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Structural Properties of Conducting PANI/Y_2O_3 Nanocomposites Synthesized by In-Situ Chemical Oxidative Polymerization

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ABSTRACT

Conducting polymer nanocomposites have attracted significant attention due to their enhanced structural, electrical, optical, and sensing properties. In the present investigation, polyaniline (PANI)/yttrium oxide (Y_2O_3) nanocomposites were synthesized by an in-situ chemical oxidative polymerization technique using ammonium persulfate as an oxidizing agent in hydrochloric acid medium. Nanocomposites containing 10, 20, 30, 40, and 50 wt.% Y_2O_3 were successfully prepared. The structural and morphological characteristics of the synthesized materials were analyzed using X-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD analysis confirmed the formation of semicrystalline PANI/Y_2O_3 nanocomposites with an average crystallite size of approximately 11 nm. SEM micrographs revealed highly interconnected sponge-like and chain-like nanostructures with uniform dispersion of Y_2O_3 particles within the PANI matrix. The incorporation of Y_2O_3 significantly modified the structural characteristics of PANI while retaining the crystalline nature of the oxide nanoparticles. The synthesized nanocomposites are promising candidates for electronic devices, gas sensors, electromagnetic shielding, and energy storage applications.

1. Introduction

Conducting polymers have emerged as an important class of functional materials because of their unique combination of electrical conductivity, environmental stability, lightweight nature, and ease of synthesis. Among various intrinsically conducting polymers, polyaniline (PANI) has received considerable attention owing to its reversible proton doping mechanism, excellent environmental stability, low production cost, and relatively high electrical conductivity.

Recently, polymer nanocomposites containing metal oxide nanoparticles have become an active area of research because the incorporation of inorganic nanofillers substantially enhances the structural, electrical, thermal, optical, and sensing properties of conducting polymers. Yttrium oxide (Y_2O_3) is a rare-earth metal oxide possessing excellent thermal stability, high dielectric constant, chemical inertness, and optical transparency. The incorporation of Y_2O_3 nanoparticles into the PANI matrix is expected to improve crystallinity, interfacial interaction, and charge transport characteristics.

Various synthesis techniques have been reported for preparing conducting polymer nanocomposites, including chemical oxidative polymerization, electrochemical polymerization, sol-gel processing, and in-situ polymerization. Among these, the in-situ chemical oxidative polymerization method is simple, economical, and suitable for large-scale production while ensuring homogeneous dispersion of nanoparticles.

The present work focuses on the synthesis of PANI/Y_2O_3 nanocomposites with different weight percentages of Y_2O_3 and investigates their structural and morphological properties using XRD and SEM techniques.

2. Materials and Methods

2.1 Materials

The chemicals used in this investigation were of analytical reagent (AR) grade and used without further purification.

- Aniline ($C_6H_5NH_2$)
- Ammonium Persulfate (APS) ($(NH_4)_2S_2O_8$)
- Hydrochloric Acid (HCl)
- Yttrium Oxide (Y_2O_3)
- Double distilled water

2.2 Synthesis of Pure Polyaniline

Pure polyaniline was synthesized using the chemical oxidative polymerization method. A 0.1 M aniline solution was prepared in 1 M hydrochloric acid. The oxidizing agent, 0.1 M ammonium persulfate, was added dropwise under continuous magnetic stirring. The reaction was maintained at approximately 70°C for four hours. The resulting dark-green precipitate was filtered, repeatedly washed with deionized water until neutral pH was obtained, and dried in a hot-air oven at 60°C for 24 hours.

2.3 Synthesis of PANI/Y_2O_3 Nanocomposites

Different weight percentages (10, 20, 30, 40, and 50 wt.%) of Y_2O_3 nanoparticles were dispersed in the aniline-HCl solution under vigorous stirring. Polymerization was initiated by the slow addition of APS solution. The reaction mixture was continuously stirred for four hours at 70°C. The obtained nanocomposite powders were vacuum filtered, washed thoroughly with distilled water, and dried in an oven for 24 hours.

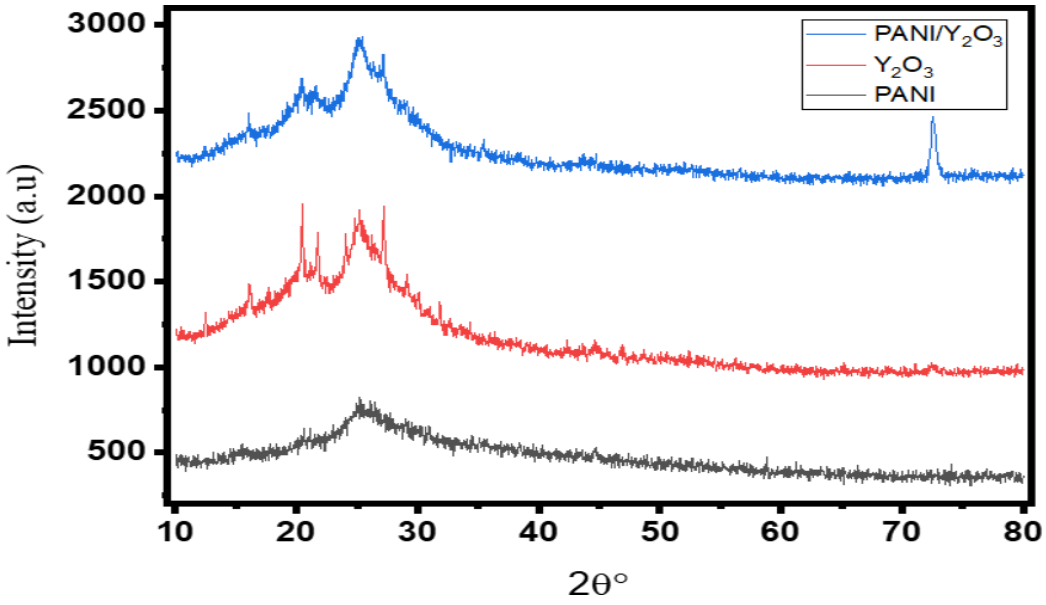
3. Characterization

The synthesized samples were characterized using the following techniques:

1. **X-ray Diffraction (XRD):** The crystal structure and phase identification were carried out using a Philips X-ray diffractometer with Cu-Kα radiation (λ = 1.5406 Å).
2. **Scanning Electron Microscopy (SEM):** Surface morphology was examined using an EVO-18 Carl Zeiss scanning electron microscope.
3. Powder samples were pelletized under a pressure of 10 tons before SEM observation.

4. Results and Discussion

4.1 X-ray Diffraction Analysis



The XRD patterns of pure PANI, pure Y_2O_3, and PANI/Y_2O_3 nanocomposites are presented in Figure 1. Pure PANI exhibited a broad diffraction peak centered at approximately 2θ = 25.56° confirming its predominantly amorphous nature. The broad diffraction arises from periodicity parallel and perpendicular to the polymer chains.

In contrast, pure Y_2O_3 displayed several sharp diffraction peaks corresponding to its cubic crystalline phase, indicating high crystallinity. After incorporation of Y_2O_3 nanoparticles into the PANI matrix, the diffraction peaks of Y_2O_3 remained clearly visible along with the broad PANI peak. This confirms that the crystalline structure of Y_2O_3 was preserved during polymerization. The coexistence of broad and sharp diffraction peaks indicates the formation of a semicrystalline nanocomposite.

The average crystallite size was estimated using the Debye-Scherrer equation:

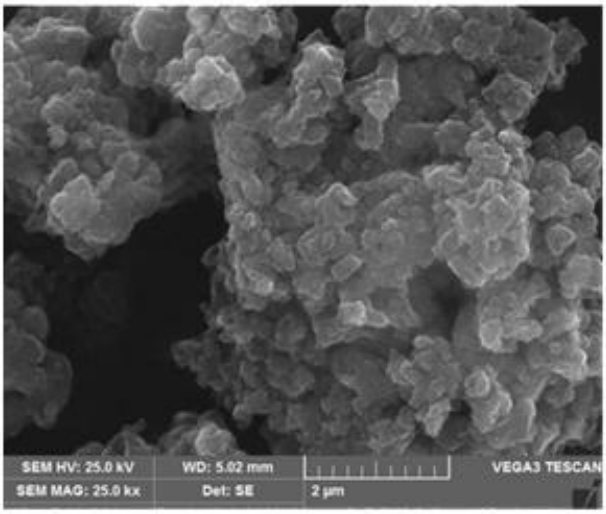
$$D = 0.89\lambda / (\beta \cos\theta)$$

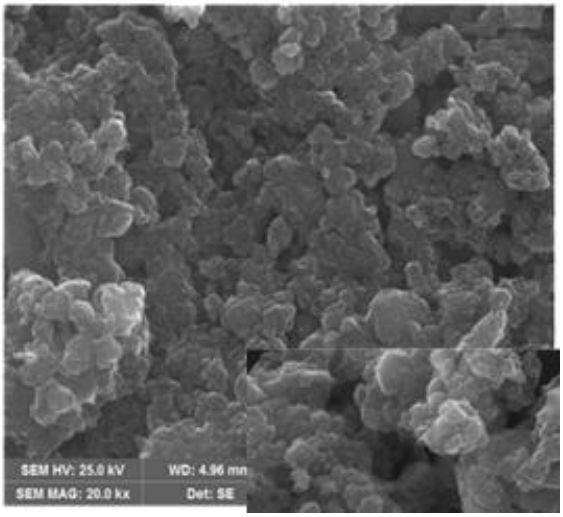
where:

- D = crystallite size
- λ = wavelength of X-rays
- β = Full Width at Half Maximum (FWHM)
- θ = Bragg angle

The average crystallite size was found to be approximately 11 nm, confirming the nanocrystalline nature of the synthesized composite.

4.2 SEM Analysis





SEM images of pure PANI and PANI/Y_2O_3 nanocomposites are shown in Figures 2 and 3. Pure PANI exhibits an irregular agglomerated morphology consisting of interconnected granular particles. The average particle size estimated using the linear intercept method ranges from 45 to 65 nm.

After the addition of Y_2O_3 nanoparticles, the morphology changes considerably. The nanocomposite exhibits sponge-like and chain-like interconnected structures with improved particle distribution. The Y_2O_3 nanoparticles appear uniformly dispersed throughout the conducting polymer matrix, indicating strong interfacial interaction between the polymer chains and oxide nanoparticles. Such porous morphology is advantageous for gas sensing, energy storage, and catalytic applications because of the increased surface area.

5. Formation Mechanism

During oxidative polymerization, APS oxidizes aniline monomers to radical cations, which subsequently couple to form long PANI chains. Simultaneously, dispersed Y_2O_3 nanoparticles act as nucleation centers, facilitating the growth of polymer chains around their surfaces. This leads to homogeneous dispersion of nanoparticles within the polymer matrix and enhances structural stability.

6. Applications

The synthesized PANI/Y_2O_3 nanocomposites possess potential applications in:

1. Gas sensors
2. Supercapacitors
3. Rechargeable batteries
4. Electromagnetic interference (EMI) shielding
5. Flexible electronics
6. Corrosion-resistant coatings
7. Chemical sensors
8. Energy storage devices

7. Conclusions

PANI/Y_2O_3 nanocomposites were successfully synthesized through the in-situ chemical oxidative polymerization technique. XRD analysis confirmed the semicrystalline nature of the nanocomposites with an average crystallite size of approximately 11 nm. SEM studies revealed sponge-like interconnected nanostructures and homogeneous dispersion of Y_2O_3 nanoparticles throughout the PANI matrix. The incorporation of Y_2O_3 effectively modified the structural characteristics of PANI while preserving the crystalline phase of the oxide. These nanocomposites exhibit improved structural properties and are promising materials for future applications in sensing, electronics, and energy storage technologies.

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