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DLS and Zeta Potential Studies of PVB, ZnO and their Nanocomposites

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Abstract
Here we are reporting preparation and characterization of ZnO incorporated PVB matrix based Nanocomposites in Methyl trichloride ($CHCl_3$) organic solvent. Solution casting technique is used to prepare the Nanocomposite films in Teflon Petri dishes at ambient temperature. In the characterization part we have studied XRD, FESEM, DLS, Zeta and Dielectric Studies of PVB, ZnO and their nanocomposites. The XRD studies of ZnO are showing crystalline structure but when nanocomposite is formed with amorphous PVB the ZnO is losing its crystallinity. FESEM images also indicating that strong interaction between ZnO and PVB polymer fibers. In DLS studies the hydrodynamic diameters of ZnO, PVB and their Nanocomposites are measured in $CHCl_3$ as 357.8 nm, 41.7 nm and 370.6 nm these results are indicating interaction of ZnO with PVB and forming a Core-Shell structured Nanocomposite. The zeta potentials of individuals and their Nanocomposite are measured and these values are observed as 36.1mV for ZnO, 41.7mV PVB and 17.6mV for Composite. These results are indicating that the PVB is forming a shell on ZnO core due to strong interaction between hydrophobic butyl functional group of PVB with ZnO. Similarly it is observed that Nanocomposite is more unstable than the individuals in chloroform.
Key Words:- ZnO/PVB, XRD, FESEM, DLS, zeta potential

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I. Introduction:-

A lot of amount of research has been carried out in the field of Polymer matrix based nanocomposites since last two decades due to their importance in Science and technology. These Nanocomposites contains remarkable structural, mechanical, thermal and electrical properties which can be used in Electronics devices. Polymer Nano-composites generally consists a polymer matrix reinforced with nano-scale fillers. This new class of composite materials has shown improved physical properties and is of major scientific and technological concern. The physical properties of such nano-composites in lower percentage of loadings may change remarkably compared to micro composites due to the larger Aspect ratio. The polymer nano-composites have a diversity of applications in various fields such as optical integrated circuits, automobile, energy storage and saving, defense and medical devices [1-5].

Poly Vinyl Butyral (PVB) is an environment friendly (Non-toxic and odorless) thermoplastic polymer which is widely used as a functional material in various fields for the fabrication of organic/inorganic hybrid composites. The IUPAC name of the polymer is Poly[(2-propyl-1,3- dioxane-4,6-diyl) methylene] with chemical structure as mentioned below It is in white powder form with specific gravity 1.083gcm^{-3}. PVB has hydrophilic vinyl alcohol groups and hydrophobic vinyl butyral groups. These groups behave as promoters of polymer adhesive and binders for organic moieties. It is one of the high priority materials in the industry and fulfils many essential aspects, owing to the good elasticity, water resistance, adhesion to various surfaces, sharp optical clarity, high compatibility with other polymers and very good processing options. It is also used as the interlayer of laminated glass in automotive wind-shields, optical windows for equipment, and emergency glass for buildings due to its excellent properties such as high radiation resistance.

The main goal is to produce high-performance polymer nano-composites, which mainly depends on inter particle forces, polymer–nano-particle interactions and nano-particle shape, as well as on the preparation procedure. Polymer nano-composite films can be fabricated by various methods. Solution casting is one of the best methods when the solvent used is less toxic. This is the easiest method to obtain fine nano-composite films hence we adopted this method to prepare ZnO reinforced PVB matrix Nanocomposite films [3-7].

II. Materials and Methods:-

2.1 Materials:-

PVB (Poly Vinyl Butyral, molar mass 300.395g/mole, melting point around 109^0C) powder purchased from Rolex chemical industries, Bengaluru.

ZnO (Average diameter 10 – 40nm, purity > 99.9%) purchased from Ad-Nano Technologies Pvt. Ltd, Shimoga, Karnataka, India. High Quality PTFE-60mm Petri dishes purchased from Canfort Laboratory Co.Ltd. USA. Chloroform was purchased from Molychem laboratory, Mumbai, India.

2.2 Preparation of PVB-ZnO composite films:-

The PVB-ZnO composite films were fabricated using the solution casting technique. 5gm powder of polyvinyl butyral, 0.5 wt% and 1wt % of ZnO was dissolved in chloroform (50ml) and stirred using a magnetic stirrer at 500rpm at room temperature. A homogeneous viscous gel is obtained after 30 hours. This gel is transferred into Teflon Petri dishes, and left for drying slowly over 30 days of ambient temperature drying, a film of thickness around 0.7 mm is obtained. The prepared films were peeled off and cut into suitable dimensions for further analysis. Using the similar procedure, the composite films with 0.5 and 1wt. % of ZnO loaded in PVB matrix also prepared.

III. Characterization:-

The morphology of pure PVB and 1wt% ZnO doped Nanocomposite films XRD were studied by using an X-ray diffractometer (SHIMADZU XRD-7000) in the scattering range 2θ from10^0 to 80^0. These results are compared with the crystal XRD of ZnO which is given by the manufacturer. The interplanar distance (d) was determined by applying Bragg's formula ($2dsin\theta = n\lambda$), where θ, n, and λ are Bragg's angle, order of spectrum and wavelength (1.5406 Å.) of the target $(Cu - K_\alpha)$ material used, respectively). DLS and Zeta potential studies of ZnO/PVB nanocomposite and its individual components are studies with help Horiba Scientific SZ-100 instrument which is operated at 10mW with 532nm laser light. The FESEM images of PVB and 1wt% ZnO added Nanocomposite is recorded in Solid State Division BARC Mumbai and these images are compared with the SEM image of ZnO which is given by the manufacturer.

3.1. X-ray diffraction:-

The typical X-Ray diffraction patterns for pure PVB, ZnO and PVB-ZnO Nanocomposite films with 1.0 wt. % samples were recorded and shown in Fig-1. The XRD pattern of pure PVB exhibited a large specific diffraction peak observed at scattering angle (19°<2θ<20°) represents the "d" spacing value of 0.45 nm, which is a typical result for high molecular-weight amorphous polymer. The XRD of Pure ZnO is given by the supplier (Ad-nano) is showing standard peaks of ZnO as per the literature. The pure PVB and complexed films showed another broad and low-intensity crystalline peak at a 2θ position around~42°. After composite were dispersed into the PVB matrix, the XRD of the resulted nano-composites only showed the PVB diffraction peak. This demonstrates that nano particles were homogenized in the PVB matrix. There are no changes found in the structure of the PVB matrix after ZnO being mixed, due to the plenty of hydroxyl groups present in its back-bone. This is evident for the purity and successful formation of ZnO loaded composite films. It can be seen from Fig-1 that the broad peak of the PVB polymer becomes more broadened with increasing nanoparticles concentration. This is the evidence for the increase of the amorphous phase in composite films in comparison with the pure PVB film, similarly the PVB shell is nullifying the crystallinity of ZnO due to core/shell structured composite formation.[2,7,8].

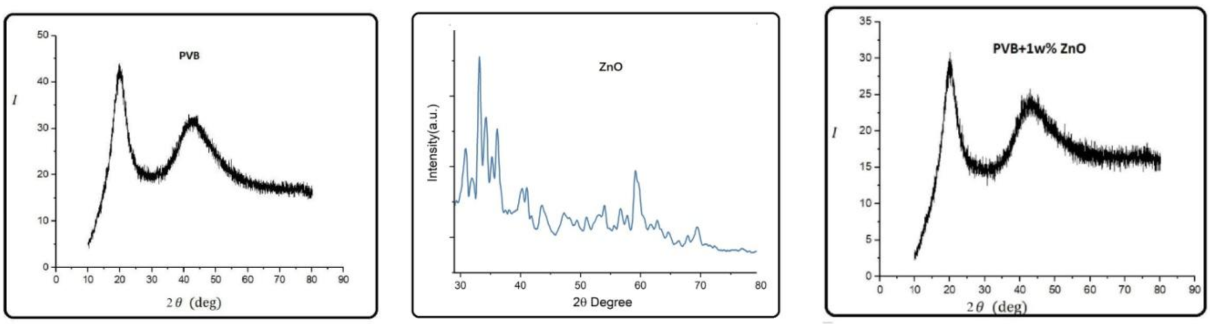


Fig**.1. XRD Pattern of PVB, ZnO and Their Nanocomposite**

3.2 Scanning Electron Microscopy:-

The SEM micrograph of PVB shows that the morphology is in fiber form with diameter 30nm and length

550nm. The SEM image of ZnO indicates that the particles are in spherical shape. Finally the Nanocomposite SEM image indicates compact structure with fewer voids. The image of nanocomposite reveals that there is agglomeration of ZnO in PVB matrix. However, ZnO are unevenly distributed in PVB matrix. According to the SEM images, it had been considered that the nanostructure ZnO particles are embedded within the structure built by PVB chains. It implies that in composite ZnO and PVB fibers are forming nanocomposite in Core-shell form and it is in highly dense with close interaction between ZnO and PVB [7-9].

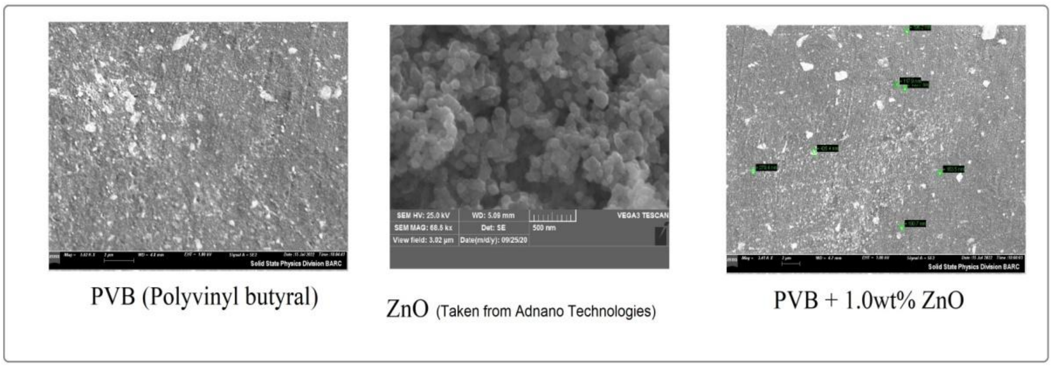


Fig. 2. SEM images of PVB, ZnO and Their Nanocomposite

3.3 Dynamic light scattering:-

Dynamic light scattering is a technique is used to determine the size distribution profile of small particles of PVB/ZnO and their individual components in chloroform solvent. The sample is illuminated by a laser beam and the fluctuations of the scattered light are detected at a known scattering angle θ by a fast photon detector. The diameter measured by DLS is a value that refers to how a particle moves within the solvent, called the hydrodynamic diameter. The Hydrodynamic diameters of PVB, ZnO and their composites are measured with help of Stokes Einstein equation. [11,12]

$$R_H = \frac{k_B T}{6\pi\eta D} \qquad \text{Where}$$

D Translational diffusion coefficient [m²/s] – "speed of the particles'

k_B Boltzmann constant [m²kg/Ks²], T Temperature [K], η Viscosity [Pa.s]

R_H Hydrodynamic radius [m]

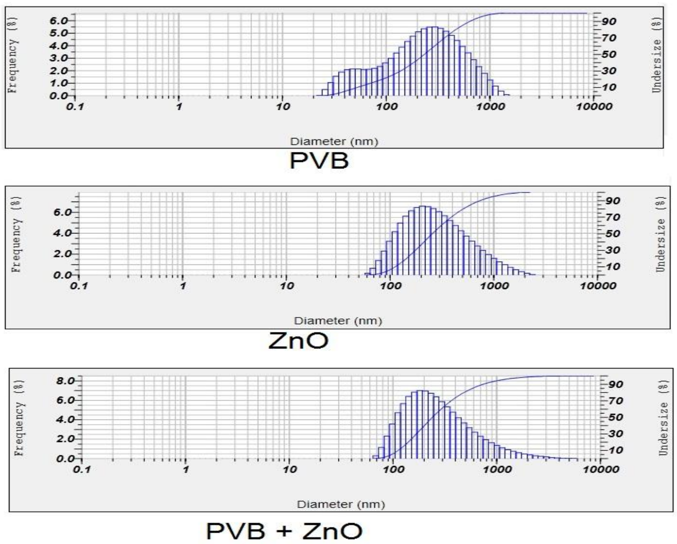


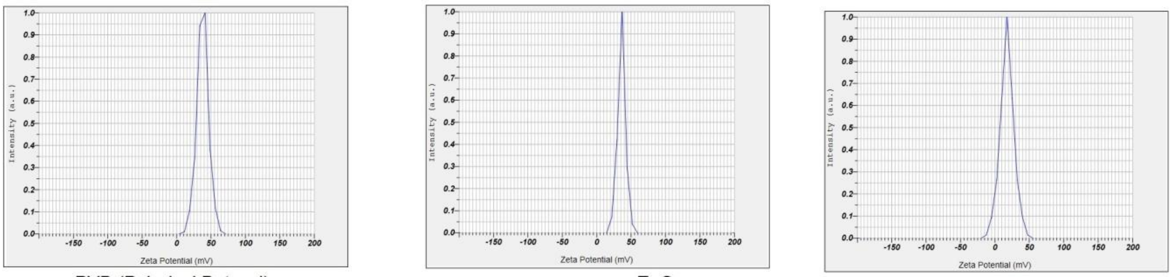
Fig. 3. DLS graphs of PVB, ZnO and Their Nanocomposite

The DLS studies are indicating that the diameter and lengths of pure PVB fiber are 47.7 nm and 333.4nmrespectively. The ZnO diameter is identified as 357.8nm. Similarly PVB/ZnO nanocomposite diameter is measured as 370.6 nm respectively. These results are indicating that the ZnO nanoparticles are showing highly agglomerated in chloroform and also swelling. Generally the hydrodynamic diameters are more than the real diameter. Hence the diameters recorded in DLS are more than the diameters recorded in FESEM. These

DLS studies are also conforming that PVB polymer is forming a shell type layer on ZnO agglomerated nanoparticles which was observed in FESEM studies [2, 11, 12]

3.4. The Zeta Potential Studies:-

The Zeta Potential, also termed as electro kinetic potential, is the potential at the slipping/shear plane of a colloid particle moving under electric field. Electric potential of a surface is the amount of work that needs to be done to bring a unit positive charge from infinity to the surface without any acceleration The Zeta potential reflects the potential difference between the EDL (electric double layer) of electro phonetically mobile particles and the layer of dispersant around them at the slipping plane.



PVB (Polyvinyl Butyral) ZnO PVB+ 1 wt% ZnO

Fig. 4 Zeta Potentials of PVB, ZnO and Their Nanocomposite

The zeta potentials of individuals and their Nanocomposite are measured in Methyl trichloride solvent and these values are 37.9 mV, 36.1 and 17.6 mV respectively. These Zeta potential studies are indicating that the nanocomposite is more unstable (Zeta potential less than 30 mV) than PVB and ZnO in $CHCl_3$ [11, 12].

IV. Conclusions:

It is concluded that we have prepared ZnO incorporated PVB matrix based nanocomposite films. The XRD analysis has shown that crystallinity of ZnO is lost due to interaction with amorphous PVB in the nanocomposite. . The FESEM, DLS studies are indicating that ZnO and PVB are forming Core-Shell structure Nanocomposite. The zeta potentials of individuals and their Nanocomposite are measured in chloroform solvent.

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