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Study of the effect of magnetic nanoparticles on the hologram recording capability of photopolymer nanocomposite for development of holographic sensor/actuator

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ABSTRACT

Photopolymer nanocomposite materials utilising nanoparticles of varied refractive index are one of the most attractive materials for holography due to their tuneable properties. The preparation of the photopolymer nanocomposites can involve mixing a stable colloidal suspension in the photopolymer solution or printing of the nanoparticle colloidal suspension on top of a dried photopolymer layer. By following the first approach in this study a novel photopolymer nanocomposite material is prepared for holographic recording. It consists of N-isopropylacrylamide (NIPA)-based photopolymer as a host and iron oxide magnetic nanoparticles as nanodopant. The methodology of the photopolymer nanocomposite material preparation is explained in detail. In order to investigate the interaction of nanodopants with the host photopolymer, the photo-bleaching of the layers when exposed to a single laser beam has been studied. Initial characterisation of the holographic recording capability of the nanocomposite has been carried out. The maximum diffraction efficiency (DE) achieved in 70 μm thick layer after recording with 3.4 mW/cm^2 intensity at 532 nm wavelength is 18 %.

Keywords: Holography, Photopolymer, Photopolymer nanocomposite, Nanoparticles doping, Magnetic nanoparticles, Volume holographic gratings, holographic sensor/actuator

1. INTRODUCTION

Since the invention of holography, materials research has delivered a variety of optical materials that are capable of recording and reconstructing holograms. Among these materials, photopolymers has attracted much of the researcher's attention due to their many advantages such as self-processing nature, high spectral sensitivity and relatively inexpensive mass production cost. Hence they are successfully studied for many applications such as holographic data storage¹, optical elements² and sensors^{3, 4}.

Unlike other holographic recording materials, photopolymers can be easily functionalised utilising different methods⁴; one approach is to embed nanosized guest materials (nanoparticles, nanocrystals) into the optically sensitive host photopolymer material. The aim of nanoparticles doping is either to improve the host material properties (e.g decreasing materials shrinkage) and/or to introduce the functionality of sensitivity of the material to certain targeted analytes. The main idea in developing photopolymer nanocomposite material is to dope the host photopolymer material with nanodopants of significantly different refractive indices and then achieve spatial redistribution of the nanodopants under spatially inhomogeneous illumination of light (holographic exposure), hence contributing to the refractive index modulation (RIM) values^{5, 6}. The idea was previously successfully implemented in case of doping titanium dioxide (TiO_2) nanoparticles into

acrylate photopolymer which resulted in reduced shrinkage and RIM value upto 5.1×10^{-3} was achieved⁷. Following this other nanoparticles such as SiO₂, ZrO₂ were effectively doped into photopolymers and in 2008, Tomita et al. observed that doping leads to the effective suppression in thermal effects and thus made them suitable for photonic applications with wider range of operating temperatures⁸. Research reported that purely metal nanoparticles such as gold (Au)^{9, 10} and silver (Ag)^{11, 12} have been doped into photopolymers which showed an increase in RIM due to their redistribution.

In addition to research on solid shaped NP's there are promising studies done on doping photopolymers with porous zeolite nanoparticles^{13, 14}. Apart from improving the dynamic range and shrinkage properties of photopolymers an additional objective achieved with zeolite NP's doping was developing holographic sensors, as due to their porous nature they are able to selectively adsorb/absorb certain molecules as analytes¹⁵. Therefore a hologram recorded in material doped with zeolites NP's, when exposed to the target analytes changes its properties. In this regard successful work was carried out by Leite et al. in developing holographic sensor for toluene by doping photopolymer with zeolite Beta NP's (BEA-type structure). Holograms recorded in both doped and undoped photopolymer layers were exposed to the analyte and it was observed that the diffraction efficiency change in doped layers was two times larger than the change of diffraction efficiency in undoped layers¹⁶. In another study irreversible holographic indicator was demonstrated for humidity by doping AIPO-18 zeolite NP's in photopolymer layer¹⁷.

Following the successful research carried out previously on photopolymer nanocomposites containing a variety of nanoparticles, the work presented here focuses on preparing a unique photopolymer nanocomposite material for holographic recording by using magnetic nanoparticles (MNP's). To the best of our knowledge, the doping of MNP's into the host NIPA-based photopolymer and the characterisation of its holographic recording properties is reported here for the first time.

2. MATERIALS AND METHODS

The aim is to prepare an innovative photopolymer nanocomposite material for holographic recording through a combination of host organic photopolymer and purely inorganic MNP's as nanodopants that is responsive to magnetic field. During the search for suitable MNP's, iron oxide MNP's were found to have numerous advantages over other magnetic materials e.g. Cobalt and Nickel. The non-toxic nature, biocompatibility, relatively easier synthesis of iron oxide MNP's made them suitable for our research work. Additionally the selection of iron oxide MNP's was guided by reports in the literature, describing the use of iron oxide MNP's in (hydrogel) for biomedical and actuator like applications¹⁸. Further to narrow-down among all iron oxide MNP's, Maghemite (γ -Fe₂O₃) and Magnetite (Fe₃O₄) were identified as the two most suitable types of iron oxides as they are most commonly used MNP's¹⁹. Compared to iron oxides MNP's other magnetic materials such as cobalt and nickel are reported as toxic and susceptible to oxidation; hence, they are of little interest¹⁹⁻²¹.

In this study NIPA-based photopolymer a recently optimised media for holographic recording²² and known for its temperature sensitivity²²⁻²⁴ is selected as the host photosensitive material for doping with iron oxide MNP's. The chemicals used in NIPA-based photopolymer were purchased from Fisher Scientific and Sigma Aldrich while iron oxide MNP's (Fe₂O₃-Gamma, 30 nm) were bought from GetNanoMaterials. Table 1, shows the chemical composition of photopolymer magnetic nanocomposite doped with 0.5 % wt/wt and 1 % wt/wt of MNP's.

Table 1: The chemical components used in the preparation of the photopolymer nanocomposite material

Function/role	Chemical component	PNIPA_Nanocomposite	
Binder	(Polyvinyl alcohol 10 % w/v) PVA, ml	16	
Monomer	(N-isopropyl acrylamide) NIPA, g	0.2	
Cross-linker	(N,N'Methylenebisacrylamide) BA, g	0.15	
Free-radical generator	(N-Phenylglycine) NPG, g	0.04	
Sensitising dye	(Erythrosine B dye 0.11 % w/v) Er B, ml	2	
Plasticizer/ free radical scavenger	Glycerol, ml	0.2	
Magnetic Nanodopant	MNP's (Fe ₂ O ₃ -Gamma), g Size is 30nm	For 0.5 % wt/wt	For 1 % wt/wt
		0.0112	0.0224

2.1 Preparation of photopolymer nanocomposite solution

The preparation of the host NIPA-based photopolymer was previously reported²². In the optimized composition of NIPA-based photopolymer, 16 ml of 10 % wt./wt. PVA is mixed with the remaining photopolymer components. Here the PVA solution was added in two parts. First 8 ml of 10 % wt./wt. PVA was mixed with the photopolymer components and the remaining 8 ml of 10 % wt./wt. PVA was first mixed with the iron oxide MNP's before adding them to the photopolymer solution. All the components used in preparation of the photopolymer host material were mixed thoroughly by magnetic stirring on a stirring plate in a beaker in laboratory dark room at controlled environmental conditions (temperature 21 ± 2 °C and relative humidity (RH) 35 ± 3 %) for 1-2 hours until a homogeneous solution is obtained.

For the dispersion of the magnetic nanoparticles the following procedure was implemented. The remaining half (8 ml) of 10 % wt./wt. PVA, was used for dispersion of MNP's (Fe₂O₃-Gamma, 30 nm) through Ultrasonic Probe Sonicator (Model: GEX 750), for 30 seconds. The iron oxide MNP's suspension was then finally added to the NIPA-based photopolymer prepared in first step, for the preparation of photopolymer nanocomposite material. The amount of iron oxide MNP's used was 0.5 % wt./wt. and 1 % wt./wt., with respect to the total amount of photopolymer in the dry layer.

2.2 Preparation of the layers of photopolymer nanocomposite

For studying the holographic recording and bleaching of the photopolymer nanocomposite, layers were made from photopolymer nanocomposite solution by evenly depositing three different amounts 0.5 ml, 0.750 ml and 1 ml of solution on a microscopic glass slide with dimensions 7.6 cm x 2.6 cm. The coated slides were allowed to dry in the dark room at a levelled surface for 8–10 h.

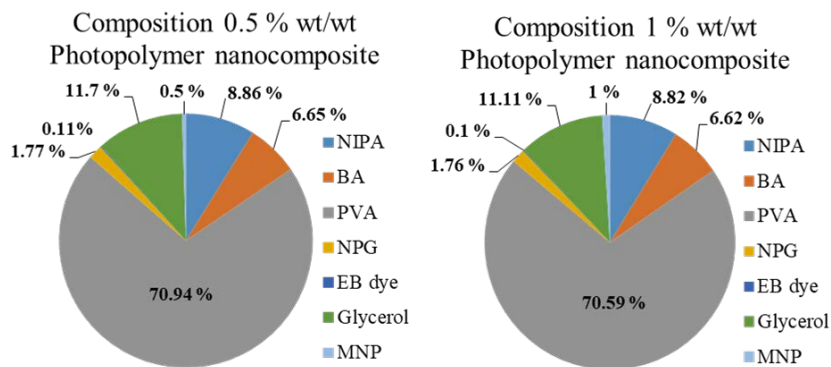


Figure 1. Pie charts representing the chemical composition in the dry layer of photopolymer nanocomposite.

2.3 Photo-bleaching of the nanocomposite material

In order to evaluate the interaction of the magnetic nanoparticles with the host photopolymer initial studies of the effect of nanoparticles on the layer's photobleaching dynamics were carried out. A single beam setup shown in figure 2 (a) was used for photo-bleaching of the photosensitive dye (Er.B dye) present in the material. The dye molecule by absorbing light photon of suitable energy initiates the photopolymerisation process in the photopolymer nanocomposite material. The Er.B dye is sensitive to wavelengths ranging 400 – 550 nm and the single beam used here for its photo-bleaching was obtained from Nd:YVO₄ laser of wavelength 532 nm. The samples of photopolymer nanocomposite layers were exposed to single laser beam for 300 s and an optical power meter (Newport) was used to monitor the transmitted beam intensity. The data acquisition was carried out with the help of a LabView computer programme allowing real –time monitoring of the bleaching dynamics.

2.4 Holographic Recording in nanocomposite material

A two-beam optical setup shown in figure 2 (b) was used for holographic recording of volume phase transmission gratings. The beams were obtained by splitting the Nd:YVO₄ laser light of wavelength 532 nm with polarising beam splitter and for achieving maximum visibility of the recorded interference fringes, half wave plate was used in the path of transmitted beam to rotate its polarisation state to get both beams vertically polarised. Unslanted holographic gratings were recorded in the photopolymer nanocomposite layers with spatial frequency of 830 lines/mm. As the host photopolymer used here has negligible absorption at 633 nm, a helium-neon (He-Ne) laser of 633 nm wavelength was used as probe beam to study real-time recording of holograms. After recording the gratings were rotated by a computer controlled rotation stage (Newport ESP300) and probed again with 633 nm for recording the Bragg angular selectivity curves. The first order diffracted beam intensity was monitored by an optical power meter (Newport) and the acquired data was sent to the computer for plotting with help of LabVIEW software. During all experiments mechanical stability was ensured for the optical setups shown in figure 2, by fixing them on a floating optical table (Newport RS 4000).

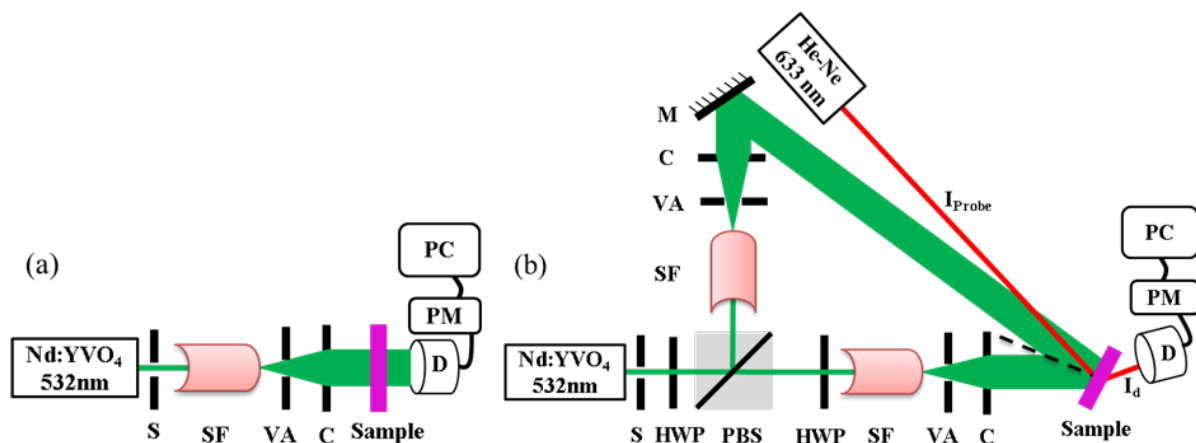


Figure 2. Experimental setup's used for (a) studying the bleaching of the photopolymer nanocomposite samples by exposure to a single beam and (b) recording of unslanted transmission holographic gratings by exposure to two beams. S-electronic shutter, HWP-half wave plate, PBS-polarising beam splitter, SF-spatial filter, VA-variable aperture, C-collimator, M-mirror, D-photodetector, PM-power meter, PC-computer.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Photo-bleaching study results

3.1.1 Effect of light intensity on photobleaching dynamics

The rate at which the photopolymer nanocomposite layers bleach will potentially have some dependency on the incident light intensity. In order to examine this dependency, the bleaching profiles were studied for three different values of the incident beam intensity 1.3 mW/cm^2 , 3 mW/cm^2 , 6 mW/cm^2 . The data was plotted as transmission (%) vs exposure time (figure 3(a)). For the three intensities the bleaching profiles are compared for the samples having same amount of photopolymer nanocomposite deposited on the substrate with same concentration of iron oxide MNP's. It worth noticing that at the beginning when the samples are exposed to laser light their bleaching profiles do not start from the same point. There is a difference in the initial transparency of the different samples. This could be due to variations in thickness or concentration or variations in the laser intensity. Both rate of bleaching and the absolute change in transmission increases with the increase of incident beam intensity.

In order to easily compare the data for the three mentioned intensities and better understand the relative change in transmission it was normalised. Here normalised transmission change was calculated by the relation $(I_n - I_i)/I_i$, where I_i is initial transmission intensity and I_n is transmission intensity at any time t_n after the start of the experiment. From the results shown in figure 3(b), it is observed that for lower intensity 1.3 mW/cm^2 the normalised transmission change is gradually increasing and does not achieve saturation in the allowed time duration of 300 s. For a slightly higher intensity 3 mW/cm^2 the normalised transmission change increases faster and still shows a linear trend without achieving a saturation point. By increasing intensity even further upto 6 mW/cm^2 the normalised transmission change is increasing sharply with faster rate compared to others and nearly achieves saturation just below 300 s. In case of 6 mW/cm^2 the increase in transmission ($I_n - I_i$) for the 300 s time duration is greater than the initial starting transmission value I_i , hence it's normalised transmission change is greater than one.

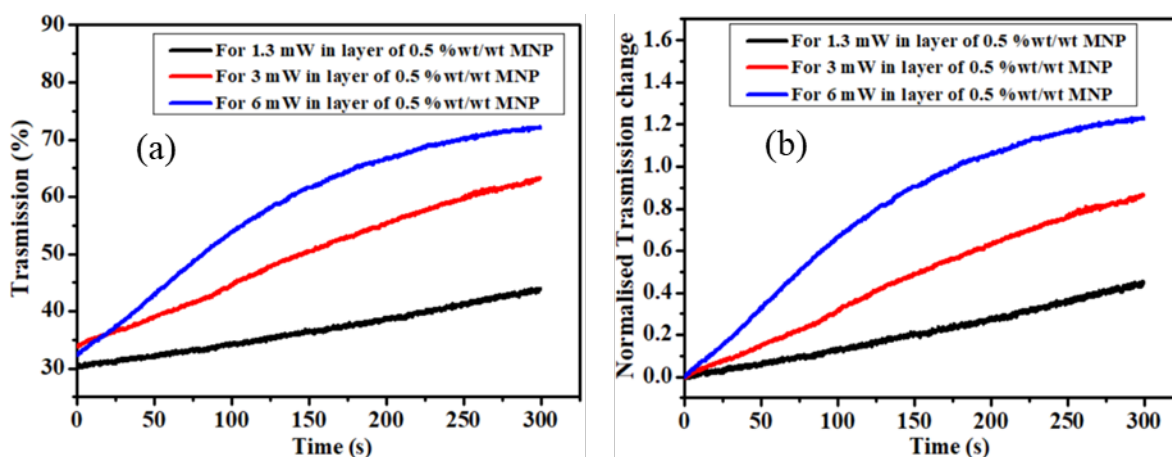


Figure 3. Bleaching profiles of photopolymer nanocomposite. (a) transmission vs time and (b) normalised transmission change vs time

3.1.2 Effect of iron oxide MNP's concentration on layer's transmission

The effect of iron oxide MNP's concentration on the bleaching of photopolymer nanocomposite was studied by comparing the results for two concentrations (0.5 % wt/wt and 1 % wt/wt) of iron oxide MNP's. Figure 4, below shows the

transmission change over 300 s duration vs total intensity for photopolymer nanocomposite layer having concentration of (a) 0.5 % wt/wt (b) 1 % wt/wt in the dry layer. The transmission change was calculated as $(I_f - I_i)$, where I_i is initial transmission intensity and I_f is the final maximum transmission intensity at end point of the bleaching experiment.

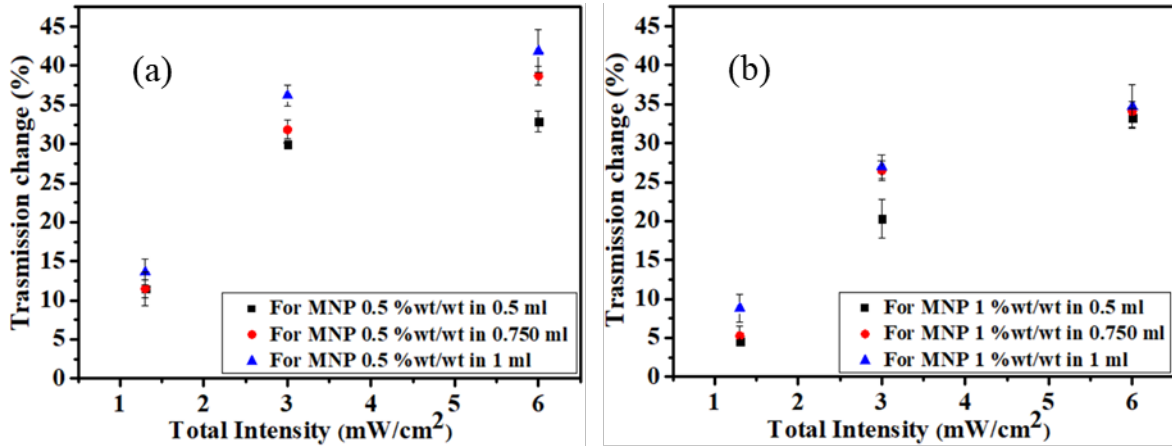


Figure 4. Transmission change vs incident intensity of laser light on layers of photopolymer nanocomposite having (a) 0.5 % wt/wt and (b) 1 % wt/wt of iron oxide MNP's in dry layers

The results presented in Fig 4 reveal that the absolute change in transmission is generally higher in the samples with lower load of magnetic nanoparticles. They also reveal that while at the lower concentration of nanoparticles there is some dependence of the absolute change in transmission (related to dye bleaching) on sample thickness in the samples with higher concentration of nanoparticles such dependence is not so clearly observed.

3.1.3 Effect of the layer thickness on transmission change

In order to study the dependence of the bleaching on the thickness of photopolymer nanocomposite layers the absolute transmission change over 300 s duration was plotted versus amount of photopolymer deposited on the glass substrate for both concentrations of MNP's separately. The results are shown in figure 5, for layers having concentration (a) 0.5 % wt/wt and (b) 1 % wt/wt of MNP's in dry condition.

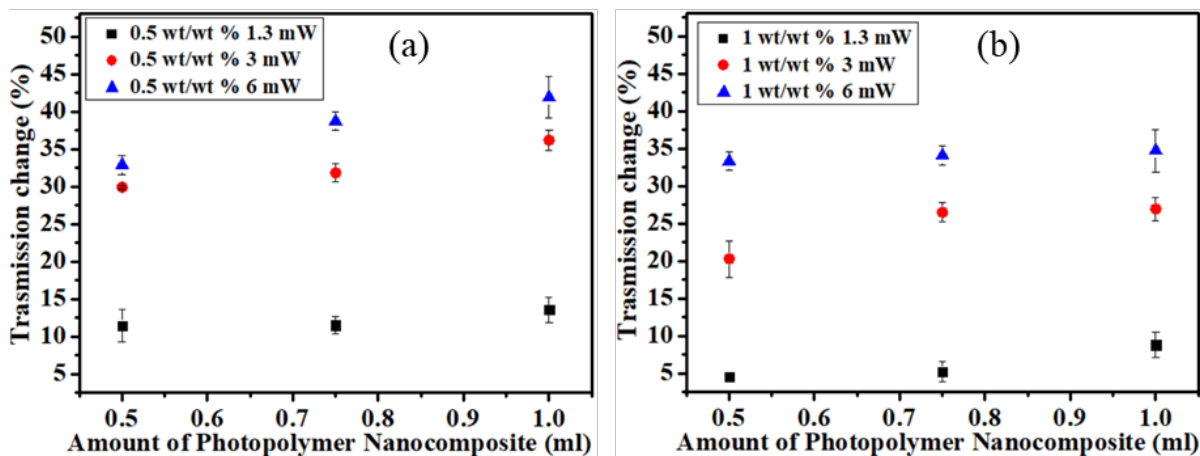


Figure 5. Transmission change vs amount of photopolymer nanocomposite solution on microscopic glass slide containing (a) 0.5 % wt/wt and (b) 1 % wt/wt of iron oxide MNP's in dry layers.

Overall at the starting point and other values of transmission are high for lower concentration of MNP's compared with their respective values for higher concentration of MNP's, except for the data in case of high intensity 6 mW/cm² at 0.5 ml of photopolymer nanocomposite. This implies that at lower concentration of MNP's there was less scattering and absorption of light and hence high transmission intensity. The results in case of lower concentration of MNP's show some trend of increase in the absolute transmission change at high intensities (3 mW/cm² and 6 mW/cm²) with the amount of photopolymer nanocomposite deposited on the glass substrate while no clear increasing trend is observed for higher concentration of MNP's.

3.2 Holographic Recording study results

3.2.1 Effect of nanoparticles concentration on holographic recording

The hologram recording capability of the photopolymer nanocomposite material (Poly-NIPA photopolymer doped with iron oxide MNP's Fe₂O₃-Gamma, 30 nm) is studied by real-time monitoring of the hologram recording process. In this experiment, unslanted volume transmission gratings are recorded and the diffraction efficiency (DE) of the gratings is calculated as the ratio of the first order diffracted beam intensity (I_d) divided by the incident beam intensity (I_i). The real-time DE growth curves obtained under same recording conditions for two different concentrations (0.5 g % wt/wt and 1 g % wt/wt) of iron oxide MNP's present in the layer are shown in figure 6 (a). For the same value of total recording intensity 3.4 mW/cm², the photopolymer nanocomposite layer having less concentration (0.5 g % wt/wt) of iron oxide MNP's are showing much smoother recording curve compared to high concentration (1 g % wt/wt). Even though layers in case of both concentrations achieve saturation after 40 s of recording time but the layer having less concentration (0.5 g % wt/wt) shows a sharp increase in DE and reaches to almost double time value of DE compared to high concentration (1 g % wt/wt) layer. Achieving less value of DE in case of higher concentration (1 g % wt/wt) may possibly be due to greater amount of light absorbance and scattering due to presence of iron oxide MNP's Fe₂O₃-Gamma in the layer.

After recording the gratings were rotated with a computer controlled rotational stage in the range -3 to +3 degree for plotting the Bragg selectivity curves. The obtained result of Bragg selectivity curves of gratings for both concentrations (0.5 g % wt/wt and 1 g % wt/wt) of iron oxide MNP's present in the layer are shown in figure 6 (b). Bragg selectivity curve of grating recorded in layer having less concentration (0.5 g % wt/wt) of iron oxide MNP's shows a higher value of DE (upto 18 %) which is two times higher than the value achieved in layers with higher concentration (1 g % wt/wt) of iron oxide MNP's. For achieving higher values of DE during holographic recording, the photopolymer nanocomposite layers still needs to be optimised for an appropriate value of iron oxide MNP's doping concentration.

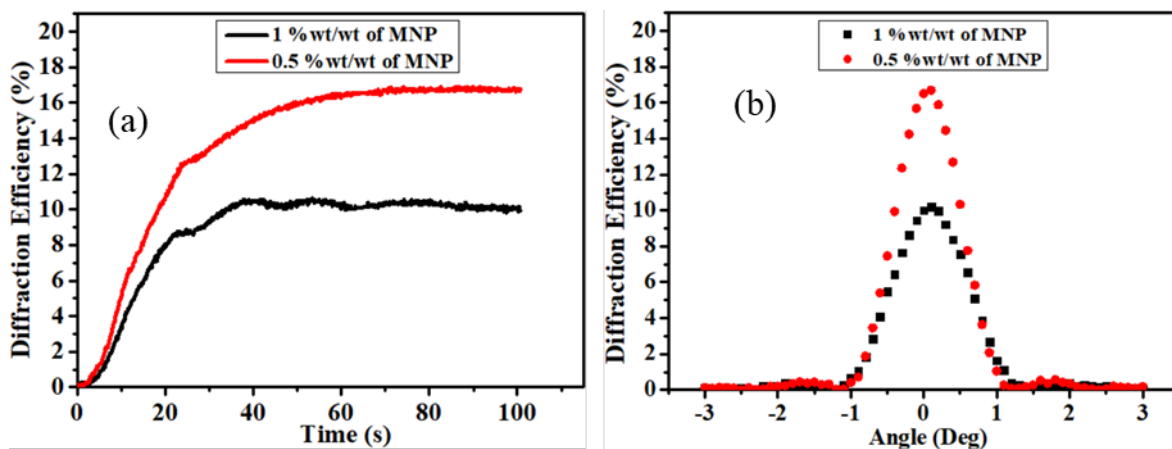


Figure 6. Characterisation of the diffraction grating recorded in photopolymer nanocomposite (a) real-time growth curve of DE and (b) Bragg selectivity curve of DE

3.2.2 Effect of recording intensity on holographic recording

As for the pure photopolymer, the two main processes that govern the holographic gratings formation are polymerisation accompanied by the monomer diffusion and in case of photopolymer nanocomposite an additional process is the spatial redistribution of the nanodopants under spatially inhomogeneous illumination of light (holographic exposure). In holographic recording the incident light beams intensity plays an important role in the formation of holographic gratings due to the fact that the rate of polymerisation is dependent on the recording intensity. The recording intensity controls the availability of light photons that are essential for producing free radicals which then initiates the photopolymerisation reaction in photopolymers.

The effect of recording intensity on the holographic recording capability of the prepared photopolymer nanocomposite material was investigated in transmission mode by recording volume holographic gratings at different total recording intensities having values 1.5 mW/cm², 3.1 mW/cm², 6.3 mW/cm² and 9.5 mW/cm². During this study holographic gratings were recorded in photopolymer nanocomposite layers having concentration (0.5 g % wt/wt) of iron oxide MNP's and the obtained results are plotted as shown in figure 7. From the comparison of DE growth curves figure 7 (a) and their respective Bragg selectivity curves given in figure 7 (b), it is clear that at lower intensity 1.5 mW/cm², the rate of grating development is slower and only achieves DE upto 6 %. By increasing the recording intensity to 3.1 mW/cm², it was found to be the best during recording in the present study conditions as in this case maximum value of DE (13%) was achieved compared to other studied intensities. Using further higher recording intensities 6.3 mW and 9.5 mW, both the DE growth curves attain even more similar steeper slope quickly after the recording started but finally they end up with about 10 % and 8 % of DE respectively. A thorough research study will still be of immense importance to determine the optimum recording intensity at different spatial frequencies, thickness of the layer.

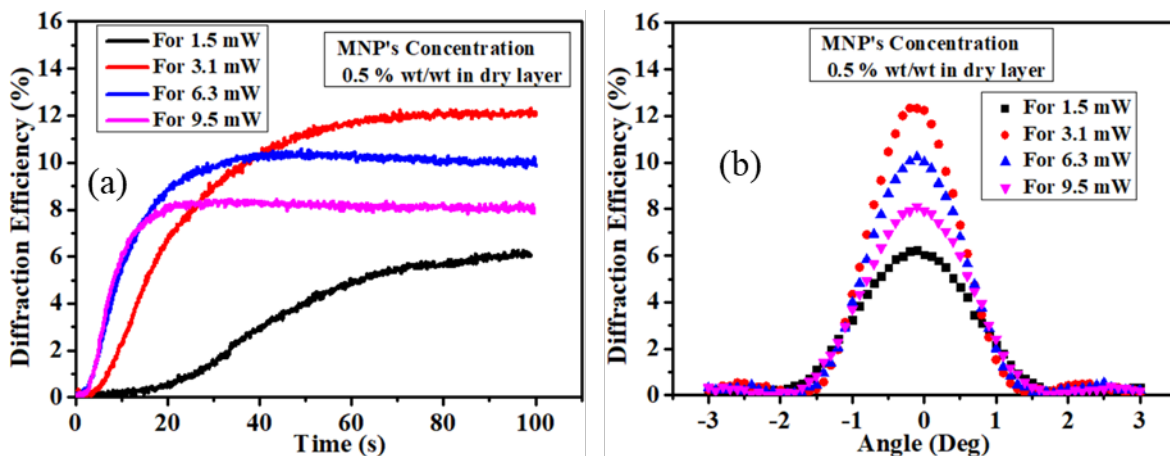


Figure 7. Effect of recording intensity on holographic recording in photopolymer nanocomposite layers having 0.5 % wt/wt of MNP (a) the real-time growth curves of DE and their (b) Bragg selectivity curves

4. CONCLUSION

In this study MNP's were discussed and iron oxide MNP's were particularly selected for our photopolymer nanocomposite due to their non-toxic nature and biocompatibility. The doping of MNP's into the host NIPA-based photopolymer is investigated here for the first time in developing a photopolymer nanocomposite material for holographic recording. Good optical quality layers are prepared successfully from the photopolymer nanocomposite material for which the photo-bleaching and its hologram recording capability are then studied. Preliminary results are presented on the holographic recording capability of this photopolymer nanocomposite (Poly-NIPA photopolymer doped with iron oxide MNP's) confirming the possibility of holographic recording and thus opening a window in this field of photopolymer nanocomposite used for holographic recording. Still significant research effort is required for optimizing the material (e.g.

size of MNP's, concentration of MNP's) and recording conditions (e.g. optimum recording intensity, exposure time, spatial frequency, etc) in order to achieve higher diffraction efficiency.

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