

# Structural and Morphological Analysis of ZnO Nanoparticles filled Low Density Polyethylene Thin Films

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## Abstract

This paper describes the studies related to ZnO nanoparticles (0, 0.5, 1, 3 and 5 wt. %) filled Low Density Polyethylene (LDPE) thin films prepared by solution casting technique. The synthesized ZnO/LDPE nanocomposite thin films were analyzed by XRD, SEM and FTIR. Peaks in X-Ray Diffraction (XRD) pattern indicate the presence of ZnO nanoparticles in LDPE matrix and absence of any extra peak in the pattern shows there is no new phase formation. The Scanning Electron Microscope (SEM) images showed that the nano inclusions are uniformly distributed in the LDPE matrix. Fourier Transform Infrared (FTIR) result indicates the presence ZnO nanoparticles in LDPE thin films.

**Keywords:** Nanocomposite, Thin Film, XRD, SEM, FTIR

## I. INTRODUCTION

The integration of inorganic nanoparticles into a polymer matrix allows both properties from inorganic nanoparticles and polymer to be combined/ enhanced. Polymers- inorganic nanocomposites attract considerable interest due to the outstanding mechanical, optical, electrical, and thermal properties encountered with only a small quantity of nanofiller incorporated to the polymer matrix. This is caused by the large surface area to volume ratio of fillers when compared to micro- and macro-scale additives [1].

Low density polyethylene is among the most versatile polymers, but its uses are limited due to several drawbacks, namely low strength, stiffness and poor heat resistance. To overcome these drawbacks and to prepare material with improved properties, fillers are incorporated into the matrix [2]. Fillers can affect the dimensional stability, crystallinity, mechanical and other properties of polymers [3, 4]. Numerous studies have also been performed to investigate the space charge distribution and electrical conduction in polyethylene systems.

Zinc Oxide is an important electronic and photonic material which possesses semiconducting and piezoelectric properties. It is a semiconductor, with a direct band gap of 3.37 eV and a large excitation binding energy (60 meV) [5]. Nanosized inorganic semiconductor materials have attracted considerable efforts in recent years. Zinc oxide is an interesting material has been widely used in application such as UV protection, photo catalysis, filed emission displays, veristors, gas sensors, function devices, thermoelectric materials, etc due to its exceptional physical & chemical properties [6].

Many research groups have focused on dispersing metal oxide nanoparticles into polymer matrix, as the hybrid nanocomposites not only inherit the functionalities of semiconductor nanoparticles but also possess advantages of polymers such as flexibility, film integrity, and conformity [7].

Polymer Inorganic Nanocomposite of LDPE and Zinc Oxide nanoparticles can exhibits some novel properties. Thus in the present work, Zinc oxide/ LDPE nanocomposites were prepared by adding the ZnO nanoparticles in different weight % (0, 0.5, 1, 3 and 5) in LDPE. The structural and morphological studies of ZnO/polymer nanocomposite films were carried out using XRD, SEM and FTIR analysis.

## II. EXPERIMENTAL

Thin films of ZnO/LDPE nanocomposites were prepared by using solution - cast technique [8]. Commercial grade granular LDPE was dissolved in Xylene using hot plate magnetic stirrer at 100°C for 2 hrs so as to obtain clear viscous solution. The hexagonal wurtzite structured Zinc oxide nanoparticles (ZnO NPs) with average crystalline size about 80 nm, synthesized by a simple

chemical solution method followed by combustion were used as the filler in different weight % (0, 0.5, 1, 3 and 5), reported elsewhere [9]. For maximum dispersion the solution was further stirred for 2 hrs by keeping the temperature constant.

The melt was then poured on clean optically plane glass plate kept in pre-heated oven at 80°C for 1 hr. After complete evaporation the film was detached from the glass surface. The thickness of samples was measured by compound microscope in conjunction with an occulometer having least count 15.38  $\mu\text{m}$  similar to the method reported by [10]. The thickness of all samples was kept constant and it is of the order of  $\approx 61.52 \mu\text{m}$ .

### III. RESULT AND DISCUSSION

#### A. X-Ray Diffraction (XRD) Analysis

To determine the nature of the samples and to confirm the presence of crystalline metal oxide within the polymer matrix, the structure of the polymer inorganic nanocomposites thin films were characterized by X-ray diffraction.

The structure of the Polymer Inorganic Nanocomposites (PINCs) thin films has been characterized by XRD in order to confirm the structure and the presence of Zinc oxide nanoparticles (ZnO NPs) within the LDPE matrix. The X-ray diffraction patterns using  $\text{CuK}\alpha$  radiation for pure LDPE and ZnO NPs (0.5 and 5 wt. %) filled LDPE nanocomposite thin films are represented in Figure 1.1, 1.2 and 1.3 respectively. Results of only two concentrations (0.5% and 5%) are discussed in detail because they show a remarkable effect.

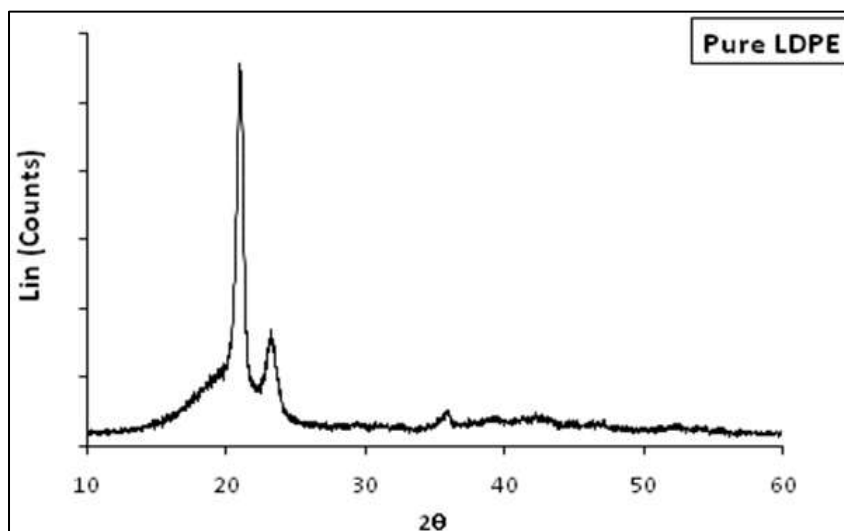


Fig. 1.1: X-ray Diffractogram of Pure LDPE Thin Film [LDPEZnO(0)]

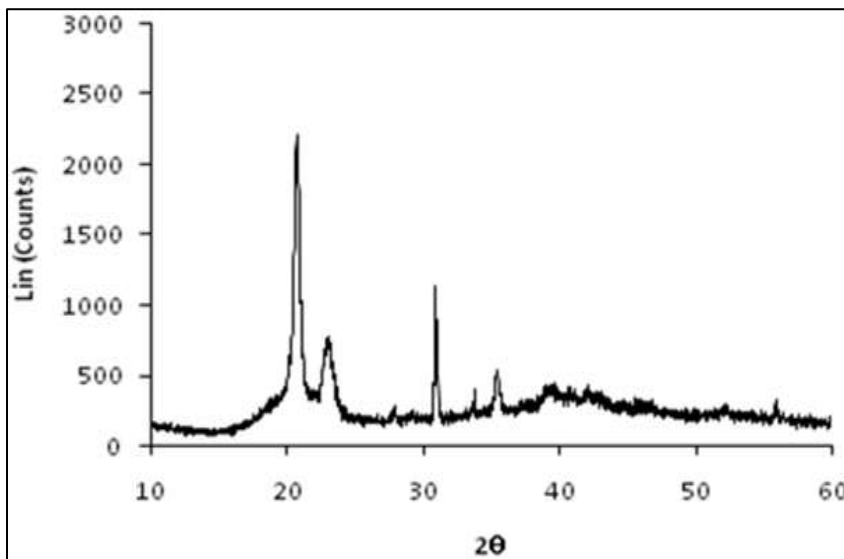


Fig. 1.2: X-ray Diffractogram of 0.5 wt. % ZnO NPs Filled LDPE Thin Film [LDPEZnO(0.5)]

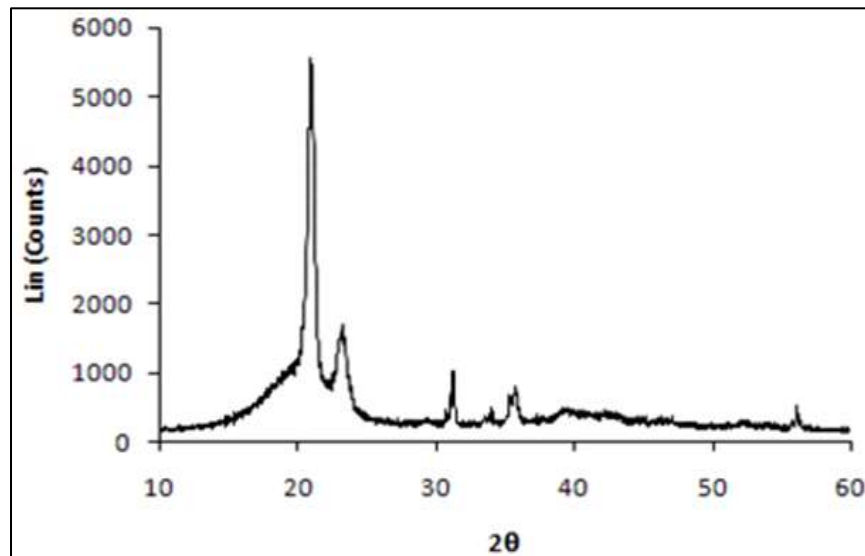


Fig. 1.3: X-ray Diffractogram of 5 wt. % ZnO NPs Filled LDPE Thin Film [LDPEZnO(5)]

The XRD pattern in figure 1.1, 1.2 and 1.3, dominant fairly sharp peak at  $21.4^\circ$  and a weak broad peak at  $23.7^\circ$  corresponds to LDPE. The peaks for LDPE sample appearing at 21.4 and 23.7, corresponds to (110) and (200) reflections from the orthorhombic polyethylene crystals [11]. This dominant peak and the small weak broad peak arose from crystalline and amorphous regions in LDPE [12; 13].

In figure 1.2 and 1.3 the typical diffraction peaks of ZnO NPs are observed at 31.3, 34.2, 35.9 2-theta and all diffraction peaks are in agreement with the hexagonal wurtzite structure [14]. Other signals are of very low intensities, probably because of the low concentration of the zinc oxide phase in the polymer matrix and the very small crystallite sizes. The composites possesses typical crystal peaks of ZnO (wurtzite) and also increasing ZnO NPs content increases the intensity of the peaks as observed in figure 1.3. Thus Crystallinity of the LDPE matrix is essentially affected by ZnO NPs addition. The X-ray diffraction analysis proved that introducing increasing ZnO NPs content resulted in an increase of crystallization degree of LDPE.

There was a noticeable change in the intensity of XRD peaks of the ZnO NPs filled samples. ZnO NPs were added into LDPE, making ZnO/LDPE nanocomposites, the intensity of LDPE peaks increased and became sharper. An increase in the intensity and decrease in the width of ZnO/LDPE nanocomposites are observed indicating an increase in semi-crystalline structure, which leads to their good compatibility [15].

### **B. Scanning Electron Microscopy (SEM) Analysis**

The morphology of ZnO/LDPE nanocomposite film surfaces was examined by SEM. Figures 2.1 and 2.2 shows the micrographs of surfaces of the nanocomposites films filled with 0.5 and 5 wt. % of ZnO NPs in LDPE respectively.

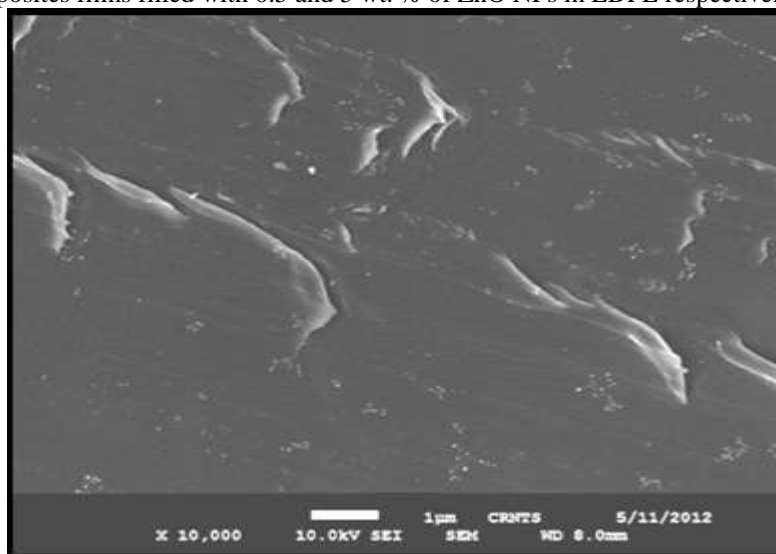


Fig. 2.1: SEM Micrographs of 0.5 wt. % ZnO NPs Filled LDPE Thin Film [LDPEZnO(0.5)]

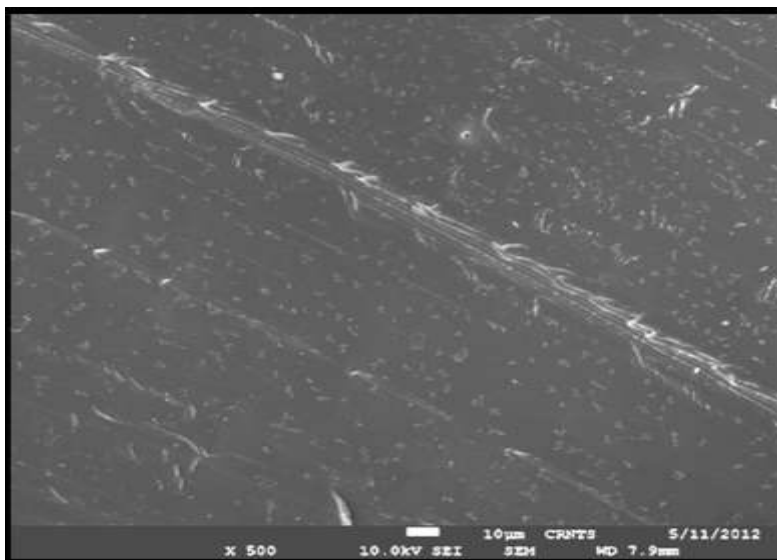


Fig. 2.2: SEM Micrographs of 5 wt. % ZnO NPs Filled LDPE Thin Film [LDPEZnO(5)]

The micrographs exhibit presence of clusters of ZnO NPs embedded into LDPE matrix. From figure 2.1 and 2.2, it is observed that the aggregate was formed as cluster. The small clusters grew to larger with increase in ZnO NPs concentration and are uniformly aggregate in the LDPE film surface.

It was observed that the dispersion of ZnO NPs particles was relatively good and uniformly dispersed throughout the entire polymer matrix. However the ZnO particles aggregate to each other to become a cluster. The dispersion of ZnO NPs could have an influence on the mechanical properties of ZnO/LDPE nanocomposites.

The filler added to the polymer matrix forms the clusters that can form the conductive networks and decrease the resistivity of the composite as explained by the percolation theory [16]. Since the convergence of particles may lead to the probability of transfer of current carriers between them greater than zero. Thus ZnO NPs can improve the electric properties also.

### C. Fourier Transform Infrared (FTIR) Analysis

FT-IR spectroscopy was employed in order to reveal the incorporation of ZnO NPs in LDPE matrix. Fourier transform infrared (FTIR) spectra of the samples were obtained in the wave number range  $400\text{--}4000\text{ cm}^{-1}$ . Figure 3 shows the FT-IR spectra of LDPE, and ZnO/LDPE nanocomposite. The spectrum of the nanocomposite exhibits the characteristic absorption bands corresponding to polymeric groups and ZnO.

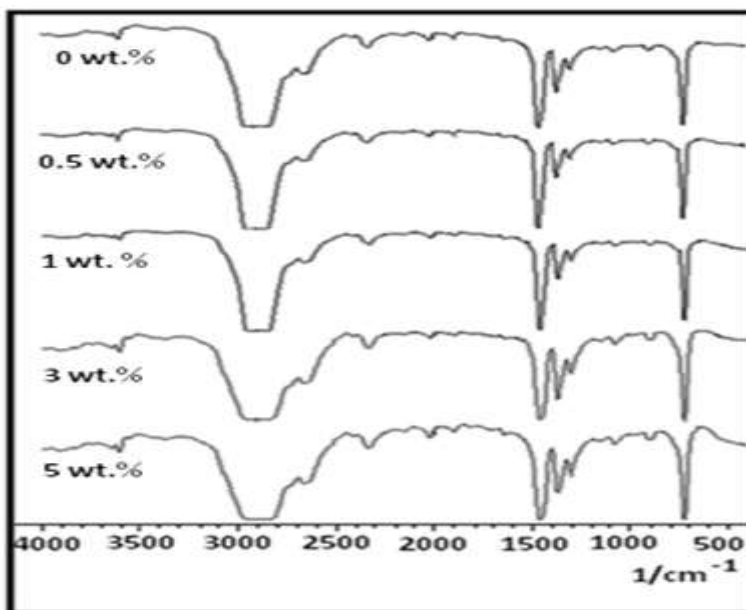


Fig. 3: Infrared Spectra of Unfilled and ZnO NPs (0.5, 1, 3, 5 wt. %) Filled LDPE Thin Film

The peaks in the region  $3370\text{--}3750\text{ cm}^{-1}$  are due to O-H stretching of hydroxyl group. A strong peak observed at  $3000\text{--}2800\text{ cm}^{-1}$  corresponds to  $\text{sp}^3$  C-H stretching. The bands at  $2915$  and  $2332\text{ cm}^{-1}$  are due to the aliphatic C-H and C-C respectively. The

prominent bands of LDPE at 1465 and 1375  $\text{cm}^{-1}$  are arising from the  $-\text{CH}_2-$  group in the PE chain. Other characteristic bands of LDPE are 1304  $\text{cm}^{-1}$  ( $\text{CH}_2$  wagging), 1080.14 (C-H bond) and 890 ( $\text{C}=\text{CH}$ ). The peak at 723.3  $\text{cm}^{-1}$  is probably due to C-H rocking.

On comparing the FTIR spectra of unfilled and ZnO filled LDPE thin films, it is observed that ZnO filled LDPE thin film spectra contain one additional peak at  $\sim 422 \text{ cm}^{-1}$  corresponding to ZnO bond along with all the characteristic peaks of unfilled LDPE spectra. This indicates that there is no chemical bonding between the ZnO and LDPE. There is even no significant shift in the characteristic transmittance peaks indicating that LDPE did not have strong interaction with ZnO. It is clear from the spectra that ZnO/LDPE nanocomposite was successfully synthesized in this work.

Since no noticeable change in peak positions observed in all the FTIR spectra, the peak frequencies and their co-relation has been tabulated in single table (table 3.1). Our results are in good agreement with [17, 18, 19].

Table - 3.1  
Frequency Correlation in Unfilled and ZnO NPs (0.5, 1, 3, 5 wt. %) Filled LDPE Thin Film

Sr. No.	Frequency ( $\text{cm}^{-1}$ )	Correlation
1	3604	O-H stretching
2	3000-2800	Strong C-H stretching
3	2915	Aliphatic C-H
4	2332	Aliphatic C-C
5	1465.9	C-H bending $\text{CH}_2$
6	1375	$-\text{CH}_2-$ group
7	1304	$\text{CH}_2$ wagging
8	1080.14	CH
9	890	$\text{C}=\text{CH}$
10	723.3	C-H rocking
11	422	Zn-O bond

#### IV. CONCLUSION

The ZnO/LDPE nanocomposites films were prepared by solution - cast technique. The XRD pattern confirms the presence of crystalline metal oxide within the polymer matrix. SEM images for ZnO/LDPE show heterogeneity of phases and immiscibility of ZnO NPs with the polymers. The functional groups present in the samples are identified using FTIR spectroscopy for ZnO/LDPE. Thus the ZnO nanoparticles doped in LDPE shows good compatibility which may influence the optical, electrical and mechanical properties of LDPE.

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