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A comparative study of the effect of solvents on the optical, structural and morphological properties of ZnO–GO nanocomposites synthesized by sol-gel method

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Zinc Oxide-Graphene Oxide (ZnO–GO) nanocomposites were prepared using solvents polyvinylpyrrolidone (PVP) and N-Methyl-2 Pyrrolidone (NMP) by sol-gel technique and their optical, structural and morphological properties were investigated. X-ray Diffraction (XRD) studies show the presence of planes of GO and ZnO confirming the formation of composites. The particle sizes were calculated using Scherrer's formula and were found to be in the nanometer range. Scanning Electron Microscopy (SEM) images show the formation of layered structures dispersed non-uniformly over clusters of particles. Energy Dispersive X-Ray (EDX) spectra confirm the presence of carbon, zinc and oxygen in the composites. The optical absorbance of ZnO–GO synthesized using PVP was higher than ZnO–GO with NMP with the absorption edge shifting to shorter wavelength in the presence of NMP. The band gap values were found to be in the range of 2.7–3.0 eV. The band gap of ZnO–GO synthesized using NMP was higher than ZnO–GO synthesized using PVP.

Keywords: GO, ZnO, ZnO–GO Nanocomposite, PVP, NMP.

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1. Introduction

Zinc Oxide (ZnO), a wide band gap II–VI semiconductor has been a subject of extensive research owing to its numerous interesting properties, making it a desirable material for application in conductive oxide [1], solar cell [2], display and sensors [3, 4], and photocatalysis [5, 6]. However, pure ZnO suffers from weak photo response [7], fast recombination of electron-hole pairs [8] and low operating speed.

GO is a layer of graphene functionalized with oxygen-containing moieties, such as hydroxyl (OH), carbonyl (C=O) and alkoxy (C–O–C) groups. It possesses unique properties that are different from graphene due to the existence of various oxygenated functional groups on the surface of GO. Graphene Oxide (GO) has attracted researchers due to its high surface area, high mobility of charge carriers and excellent stability. Hybrid of GO and ZnO (nanocomposites) can offer better prospects to enhance the photoresponsivity, stability and flexibility of ZnO for various applications [9, 10].

ZnO can be prepared by different means, of which the sol-gel is a popular method because of its low cost, reliability, reproducibility, simplicity. The choice of solvents used for reaction and capping the particles affects the shape and size of the composites [11]. Although there have been several reports on the synthesis of these composites using different solvents, a comparative study of the effect of solvents on the properties of the composites have not been presented to date. In the present work ZnO–GO nanocomposites were prepared using two polymer solvents – PVP and NMP. There are reports that a polymer matrix is useful in the formation of nanoparticles as it possesses the properties of the host polymer as well as the guest nanoparticles [12]. The polymers can also help in easier shaping and formation of the composite materials. PVP is a good surface stabilizer, growth modifier and particle disperser [13]. It also inhibits agglomeration by steric effect [14]. NMP is a 5 member ring compound containing nitrogen. It has high dissolving power and high purity. It can dissolve organic and inorganic compounds well or even better than chlorofluorocarbon (CFC) solvents. This paper presents the structural, morphological and optical characterization of ZnO–GO nanocomposites prepared using PVP and NMP as capping agents.

2. Experimental techniques

2.1. Synthesis of Zinc Oxide (ZnO)

ZnO nanoparticles were synthesized by sol gel method. The appropriate quantity of Zinc acetate was dissolved in deionized water and mixed with an equal quantity of aqueous 0.05 M NaOH solution slowly with continuous stirring at 50 °C. The reaction mixture was maintained at this temperature for 2 hours and then cooled to room temperature. The resultant precipitate was centrifuged and then washed with deionized water and dried at room temperature.

2.2. Synthesis of Graphene Oxide (GO)

Graphene oxide (GO) was synthesized using modified Hummers method from pure graphite powder [15]. First, 108 ml of sulphuric acid (H_2SO_4) and 12 ml of phosphoric acid (H_3PO_4) (volume ratio 9:1) were mixed and stirred using ultrasonic bath (53 KHz, 80 W) for several minutes. 0.9 g of graphite powder was added into the solution under stirring condition. The mixture was ultrasonicated for an hour and then kept in an ice bath keeping the temperature at approximately 5°C . Then, 5.28 g of potassium permanganate (KMnO_4) was then added slowly into the solution. This mixture was stirred under ultrasonic irradiation for 6 hours until the solution became dark green and thereafter was magnetically stirred for 1 hr after removing from the ice bath. To eliminate excess of KMnO_4 , 2.7 ml of hydrogen peroxide (H_2O_2) was dropped slowly and stirred for 10 minutes. On adding hydrogen peroxide, the residual KMnO_4 and MnO_2 was reduced to soluble salts. The solution was cooled and 20 ml of hydrochloric acid (HCl) and 60 ml of deionized water (DIW) was added and centrifuged for 7 minutes. The suspension was filtered using high quality Whatman filter paper. The residue obtained after decantation was washed with HCl and DIW for 3 times to remove the metal ions. The filtrate was again mixed with DIW several times so as to remove any other impurities and then ultrasonicated for 1 hr to obtain dispersion of GO. The washed GO solution was dried using oven at 90°C for 24 hours to produce the GO powder.

2.3. Synthesis of ZnO–GO nanocomposites

ZnO–GO nanocomposites were synthesized with two different ratios of ZnO to GO – namely $\text{ZnO}_{0.5}\text{–GO}_{0.5}$ and $\text{ZnO}_{0.7}\text{–GO}_{0.3}$. 0.5 g graphene oxide and 0.1 g PVP/NMP was dispersed in 500 ml deionised water to form graphene oxide solution. 0.5 g ZnO nanoparticles were added into graphene oxide solution and the mixture was stirred at room temperature for 2 hrs. The resultant precipitate was filtered and thoroughly washed with deionized water to remove impurities and dried at 80°C for 5 hrs to obtain $\text{ZnO}_{0.5}\text{–GO}_{0.5}$ nanocomposites. $\text{ZnO}_{0.7}\text{–GO}_{0.3}$ nanocomposites were also synthesized using the above method.



FIG. 1. ZnO–GO nanocomposites

2.4. Characterization Methods

XRD studies were performed at NIT Raipur using PANalytical 3KW X'pert powder – Multifunctional X-ray diffractometer, SEM and EDX studies were also carried out at NIT Raipur using ZEISS EVO 18 Scanning Electron Microscope and INCA 250 EDS with X-MAX 20 nm detector. Optical absorbance spectral studies were done using ELICO-SL210 UV-VIS spectrophotometer.

3. Results and discussion

3.1. Absorbance spectra

Fig. 2(a) depicts the optical absorbance spectra of $\text{ZnO}_{0.5}\text{–GO}_{0.5}\text{–PVP}_{0.1}$ and $\text{ZnO}_{0.5}\text{–GO}_{0.5}\text{–NMP}_{0.1}$ while Fig. 2(b) shows the absorbance spectra of $\text{ZnO}_{0.7}\text{–GO}_{0.3}\text{–PVP}_{0.1}$ and $\text{ZnO}_{0.7}\text{–GO}_{0.3}\text{–NMP}_{0.1}$ nanocomposites. The absorption edge in these composites is around 420 nm for those prepared with PVP while in the case of composites

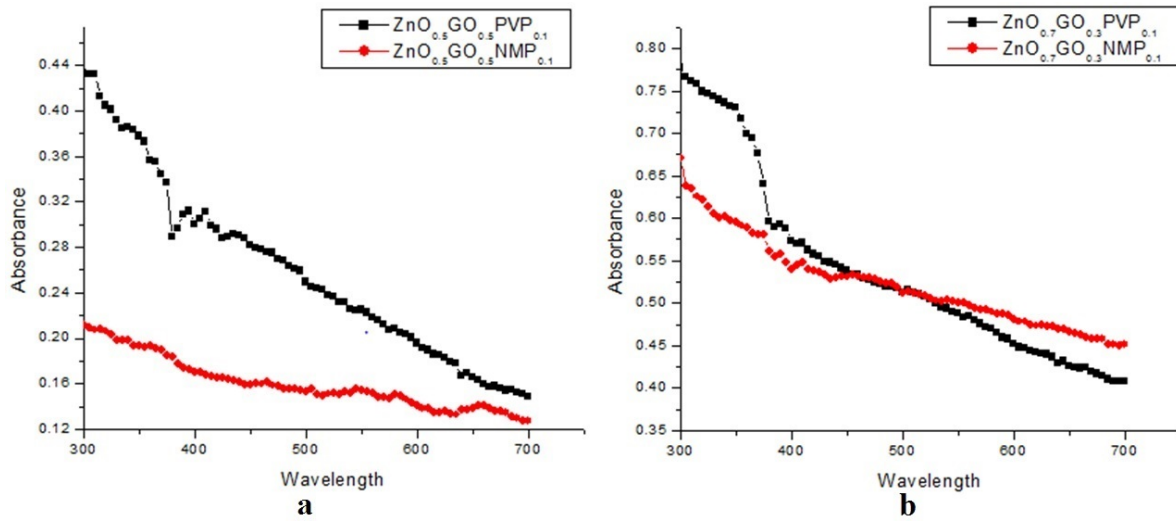


FIG. 2. (a) Absorbance spectra of $ZnO_{0.5}-GO_{0.5}-PVP_{0.1}$ and $ZnO_{0.5}-GO_{0.5}-NMP_{0.1}$ Nanocomposites; (b) Absorbance spectra of $ZnO_{0.7}-GO_{0.3}-PVP_{0.1}$ and $ZnO_{0.7}-GO_{0.3}-NMP_{0.1}$ Nanocomposites

prepared with NMP, the absorption edge shifts to shorter wavelength around 400 nm. This is in agreement with other reports on the absorption edge of GO-ZnO composites [8, 16]. Tauc's plots (plot between $(\alpha h\nu)^2$ vs $h\nu$ where α is the absorption coefficient) were used to determine the band gap of the material from the absorbance data. Fig. 3(a) represents the Tauc's plot of $ZnO_{0.5}-GO_{0.5}-PVP_{0.1}$ and $ZnO_{0.5}-GO_{0.5}-NMP_{0.1}$ and Fig. 3(b) shows the Tauc's plot of $ZnO_{0.7}-GO_{0.3}-PVP_{0.1}$ and $ZnO_{0.7}-GO_{0.3}-NMP_{0.1}$. The corresponding values of band gap are presented in Table 1 and are found to be in the range between 2.7 and 3.0 eV. The band gap values of composites prepared with NMP are higher than those with PVP.

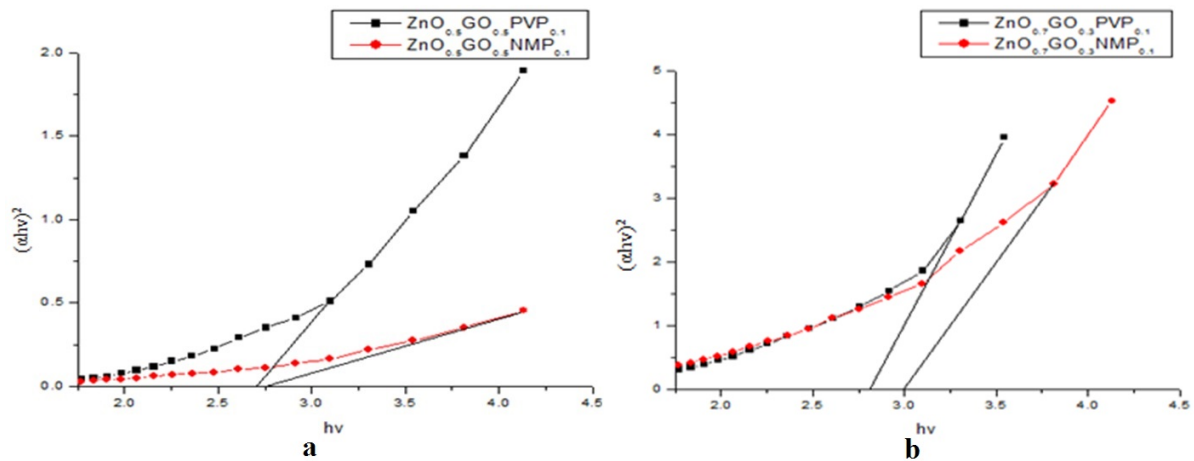


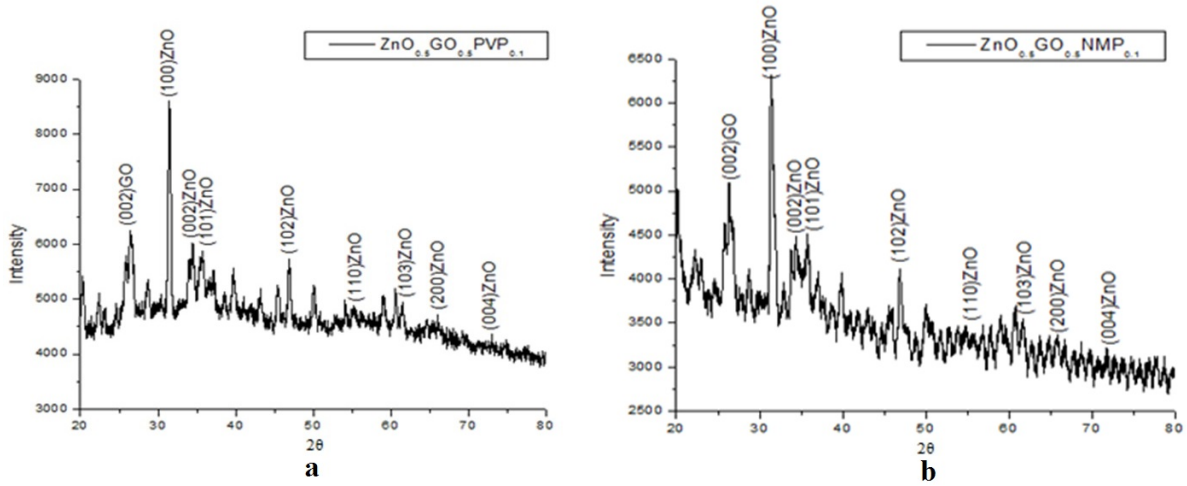
FIG. 3. (a) Tauc's plot of $ZnO_{0.5}-GO_{0.5}-PVP_{0.1}$ and $ZnO_{0.5}-GO_{0.5}-NMP_{0.1}$ Nanocomposites; (b) Tauc's plot of $ZnO_{0.7}-GO_{0.3}-PVP_{0.1}$ and $ZnO_{0.7}-GO_{0.3}-NMP_{0.1}$ Nanocomposites

3.2. XRD Studies

Figure 4(a) and 4(b) represent the X-ray diffractograms of $ZnO_{0.5}-GO_{0.5}-PVP_{0.1}$ and $ZnO_{0.5}-GO_{0.5}-NMP_{0.1}$ and the corresponding data are presented in Tables 2 and 3 respectively. The assignments of the different peaks were made by comparison with JCPDS data of GO and JCPDS data 36-1451 of ZnO and calculation of lattice constants, which showed agreement with the reported values. In both the cases, maximum intensity corresponds to the (100) plane of ZnO. The other prominent planes corresponding to ZnO are (002), (101), (102), (110), (103), (200), (004) which have been observed in both the cases. Diffraction line (002) of GO is also observed.

TABLE 1. Band Gap values of ZnO–GO Nanocomposites

S No.	Sample	Band Gap (eV)
1	ZnO _{0.5} GO _{0.5} PVP _{0.1}	2.7
2	ZnO _{0.5} GO _{0.5} NMP _{0.1}	2.75
3	ZnO _{0.7} GO _{0.3} PVP _{0.1}	2.8
4	ZnO _{0.7} GO _{0.3} NMP _{0.1}	3.0

FIG. 4. (a)X-ray diffractogram of ZnO_{0.5}–GO_{0.5}–PVP_{0.1} Nanocomposites; (b)X-ray diffractogram of ZnO_{0.5}–GO_{0.5}–NMP_{0.1} NanocompositesTABLE 2. XRD data of ZnO_{0.5}–GO_{0.5}–PV_{0.1} Nanocomposites

d Value(A ⁰)		Relative Intensity		hkl	Lattice Constant c (A ⁰)	
(Obs)	(Rep)	(Obs)	(Rep)		(Obs)	(Rep)
3.3714	3.3608	–	–	(002)GO	–	–
2.8392	2.8143	100	57	(100)ZnO	a = 3.278	a = 3.25, c = 5.207
2.6031	2.6033	70	44	(002)ZnO	c = 5.206	a = 3.25, c = 5.207
2.5104	2.4759	68.24	100	(101)ZnO	a = 3.308, c = 5.206	a = 3.25, c = 5.207
1.9347	1.9111	66.69	23	(102)ZnO	a = 3.339, c = 5.206	a = 3.25, c = 5.207
1.6327	1.6247	55.52	32	(110)ZnO	a = 3.265	a = 3.25, c = 5.207
1.5054	1.4771	57.58	29	(103)ZnO	a = 3.49, c = 5.206	a = 3.25, c = 5.207
1.4125	1.4072	54.78	4	(200)ZnO	a = 3.262	a = 3.25, c = 5.207
1.2941	1.3017	50.70	2	(004)ZnO	c = 5.176	a = 3.25, c = 5.207

Fig. 5(a) and 5(b) represent the X-ray diffractograms of ZnO_{0.7}–GO_{0.3}–PVP_{0.1} and ZnO_{0.7}–GO_{0.3}–NMP_{0.1} and the corresponding data are presented in Tables 4 and 5 respectively. The diffraction lines observed with ZnO_{0.7}–GO_{0.3} are quite different from the lines observed in the case of ZnO_{0.5}–GO_{0.5}. The maximum intensity peak in both ZnO_{0.7}–GO_{0.3}–PVP_{0.1} and ZnO_{0.7}–GO_{0.3}–NMP_{0.1} composites is (002) plane of ZnO. The other planes observed are (100), (101), (102), (110) of ZnO and (002) plane of GO. The presence of diffraction lines of both GO and ZnO confirms the formation of the nanocomposites. The existence of only one GO peak may be due to the partial reduction of the GO to Graphene sheet [15].

The particle sizes were calculated using the Debye–Scherrer's formula ($D=0.94\lambda/\beta_{1/2} \cos \theta$) and the corresponding values are presented in Table 6. The average particle sizes were found to be in the nanometer range.

TABLE 3. XRD data of ZnO_{0.5}-GO_{0.5}-NMP_{0.1} Nanocomposites

d Value(A ⁰)		Relative Intensity		hkl	Lattice Constant (A ⁰)	
(Obs)	(Rep)	(Obs)	(Rep)		(Obs)	(Rep)
3.3846	3.3608	–	–	(002)GO	–	–
2.8462	2.8143	100	57	(100)ZnO	a = 3.286	a = 3.25, c = 5.207
2.5992	2.6033	70.93	44	(002)ZnO	c = 5.198	a = 3.25, c = 5.207
2.5104	2.4759	71.29	100	(101)ZnO	a = 3.31, c = 5.198	a = 3.25, c = 5.207
1.9347	1.9111	65.01	23	(102)ZnO	a = 3.345, c = 5.198	a = 3.25, c = 5.207
1.6168	1.6247	55.03	32	(110)ZnO	a = 3.233	a = 3.25, c = 5.207
1.5008	1.4771	56.17	29	(103)ZnO	a = 3.466, c = 5.198	a = 3.25, c = 5.207
1.417	1.4072	53.35	4	(200)ZnO	a = 3.272	a = 3.25, c = 5.207
1.2986	1.3017	49.47	2	(004)ZnO	c = 5.194	a = 3.25, c = 5.207

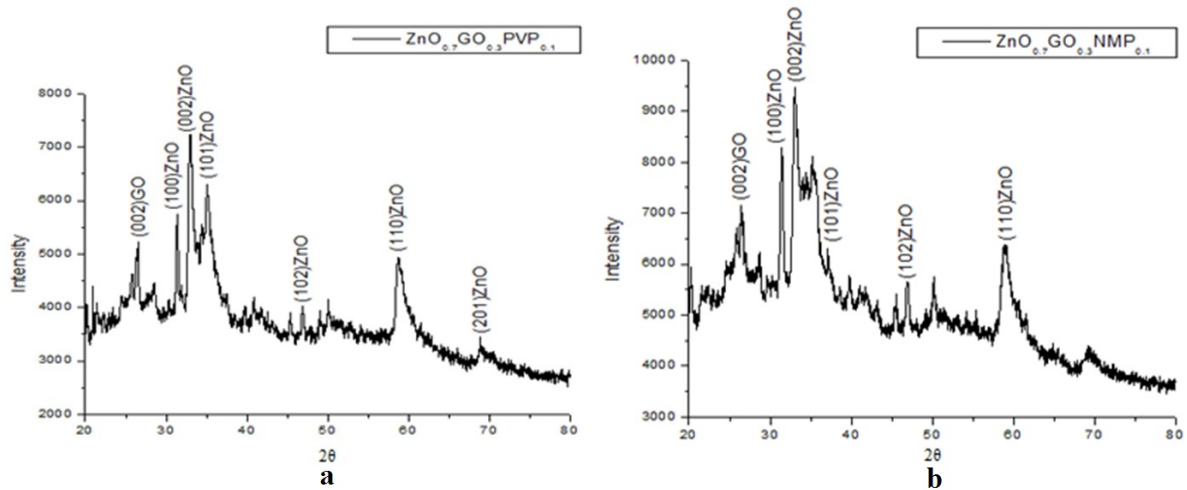


FIG. 5. (a)X-ray diffractogram of ZnO_{0.7}-GO_{0.3}-PVP_{0.1} Nanocomposites; (b)X-ray diffractogram of ZnO_{0.7}-GO_{0.3}-NMP_{0.1} Nanocomposites

TABLE 4. XRD data of ZnO_{0.7}-GO_{0.3}-PVP_{0.1} Nanocomposites

d Value(A ⁰)		Relative Intensity		hkl	Lattice Constant (A ⁰)	
(Obs)	(Rep)	(Obs)	(Rep)		(Obs)	(Rep)
3.3583	3.3608	–	–	(002)GO	–	–
2.8485	2.8143	79.52	57	(100)ZnO	a = 3.289	a = 3.25, c = 5.207
2.7116	2.6033	100	44	(002)ZnO	c = 5.423	a = 3.25, c = 5.207
2.554	2.4759	87.14	100	(101)ZnO	a = 3.289, c = 5.768	a = 3.25, c = 5.207
1.9347	1.9111	55.58	23	(102)ZnO	a = 3.289, c = 5.272	a = 3.25, c = 5.207
1.5724	1.6247	68.22	32	(110)ZnO	a = 3.144	a = 3.25, c = 5.207

TABLE 5. XRD data of ZnO_{0.7}-GO_{0.3}-NMP_{0.1} Nanocomposites

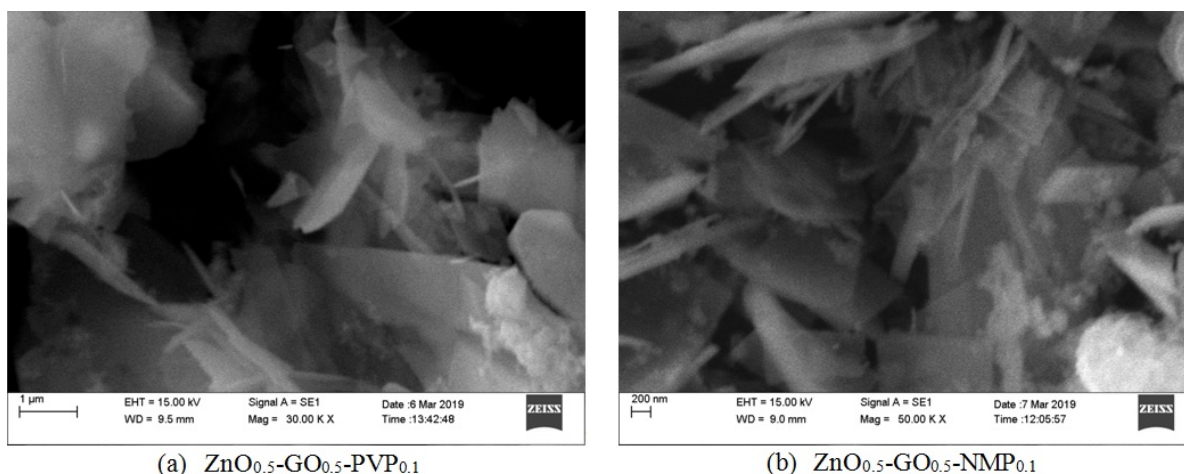
d Value(A ⁰)		Relative Intensity		hkl	Lattice Constant (A ⁰)	
(Obs)	(Rep)	(Obs)	(Rep)		(Obs)	(Rep)
3.3681	3.3608	–	–	(002)GO	–	–
2.8392	2.8143	87.63	57	(100)ZnO	$a = 3.278$	$a = 3.25, c = 5.207$
2.7095	2.6033	100	44	(002)ZnO	$c = 5.419$	$a = 3.25, c = 5.207$
2.4804	2.4759	67.96	100	(101)ZnO	$a = 3.278, c = 5.099$	$a = 3.25, c = 5.207$
1.9326	1.9111	59.97	23	(102)ZnO	$a = 3.278, c = 5.276$	$a = 3.25, c = 5.207$
1.6554	1.6247	53.88	32	(110)ZnO	$a = 3.31$	$a = 3.25, c = 5.207$

TABLE 6. Particle sizes of ZnO–GO Nanocomposites

S No.	Sample	Particle size (nm)
1	ZnO _{0.5} GO _{0.5} PVP _{0.1}	9.55
2	ZnO _{0.5} GO _{0.5} NMP _{0.1}	6.48
3	ZnO _{0.7} GO _{0.3} PVP _{0.1}	7.6
4	ZnO _{0.7} GO _{0.3} NMP _{0.1}	6.55

3.3. SEM Studies

The SEM micrographs of the ZnO_{0.5}-GO_{0.5}-PVP_{0.1}, ZnO_{0.5}-GO_{0.5}-NMP_{0.1}, ZnO_{0.7}-GO_{0.3}-PVP_{0.1} and ZnO_{0.7}-GO_{0.3}-NMP_{0.1} at a magnification of 50KX are shown in Fig. 6(a), 6(b), 7(a) and 7(b) respectively. A layered structure consisting of a mixture of sheets and rods along with small clusters of particles distributed on the surface of GO is seen in all the cases, confirming the interaction between ZnO and GO. The distribution of sheets and rods is on the surface of GO in the case of ZnO–GO–NMP composites in comparison to ZnO–GO–PVP composites. The images suggest that the formation of sheets and rods of ZnO prevent the stacking of graphene sheets and contribute to the electron transfer between ZnO rods and GO.

FIG. 6. SEM image of (a) ZnO_{0.5}-GO_{0.5}-PVP_{0.1} Nanocomposites and (b) ZnO_{0.5}-GO_{0.5}-NMP_{0.1} Nanocomposites

3.4. EDX Studies

Figures 8(a), 8(b), 9(a) and 9(b) represent the EDX spectra of ZnO_{0.5}-GO_{0.5}-PVP_{0.1}, ZnO_{0.5}-GO_{0.5}-NMP_{0.1}, ZnO_{0.7}-GO_{0.3}-PVP_{0.1} and ZnO_{0.7}-GO_{0.3}-NMP_{0.1} respectively. The presence of C, Zn and O is detected in all the cases confirming the formation of the nanocomposites. In the case of the ZnO–GO composites prepared in the presence of NMP, the C content is higher in comparison to the composite prepared in the presence of PVP. PVP is a

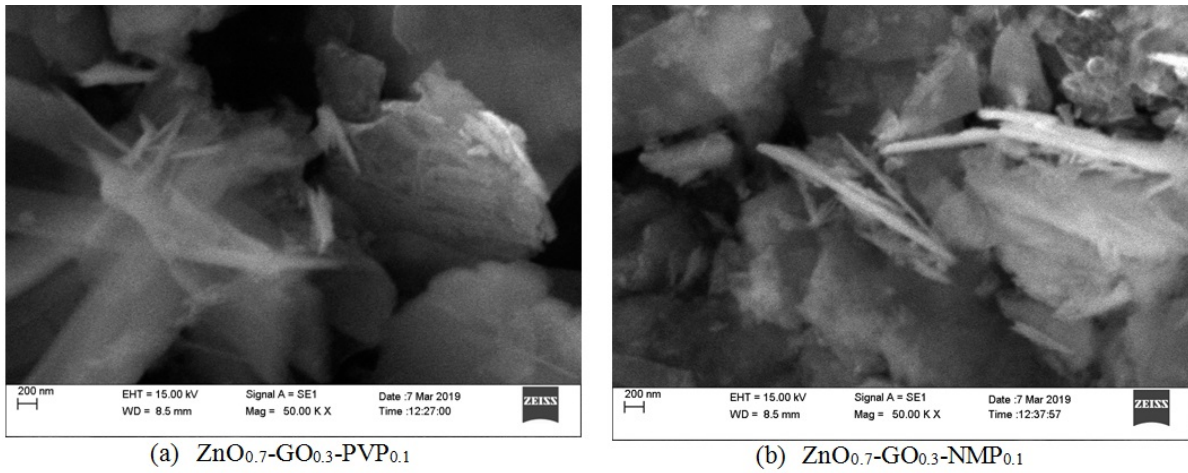


FIG. 7. SEM image of (a) ZnO_{0.7}-GO_{0.3}-PVP_{0.1} Nanocomposites and (b) ZnO_{0.7}-GO_{0.3}-NMP_{0.1} Nanocomposites

known surface stabilizing water soluble polymer effective in solubilization of Carbon materials [8], thereby resulting in the decreased percentage of Carbon atoms in these composites.

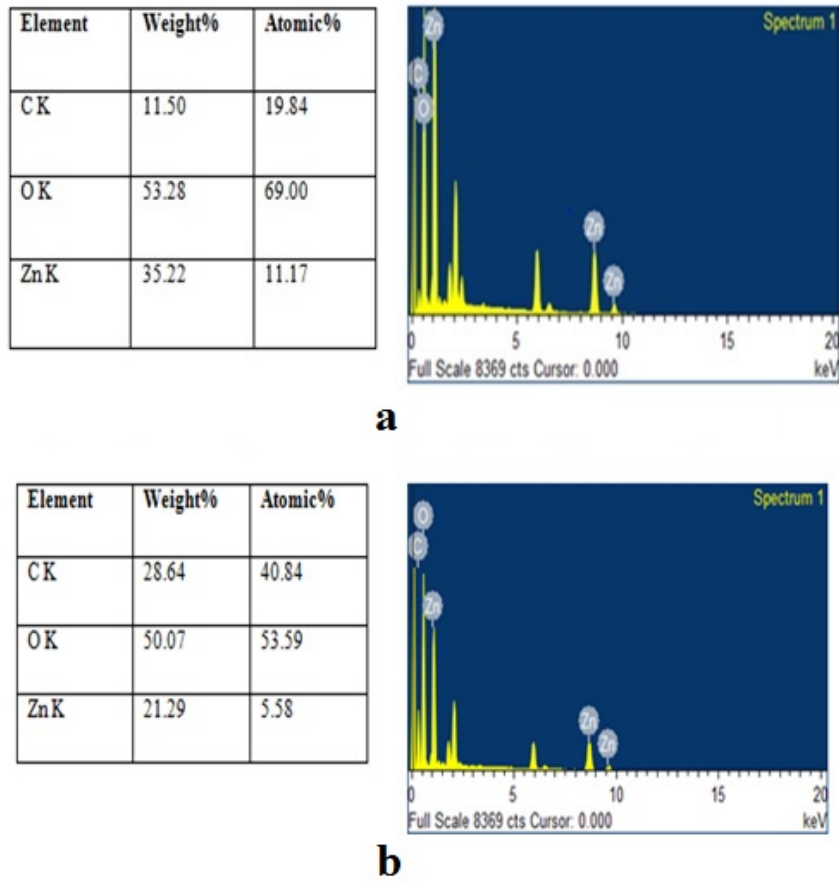


FIG. 8. EDX spectra of (a) ZnO_{0.5}-GO_{0.5}-PVP_{0.1} Nanocomposites and (b) ZnO_{0.5}-GO_{0.5}-NMP_{0.1} Nanocomposites

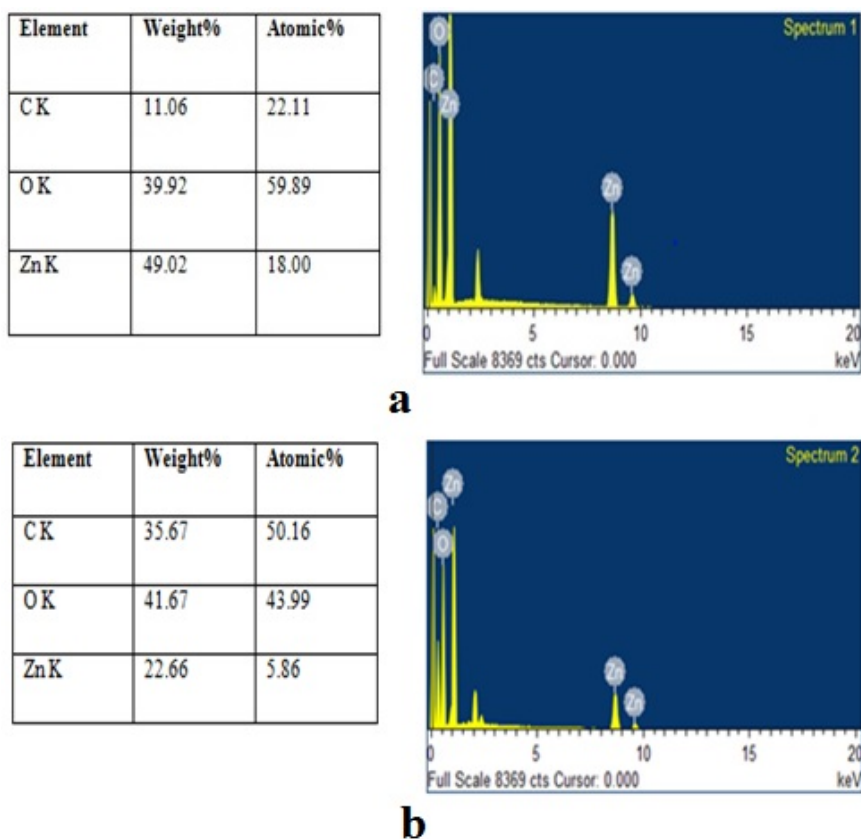


FIG. 9. EDX spectra of (a) $\text{ZnO}_{0.7}\text{-GO}_{0.3}\text{-PVP}_{0.1}$ Nanocomposites and (b) $\text{ZnO}_{0.7}\text{-GO}_{0.3}\text{-NMP}_{0.1}$ Nanocomposites

3.5. Conclusion

ZnO-GO nanocomposites were synthesized successfully using PVP and NMP as solvents by the sol-gel technique. The structural properties investigated using XRD studies showed the presence of planes of both GO and ZnO confirming the formation of composites. The particle sizes determined from XRD were found to be in nano range. A layered structure comprising of sheets and rods and a non-uniform distribution of agglomerating particles was observed from SEM micrographs. EDX spectra showed reflections of Carbon, Zinc and Oxygen in the composites. ZnO-GO synthesized using PVP showed higher absorbance in comparison to ZnO-GO with NMP. A shift in absorption edge to shorter wavelength in the presence of NMP was also observed. The band gap values were found to range from 2.7–3.0 eV with the band gap of ZnO-GO with NMP showing a higher value than ZnO-GO with PVP. It is quite clear from our observations that the optical band gaps of the nanocomposites are affected by varying the solvents. However, a marked difference in the structure of the composites is not observed by the presence of different solvents.

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