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Synthesis of SiO_2 nanoparticles and their effect on polystyrene: structural analysis

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Abstract

In this study, polystyrene (PS)/silicon dioxide (SiO_2) nanocomposites were fabricated using an eco-friendly, fluoride-assisted thermochemical synthesis method for nanoparticle production. This approach offers a sustainable and scalable route to obtaining high-purity, amorphous SiO_2 nanoparticles, which were subsequently incorporated into a PS matrix to enhance its optical performance. Structural, morphological, and spectroscopic analyses were carried out using SEM, XRD, and Raman spectroscopy. SEM revealed a uniform dispersion of SiO_2 nanoparticles within the PS matrix, while XRD confirmed the amorphous nature of the silica phase. Raman spectroscopy indicated notable changes in the vibrational dynamics of PS upon nanoparticle incorporation, particularly a reduction in phenyl ring bending vibrations, suggesting strong polymer–nanoparticle interactions. The inclusion of SiO_2 not only altered the structural characteristics but also introduced interfacial effects that can modulate the optical behavior of PS. These results demonstrate that incorporating fluoride-synthesized SiO_2 nanoparticles is an effective strategy for engineering PS-based nanocomposites with improved optical properties, making them suitable candidates for applications in optoelectronics and photonic devices.

Keywords: Polystyrene (PS), Nanocomposite films; Si-O-Si vibrations; Thermochemical synthesis; Interfacial energy states; Light absorption enhancement
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1. Introduction

Polymer-based nanocomposites have emerged as a promising class of materials due to their tunable physical, chemical, and optical properties. Incorporating inorganic nanoparticles into polymer matrices can significantly improve characteristics such as thermal stability, mechanical strength, dielectric behavior, and optical activity, thereby broadening their applicability in advanced technologies including optoelectronics, coatings, and photonic devices [1, 2].

Among various polymers, polystyrene (PS) has attracted considerable attention owing to its transparency, low cost, ease of processing, and good film-forming properties. However, its inherent limitations such as brittleness, low thermal stability, and limited optical absorption range restrict its performance in high-demand applications. These drawbacks can be mitigated by embedding inorganic nanoparticles into the polymer matrix, creating hybrid materials with synergistically enhanced functionalities [3].

Silicon dioxide (SiO_2) is one of the most commonly used nanoparticles in polymer nanocomposites due to its excellent optical transparency, chemical inertness, nontoxicity, and strong interfacial interactions with polymer chains [4, 5]. The inclusion of SiO_2 into PS has been shown to influence not only the structural and mechanical properties but also the optical behavior of the composite material. In particular, the presence of SiO_2 nanoparticles can introduce localized energy states at the polymer–nanoparticle interface, which in turn affects the optical absorption and band gap characteristics of the host polymer [6, 7].

Most conventional methods of SiO_2 synthesis involve harsh chemicals or produce significant amounts of waste, which poses environmental concerns. Therefore, there is growing interest in developing eco-friendly, cost-effective, and scalable approaches for nanoparticle synthesis [8]. In this work, SiO_2 nanoparticles were synthesized using a thermochemical route assisted by fluoride ions, which is not only environmentally benign but also enables the efficient recycling of byproducts. This sustainable method provides a viable pathway for producing high-purity SiO_2 nanoparticles suitable for composite fabrication.

The aim of this study is to investigate the effect of SiO_2 nanoparticle incorporation on the optical properties of polystyrene. Using a combination of spectroscopic and microscopic techniques, we analyze the structural and interfacial changes induced by the nanoparticles and assess how these modifications influence the light absorption and optical band gap of the resulting nanocomposites. The findings from this work offer valuable insights into the design of PS-based materials with enhanced optical performance for potential applications in optoelectronics and photonics.

2. Materials and Methods

2.1. Materials

All chemicals were used as received without further purification. Polystyrene (PS), with CAS number 9002-86-2, has a density of 1.04 g/cm^3, a melting temperature range of 150-220°C, a spark temperature of 625°C, an ignition temperature of 500°C, and an auto-ignition temperature exceeding 1100°C. Tetrahydrofuran (THF), with PLC 143537, was also used as received. The high-purity silicon dioxide (SiO_2) nanoparticles used in this study were synthesized via an environmentally friendly, waste-free process involving the thermochemical treatment of a fluorinated precursor.

2.2. Synthesis of SiO_2 nanoparticles and PS/SiO_2 Nanocomposite

High-purity SiO_2 nanoparticles were synthesized via an environmentally friendly, nearly waste-free process using ammonium fluoride as the fluorinating agent. A mixture of 10 g ash and 37 g aqueous catalyst was sealed in a rotating reactor and heated in stages: 85–90°C initially, 125°C for several hours, and finally 180°C for two hours. Water and ammonia by-products were condensed and collected. At 300–320°C, ammonium hexafluorosilicate sublimed, was collected, dissolved in water, and reacted with 15–20% ammonia to yield solid SiO_2. The product was filtered, and the filtrate containing ammonium fluoride was evaporated for reuse. To prepare the nanocomposite, polystyrene (PS) was dissolved in tetrahydrofuran (THF), and 3 vol% of SiO_2 nanoparticles was added and stirred for 2 h. The mixture was cast in a Petri dish, air-dried, then vacuum-dried for 1 h. A thin film was formed by hot pressing at 180°C and 10 MPa, followed by water cooling.

2.3. Instrumentation

The surface morphology of the nanocomposites was examined using a scanning electron microscope (SEM; JEOL JSM-6610 LV) to analyze the dispersion and size of the nanoscale titanium oxide particles in the polymer matrix. Scanning was performed at an accelerating voltage of 30 kV.

X-ray diffraction analysis (XRD) was carried out using a Rigaku Mini Flex 600 XRD diffractometer at room temperature. For all experiments, Cu Kα radiation operating at 15 mA and 30 kV was utilized. The samples were scanned over a Bragg angle (2θ) range of 10°–90°.

The Raman spectra of the samples was recorded using an EnSpectr R532 Raman spectrometer (Enhanced Spectrometry Inc., USA). A green laser with a 532 nm wavelength was used for excitation of the samples.

The thickness of the nanocomposite film (100 µm) was measured using a digital micrometer (Model EH100) with an accuracy of 0.01 µm.

2.3.1. Scanning electron microscopy (SEM)

The surface morphology of PS/SiO_2 nanocomposites was analyzed using Scanning Electron Microscopy (SEM). Figure 1 shows the surface morphology of the PS/SiO_2 nanocomposite at 3,000x magnification, revealing bright spots. The distinct SiO_2 particles embedded in the polymer matrix indicate a relatively uniform distribution, which is beneficial for enhancing the composite's properties.

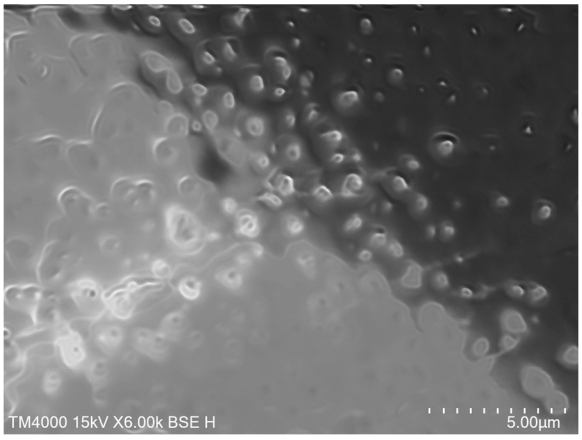

Fig. 1. SEM Micrograph and Elemental Distribution of PS/SiO_2 Nanocomposite

2.3.2. X-Ray Diffraction (XRD)

The structural properties of pure PS and PS/SiO_2 nanocomposites were analyzed using X-ray diffraction (XRD) (Fig. 2). The XRD pattern of SiO_2 nanoparticles shows a broad peak around $2\theta \approx 22^\circ$, characteristic of amorphous silica [8]. In the PS/SiO_2 nanocomposite, this broad feature is present but slightly shifted and less intense, indicating the dispersion of SiO_2 within the polymer matrix without significant crystallization. The lack of additional sharp peaks confirms that the SiO_2 remains in an amorphous phase.

2.3.3. Raman Spectroscopy

The Raman spectra of pure PS, SiO_2 nanoparticles, and their PS/SiO_2 nanocomposite are given in Figure 6. Unlike the pure PS spectrum, the SiO_2 spectrum shows a broad band, and a narrow peak with weak intensity appears at 486 cm^{-1}. This type of spectrum is indicative of the amorphous nature of SiO_2. [9] When SiO_2 is incorporated into the PS matrix, the Raman spectrum of the PS/SiO_2 nanocomposite appears “broadened,” and although the intensity of the peaks corresponding to PS is weakened, their positions remain unchanged.

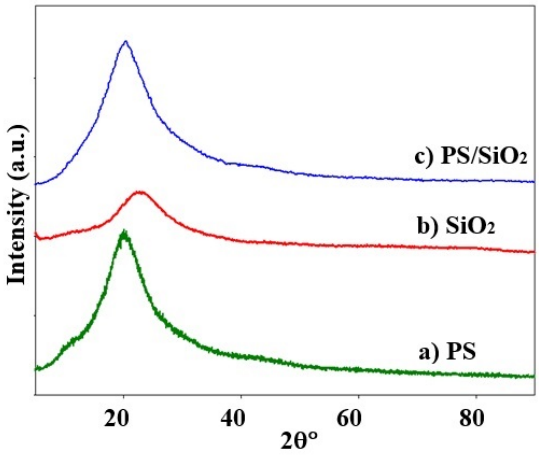

Fig. 2. XRD spectra of: a) Pure PS; b) PS/SiO_2 nanocomposite and c) SiO_2 nanoparticles

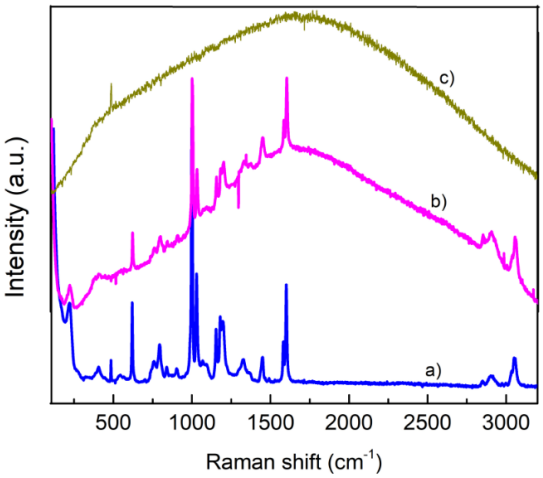

Fig. 3. Raman spectra of a) Pure PS; b) PS/SiO_2 nanocomposites and c) SiO_2 nanoparticles

Interestingly, a peak at 485 cm^{-1}, attributed to the bending vibration of the phenyl ring, is observed in the pure PS spectrum. In the PS/SiO_2 nanocomposite spectrum, this peak is nearly absent. This suggests that the incorporation of SiO_2 affects the vibrational modes of PS, likely reducing the intensity of the phenyl ring bending vibration at 485 cm^{-1} [10].

The observed changes in the Raman spectra, particularly the shift in intensity of the phenyl ring bending vibration, suggest that the SiO_2 nanoparticles interact with the PS matrix. This interaction likely modifies the polymer's molecular structure, potentially enhancing the properties of the PS/SiO_2 nanocomposite, such as

its mechanical strength or thermal stability. These findings further support the incorporation of SiO_2 nanoparticles as an effective way to tailor the characteristics of polymer nanocomposites for advanced applications.

4. Conclusion

This work presents a sustainable and efficient method for synthesizing SiO_2 nanoparticles via a fluoride-assisted thermochemical process and demonstrates their successful incorporation into a polystyrene matrix to form optically enhanced nanocomposites. SEM analysis confirmed the uniform dispersion of nanoparticles, while XRD and Raman studies revealed that SiO_2 remains amorphous and interacts strongly with the PS matrix. These interactions lead to significant modifications in the polymer's vibrational modes, particularly the attenuation of phenyl ring bending vibrations, indicative of molecular-level changes. The environmentally friendly synthesis route, coupled with the improved optical and structural characteristics of the resulting nanocomposites, underscores their potential for advanced applications in photonics and optoelectronic technologies. Future work will focus on optimizing nanoparticle loading and exploring the electrical and photoluminescent properties of these hybrid materials.

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