plane vibration of C-H on 1,4-disubstituted aromatic rings. The characteristic peak 624 cm^{-1} in the spectrum (Figure 1(b)) of Cu_2O shows the bending vibrations of the Cu_2O crystal lattice. The spectrum (Figure 1(c)) of nanocomposite represents the stretching vibration of Cu_2O which shifted from 624 cm^{-1} to 695 cm^{-1} which shows the presence of Cu_2O in the polyaniline matrix.

The SEM images of polyaniline, Cu_2O , and $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite are shown in Figure 2. The partial crystalline nature of polyaniline illustrates cauliflower-like morphology. The image of Cu_2O demonstrates well-separated irregular shape of Cu_2O nanoparticles without agglomeration. $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite (Figure 2(c)) exposes that many aggregations of agglomerated Cu_2O with diameter in the range of 15–40 nm exist in the composite matrix. This indicates the incorporation of Cu_2O in polyaniline matrix during the formation of nanocomposite which is well agreed with the result of UV analysis.

Optical spectroscopy is an important technique to understand the conducting states corresponding to the absorption bands of inter- and intragap states of conducting polymers [9]. Figure 3 shows the optical absorbance with respect to wavelength for polyaniline, Cu_2O , and $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite. Figure 3(a) illustrates the major absorption peaks at 300, 332, and 448 nm. The observed bathochromic shift at the intense band 300 nm is due to the $\pi-\pi^*$ transition of benzenoid ring which is related to the extent of conjugation between the adjacent phenylene rings in the polymeric chain and the forced planarization of π -system induced by aggregation. It leads to increased conjugation and thus lowers the band gap [10] which is well agreed with

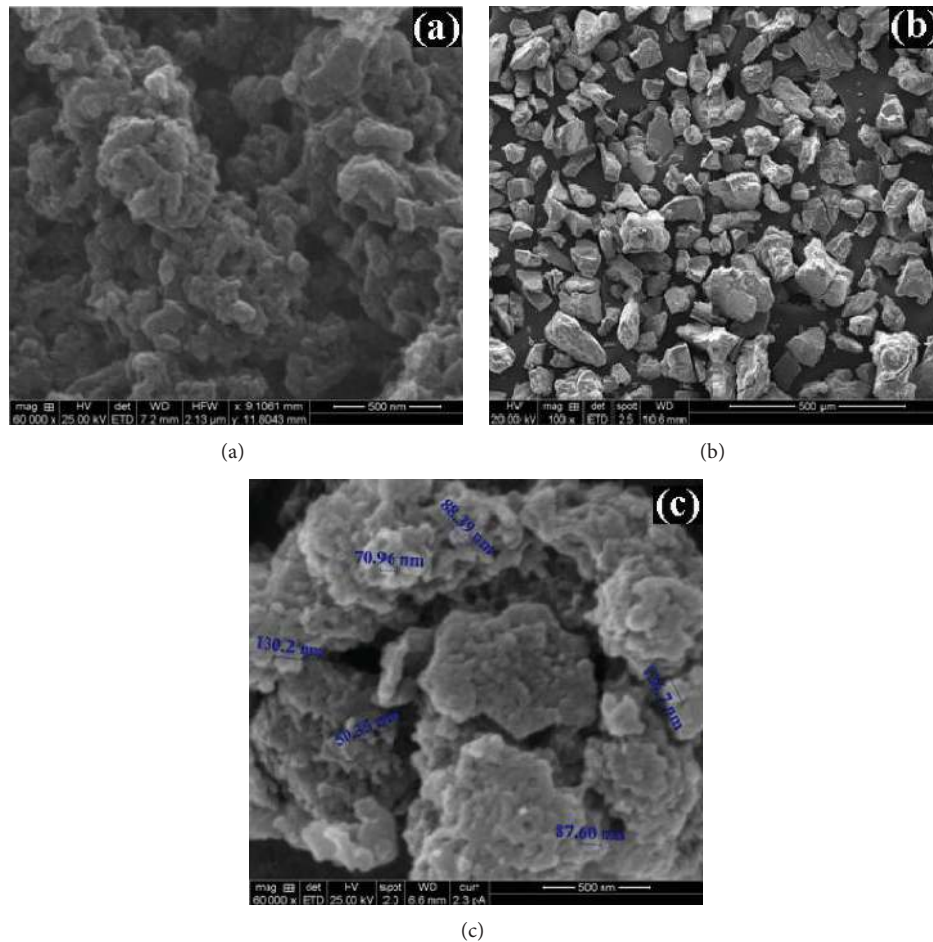


FIGURE 2: SEM images for (a) polyaniline, (b) Cu_2O , and (c) $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite.

the band gap result obtained in the polyaniline. The transition of $\pi-\pi^*$ of benzenoid ring and the formation of polaron band in the nanocomposites are responsible for increase of the electrical conductivity of the nanocomposites [11]. The less intense band at 332 nm is due to $\pi-\pi^*$ transition. The peak at 448 nm is due to polaron- π^* transition and shift of electron from benzenoid ring to quinonoid ring [12]. Figure 3(b) of Cu_2O shows the major peaks at 257 and 302 nm. Figure 3(c) demonstrates the high intense blue shift of absorption peaks of polyaniline from its actual position in the $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite which indicates that the addition of Cu_2O filler particles in the polyaniline matrix has large influence on absorption spectra in the $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite. The less intense absorption peaks of Cu_2O are noticed with in the nanocomposite which indicates the interfacial interaction of Cu_2O with polyaniline and this shows the formation of polaron and bipolaron charge carrier in the nanocomposite when compared to PANI. The substitutions in the ring affect the tensional angle; hence dihedral/torsional angle between adjacent aromatic rings is increased [13]. It causes the increase in twisting of torsional angle which increases the band gap as compared with the band gap of polyaniline. This observation is in fair agreement with others [14, 15].

The estimation of size of the particles is confirmed further by evaluating it with Meulenkamp equation [16]. The value of $\lambda_{1/2}$ (the wavelength, at which the absorption is 50% of that at the excitonic peak in nm) is determined and the size of the particles is calculated by using an empirical formula

$$1240 = \lambda_{1/2} \left(3.301 + \frac{294}{D^2} + \frac{1.09}{D} \right). \quad (1)$$

The size of the particles evaluated from Meulenkamp equation is in good agreement with the values observed in SEM analysis.

The optical energy plays a crucial role in the utilization of the materials in the optoelectronic applications. The band gaps of polyaniline, Cu_2O , and $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite are measured from the absorption coefficient data as a function of wavelength using Tauc relation. The optical band gaps of polyaniline, Cu_2O , and $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite obtained from Figure 4 are 2.06 eV, 3.12 eV, and 3.23 eV. The increase of band gap of Cu_2O compared with its bulk band gap 2.17 eV is due to the presence of bioorganic phases on Cu_2O . The increase in band gap of $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite as compared with PANI is due to the formation of polaron in the nanocomposites. The lower band gap value originates

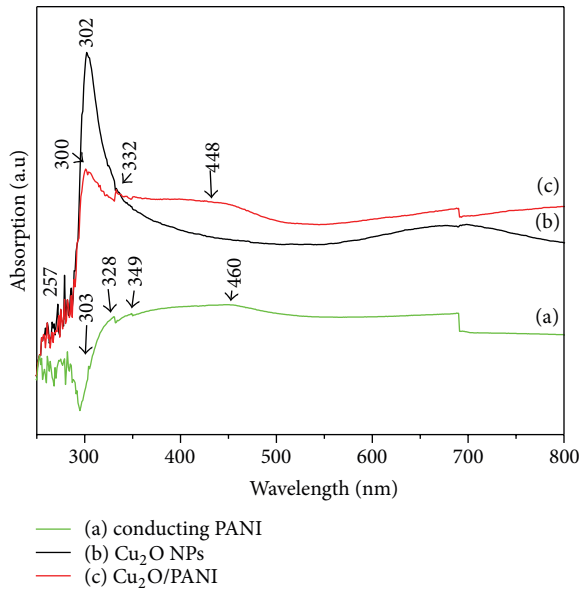


FIGURE 3: UV-Vis spectra for (a) polyaniline, (b) Cu₂O, and (c) Cu₂O/PANI nanocomposite.

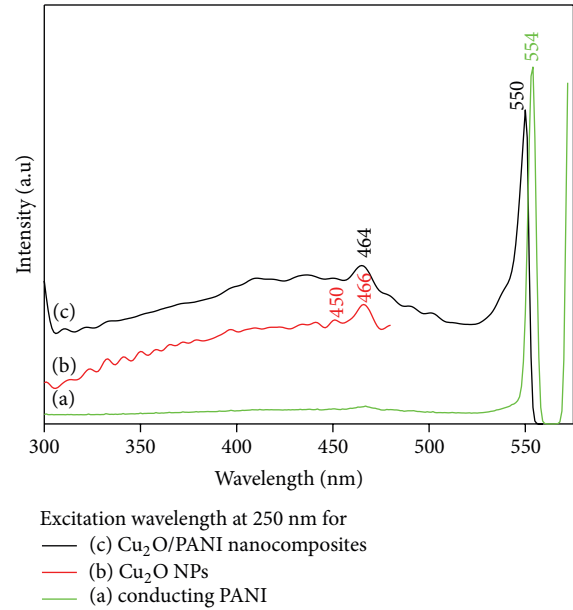


FIGURE 5: PL spectra for (a) polyaniline, (b) Cu₂O, and (c) Cu₂O/PANI nanocomposite at the excitation wavelength of 250 nm.

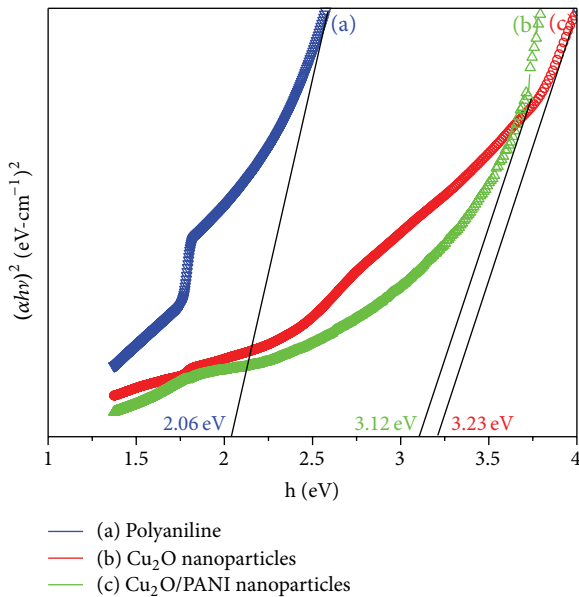


FIGURE 4: Relation between $(\alpha hv)^2$ and $h\nu$ for (a) polyaniline, (b) Cu₂O, and (c) Cu₂O/PANI nanocomposite.

from the quantum confinement effect which is caused by the nanodimensional state of materials [17].

The photoluminescence is a technique much used to characterize the defects of a semiconductor and mainly used to study the exciton spectra in conducting polyaniline, Cu₂O and its nanocomposite. Figures 5–7 illustrate the comparison of emission spectra of polyaniline, Cu₂O, and Cu₂O/PANI nanocomposite at the excitation wavelength of 250 nm, 280 nm, and 310 nm, in order to evidence the contribution of polyaniline and Cu₂O in the nanocomposites.

The photoluminescence (PL) spectra for polyaniline are illustrated in Figures 5(a), 6(a), and 7(a) at different excitation wavelengths of 250 nm, 280 nm, and 320 nm. It is noticed that the red shift at the peak 557 nm is observed in the polyaniline at the excitation wavelength of 250 nm. In addition to this peak, another green shift at 467 nm with other weaker peaks is observed at the excitation wavelength of 280 and 320 nm. The major peak at 467 nm arises due to transitions from the polaronic band to the π -band (HOMO) structures of polyaniline [18] and the emission wavelength is shifted towards the blue when compared to the bulk polyaniline. This emission is due to the recombination of electron and hole pairs. The blue shift of polyaniline demonstrates the strong quantum confinement. The appearance of many weaker peaks in the polyaniline at the excitation wavelength of 280 nm and 320 nm is due to increase of density of states.

Cu₂O frequently displays photoluminescence; the particles obtained in the Cu₂O nanoparticles are emitted in the blue to green-yellow portion of the spectra as shown in Figures 5(b), 6(b), and 7(b). Nevertheless, apart from those of excitons, two major peaks at 450 nm and 467 nm are emerged in the emission spectra of 250 nm with some other weaker peaks, but the intensities of major and weak peaks are increased with little shift at the excitation wavelength of 280 nm and 320 nm. This shows the increase of density of states. The band at 467 nm is always present and clearly visible in the entire excitation wavelength and therefore it is attributed to the Cu vacancy level. The observed luminescence band at 467 nm is due to the recombination of relaxed excitons and therefore its luminescence intensity is increased at the excitation wavelength of 320 nm. But this is not due to the direct recombination of carriers into the defect levels. Notice that, near the band at 467 nm, other weaker bands appear at 451 nm, 482 nm, and 492 nm at the excitation

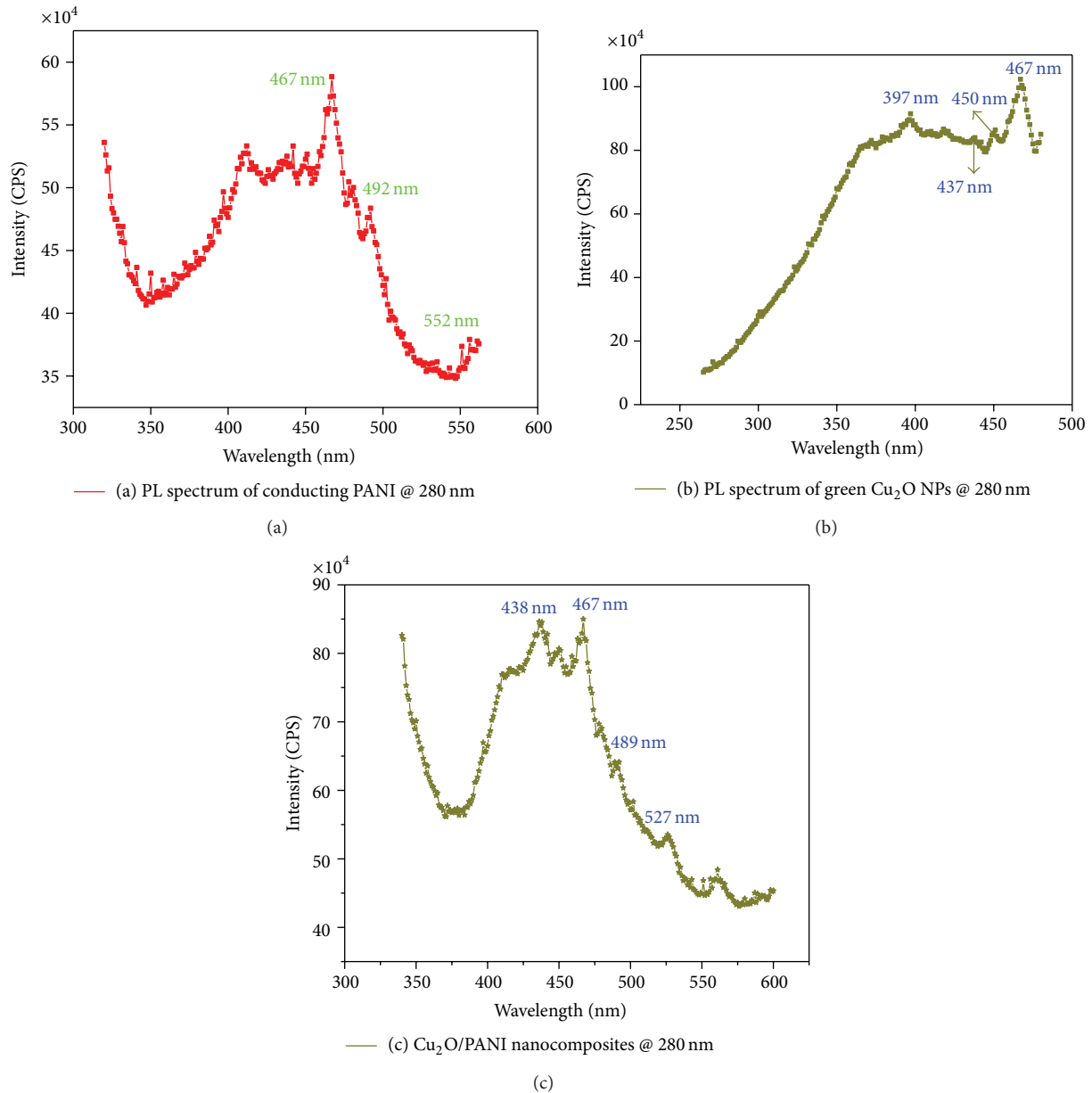


FIGURE 6: PL spectra for (a) polyaniline, (b) Cu_2O , and (c) Cu_2O /PANI nanocomposite at the excitation wavelength of 280 nm.

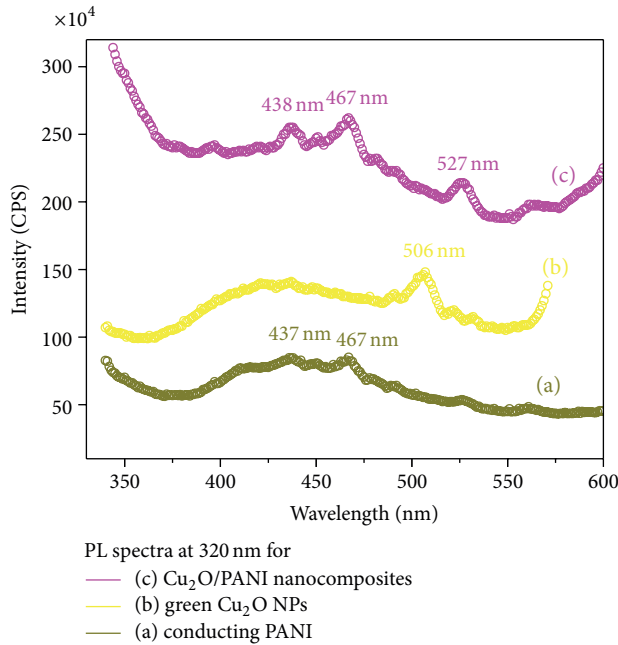
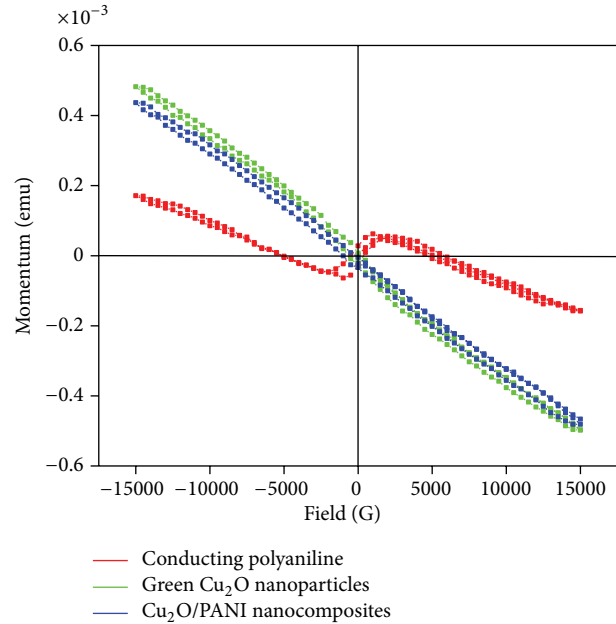
wavelength of 280 and 320 nm. The peaks are related to the crystalline defects introduced during the growth of CuO_2 . The oxygen vacancies interact with interfacial Cu vacancies leading to the formation of a considerable amount of trapped states within the band gap, which results in a PL signal in CuO_2 nanoparticles [19].

PL spectra for Cu_2O /PANI nanocomposite are illustrated in Figures 5(c), 6(c), and 7(c) at different excitation wavelengths of 250 nm, 280 nm, and 320 nm. Major peaks at 467 nm and 550 nm are observed at the excitation wavelength of 250 nm. In addition to these major peaks, other broader peaks at wavelength of 438 with some other weaker peaks appear at the excitation wavelength of 280 and 320 nm. It is apparent from Figures 5(c), 6(c) and 7(c) that the PL

intensity is increased in both blue and green-yellow regions of visible spectrum, which may be ascribed to the formation of more density of states in the energy band upon addition of CuO_2 nanoparticles. There are electron donating groups such as =NH in polyaniline, CuO_2 is an electron conducting semiconductor, and this combination enhances the electron mobility in the nanocomposite. This in turn favors the formation of singlet excitons. The singlet exciton states so formed due to decay radiatively to the ground state resulting in enhanced photoluminescence [20]. The relative intensity at 437 nm of CuO_2 PL peak is considerably low as compared to the relative peak intensity of polyaniline, so it is not prominent in the nanocomposite and it broadens the peak due to increase of the size of the particles in the nanocomposites.

TABLE 1: Magnetic parameters of conducting PANI, Cu₂O nanoparticles, and Cu₂O/PANI nanocomposites.

Materials	Positive site				Negative site			
	Ms $\times 10^{-6}$ emu/g	Mr $\times 10^{-6}$ emu/g	Hc G	SQR $\times 10^{-3}$	Ms $\times 10^{-6}$ emu/g	Mr $\times 10^{-6}$ emu/g	Hc G	SQR $\times 10^{-3}$
Polyaniline	-155	63	474	406	170	63	483	370
Cu ₂ O nanoparticles	-491	21	203	34	42	26	682	54
Cu ₂ O/PANI nanocomposite	-483	32	60	36	66	40	1161	92

FIGURE 7: PL spectra for (a) polyaniline, (b) Cu₂O, and (c) Cu₂O/PANI nanocomposite at the excitation wavelength of 320 nmFIGURE 8: Magnetic behaviour of PANI, Cu₂O, and Cu₂O/PANI nanocomposite.

The increase of size of the particles in the nanocomposite is due to intercalation of nanoparticles in the polyaniline matrix. The PL studies of Cu₂O/PANI nanocomposite show that the emission peaks of polyaniline and Cu₂O are presented with peak bordering and diminishing intensities which confirm the increase of the size of the particles in the nanocomposites which is well agreed with the result of morphology of the nanocomposite.

Figure 8 shows a typical hysteresis loop obtained for polyaniline, Cu₂O, and Cu₂O/PANI nanocomposite at room temperature. The measurement of magnetic properties of polyaniline is important to know what the type of magnetic material is and how the electrical conduction occurs. Of these properties, the parameters of magnetic hysteresis are significant factors in determining the nature of magnet and the utilization of the materials for recording medium. It is obvious from the hysteresis loop of polyaniline that the magnetic moment increases with increase of magnetic field at lower value, but it decreases at higher magnetic field. Hence the nonmagnetic polyaniline [21] exhibits diamagnetic nature in higher magnetic field. The exhibition of diminutive magnetic nature of polyaniline at low field is due to increase

of solvent and organic acid [22]. The squareness ratio (SQR = Mr/Ms) is an important assessment of the quality of the magnetic materials and its value of polyaniline is found to be 188×10^{-3} indicating that polyaniline is not having the properties of recording medium. The saturation magnetization (Ms) and retentivity (Mr) of polyaniline are found to be 171×10^{-6} emu/g and 28×10^{-6} emu/g, respectively. Figure 8(b) exhibits a typical hysteresis loop obtained for Cu₂O prepared by leaf extracts and it exhibits the diamagnetic behaviour. This is due to Cu⁺ ion electron structure $3d^{10}4s^0$, with $4s$ orbital only slightly higher in energy than the $3d$ levels [23]. This means that the bands have the same parity and no lone pair electron is found.

The hysteresis loop obtained for Cu₂O/PANI nanocomposite shows nonmagnetic nature and hysteresis loop of Cu₂O/PANI nanocomposite is shifted slightly due to incorporation of Cu₂O in the matrix of polyaniline. The saturation magnetization (Ms), retentivity (Mr), coercivity (Mc), and SQR value of polyaniline, Cu₂O, and Cu₂O/PANI nanocomposite are shown in Table 1. It is observed that the magnetic parameters of the nanocomposites exhibit diamagnetic nature as that of Cu₂O, since the incorporation of Cu₂O into

polyaniline matrix does not play to change the diamagnetic nature of the Cu_2O and polyaniline.

5. Conclusions

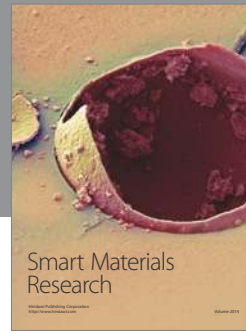
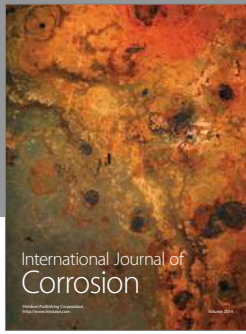
Polyaniline embedded green Cu_2O nanocomposite was synthesized by using in situ chemical polymerization method. Studies of UV-Vis absorption and SEM image reveal the incorporation of Cu_2O nanoparticles with diameter about 40 nm in the polyaniline matrix. Addition of Cu_2O into polyaniline matrix increases the band gap (3.23 eV) of the nanocomposite when compared to polyaniline. Magnetic study suggests that polyaniline does not improve the non-magnetic nature of the Cu_2O . The size of the particles observed from SEM is in fair agreement with the sizes calculated by E. A. Meulenkamp equation. The photoluminescence spectrum of nanoparticles shows a broad luminescence in green region which indicates that the nanocomposite could be used for the fabrication of photonic devices.

Conflict of Interests

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

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