



Analysis of structural, morphological, electrical properties of MgO oxides

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Abstract

Magnesium oxide (MgO) is a widely studied metal oxide due to its remarkable structural stability, high thermal resistance, and significant electrical insulation properties. In the present study, a comprehensive analysis of the structural, morphological, and electrical properties of MgO oxides has been carried out using various characterization techniques. XRD-, which confirmed the formation of crystalline MgO with a cubic phase and good crystallinity. The average crystallite size, lattice strain, and dislocation density were evaluated from the diffraction data, indicating the influence of synthesis conditions on structural parameters. Morphological analysis was performed using SEM, revealing the surface features, particle size distribution, and agglomeration behavior of MgO particles. The images showed a homogeneous distribution with porous and granular structures, which are beneficial for surface-related applications. The electrical properties were studied using techniques such as four probe method

Keywords: Polyaniline (Pn), magnesium oxide (MgO)_ DC, XRD, SEM

Introduction

Polymer science has emerged as a crucial field due to the remarkable versatility, tunability, and wide range of physicochemical properties exhibited by polymers. Their properties—including mechanical strength, thermal stability, electrical conductivity, and optical behaviour can be tailored by controlling molecular structure, composition, and synthesis methods [1, 2]. This adaptability makes polymers indispensable in diverse applications such as packaging, electronics, biomedical devices, coatings, and energy storage systems. Conducting polymers have attracted significant attention in recent decades due to their unique combination of electrical conductivity and polymeric properties [3, 4]. Among them, Polyaniline (PANI) is one of the most widely studied materials because of its ease of synthesis, environmental stability, low cost, and tunable electrical conductivity. PANI exists in different oxidation states, among which the emeraldine salt form exhibits high electrical conductivity, making it suitable for applications in sensors, supercapacitors, and electronic devices (Stejskal & Gilbert, 2002) [6]. Despite its advantages, pure PANI often suffers from poor mechanical strength and limited thermal stability, which restrict its practical applications. To overcome these limitations, researchers have focused on the development of polymer composites by incorporating inorganic fillers into the PANI matrix [5, 8]. One such promising material is Magnesium oxide (MgO), a wide band gap metal oxide known for its excellent thermal stability, high dielectric strength, and chemical inertness (Kumar & Singh, 2023) [1, 11]. The incorporation of MgO nanoparticles into the PANI matrix leads to the formation of PANI/MgO nanocomposites, which exhibit improved structural, electrical, and dielectric properties. The interaction between PANI chains and MgO particles enhances charge transport mechanisms and provides better interfacial polarization, thereby improving conductivity and dielectric behavior (Zhang *et al.*, 2024) [5, 7, 9, 10, 16]. Additionally, MgO acts as a reinforcing agent, enhancing the mechanical strength and thermal stability of the composite material. Recent studies

have demonstrated that PANI/MgO composites exhibit enhanced performance in applications such as gas sensing, electromagnetic shielding, energy storage devices, and humidity sensors due to their synergistic properties (Raza *et al.*, 2023; Sharma *et al.*, 2025) [3, 4, 11, 13, 15]. Furthermore, structural characterization techniques such as X-ray diffraction (XRD) confirm the successful incorporation of MgO into the PANI matrix by showing distinct diffraction peaks corresponding to both components, indicating the formation of a well-defined composite structure. The insulated emeraldine base of the polyaniline can be made conductive and the conductivity can be enhanced by doping with protonic acids. By doping with protonic acids with polyaniline, which can enhance the conductivity of the composite by more than 10 orders of magnitude. The enhancement of the conductivity usually depends on the strength of the acids [14, 18].

Here, we report the results of Pn and PnMgO composite fabrication using the in-situ method. Compared to other fabrication methods, the in-situ method is obtained using the easiest methods to fabricate composites.

Experimental section

Materials used

The chemical reagents utilized in the synthesis process included aniline (99%), ammonium persulfate (APS) (99%), and hydrochloric acid (HCl) of analytical grade. All other supplementary chemicals used were of analytical reagent (AR) grade. All aqueous solutions were prepared using double-distilled water. Magnesium oxide (MgO) was employed for the preparation of composites via the chemical oxidative polymerization method. Pellets of Pn and Pn–MgO composites, with a diameter of 12.4 mm and a thickness of 1.18 mm, were fabricated by applying a pressure of 5 tons.

Preparation of Polyaniline (Pn)

Polyaniline (PANI) was synthesized using the chemical oxidative polymerization (COP) method. A 0.2 M aniline

solution was prepared and mixed with 1 N hydrochloric acid (HCl) under constant stirring for 2 hours at room temperature. Separately, a 0.25 M ammonium persulfate (APS) solution was prepared and added dropwise to the aniline–HCl mixture over 1 hour while maintaining the temperature at ~50 °C with continuous stirring. The reaction mixture was further stirred for 8 hours and then kept undisturbed overnight for complete precipitation. The resulting dark green precipitate was filtered using a vacuum pump, washed with deionized water, acetone, and 1 N HCl to remove impurities, and then dried in an oven at 50 °C for 24 hours. The final product was ground into a fine powder [19].

Preparation of PnMgO composite

The PANI–MgO composites were prepared using the same chemical oxidative polymerization (COP) procedure, with magnesium oxide (MgO) incorporated into the aniline–HCl solution prior to polymerization. The subsequent steps, including APS addition, stirring, filtration, washing, and drying, were carried out under identical conditions to obtain the PANI–MgO composite.

Result and discussion

XRD of Pn and Pn-MgO composite

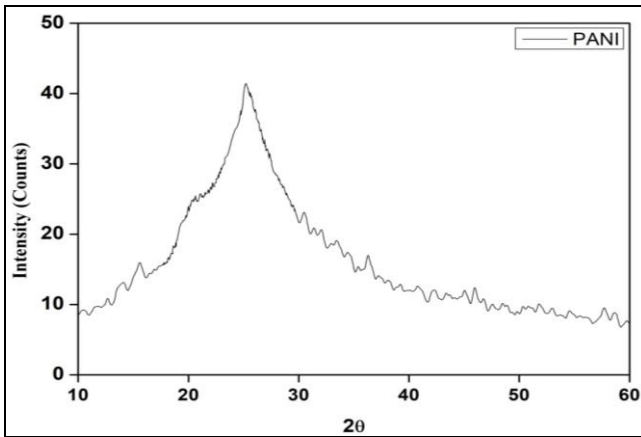


Fig 1: The X-ray diffraction (XRD) spectra of the pure Pn

Fig 1. Shows that the X-ray diffraction (XRD) spectra of the pure Pn and Pn–MgO composite samples were recorded in the 2θ range of 20° to 75°, and the corresponding patterns are presented in Figure.1a.. The XRD pattern of the Pn–MgO composite exhibits characteristic diffraction peaks corresponding to both Pn and MgO phases, indicating the successful incorporation and retention of Pn within the composite matrix. The prepared Pn–MgO composite shows well-defined diffraction peaks at 2θ values of 21.75°, 27.95°, and 30.5°, which are indexed to the (110), (200) and (210) crystallographic planes of magnesium oxide, respectively. These observed peaks are in good agreement with standard data reported in the JCPDS card No. 87-0653 and previously published literature, confirming the formation of the MgO phase in the composite [20, 24]. The average crystalline size of PN was calculated by using Scherrer's formula. The average crystalline size of PN was calculated by using Scherrer's formula

$$D = \frac{K\lambda}{\beta \cos \theta}$$

where $\lambda = 1.54060 \text{ \AA}$, θ is the Bragg angle, K is the Debye Scherrer constant and β is the peak full width at half maximum of the peak. The average crystallite size of PnMgO composite was found to be 15 nm.

Dislocation density (δ)

Dislocation density (δ) is a measure of the number of defects (dislocations) present in a crystalline material. It tells you how much the crystal structure is distorted.

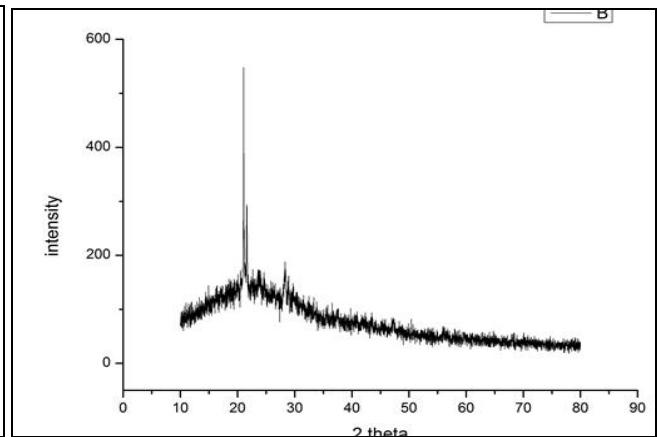


Fig 1a-: The X-ray diffraction (XRD) spectra of the Pn–MgO

Crystallite size $D \approx 15 \text{ nm} = 15 \times 10^{-9} \text{ m}$

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$\delta = 1 / (15 \times 10^{-9})^2$

$\delta \approx 4.44 \times 10^{15} \text{ lines/m}^2$ [26]

Scanning Electron Microscope

For the morphological study of material, the SEM technique was widely used in the science of modern. Also this technique was used to know the composition of the material with an additional favourable position of profundity of focus (~100 – 200 Å). Electron microscope uses the wave nature and rapidly moving electrons. The electrons accelerated to ten thousand keV and having wavelength of 0.12 Å. The magnification of the electron microscope has around the 10,00,000 diameter [27, 30]. A beam of electrons will be made to incident on the investigating sample, the beam of electrons will interact with the area of sample and generates the electrons by scattering process which includes many information related to the investigating sample. In the process of scattering of electrons from the investigating sample are categorized as elastically scattering and in-elastically scattering. The electrons scattered by the process of elastically scattering are known as 'backscattered electrons' and if scattered by the process of "in-elastically" are due to the loss in KE upon their interaction with e^- [31].

SEM image of PnMgO composite

The SEM micrographs for the pure Pn and PnMgO composite samples being magnified at 1000 times have been depicted in figure. The morphology of the Pn appears to be irregular shapes, non-fibrous with high densities. The SEM micrographs of the PnMgO composite suggest irregular arranged granular and flakes with sharp edge, looks non-porous and morphology with spherical, few oval-shaped

particles randomly distributed, micro size round shape particles with uniformity on the surface as well as a few agglomerations. The composite micrograph describing surface morphology slightly different from that of the micrograph of Pn suggesting the possible presence of magnesium oxide particles distributed in the polyaniline matrix. [32].

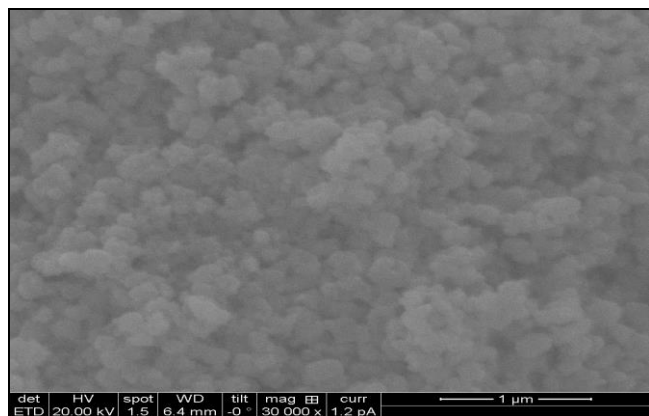


Fig 2: SEM micrographs of Pn

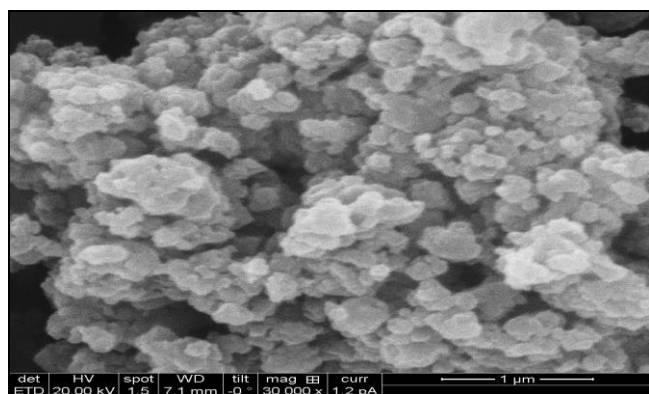


Fig 2a: The SEM micrographs for the PnMgO

DC electrical conductivity

The variation in the dc conductivity with change in temperature of Pn and PnMgO composites were carried out and represented in figure 3. The dc conductivity of all the samples increases with increase in temperature exhibits the semiconductor behavior and it rises with increase in content of MgO in the Pn matrix. This indicates the MgO particles gives positive influence on composite towards increase in conductivity. Figure 3 indicates the dc conductivity as function of different wt% of MgO. The result shows that the MgO has positive influence on the temperature dependent conducting property of the Pn. The dc conductivity of both samples increases in two phases, i.e., low temperature region and high temperature region. There is higher order increase in the conductivity at higher temperature phase and lowered conductivity at low temperature phase. The increase in the conductivity at higher temperature due excitation of electrons to the conduction band at higher temperature [33, 34]. Among the entire prepared composite, the maximum DC conductivity value was obtained with a value of 2×10^{-2} S/m for MgO at 180° temperature also depicted in figure.

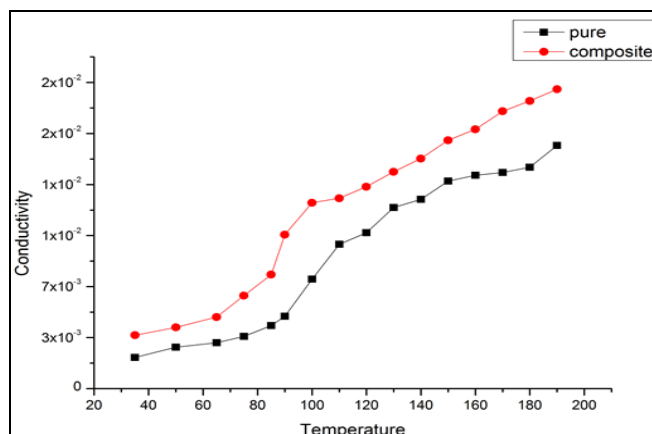


Fig 3: DC conductivity of Pn and PnMgO composites

Conclusion

Pn and PnMgO composite was prepared by incorporating MgO particles into the Pn chain using the COP technique. Moreover, the Pn and composite were examined in detail for their morphological and structural characteristics using the XRD and SEM techniques. The large peak at $2\theta \approx 25^{\circ}$ described by the pure Pn XRD pattern reveals the amorphous nature of the produced Pn, while the PnMgO composite's XRD pattern demonstrates the presence of MgO particles in the Pn with crystalline MgO peaks. The morphological analysis explains the aggregation of Pn semi-crystalline structure and homogenous surface morphology. The maximum dc conductivity value 2×10^{-2} S/m measured 195° degree temperature of MgO.

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