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Novel Dispersion of CeO_2 Nanofiller in PEO/PMMA Blended Nanocomposite Solid Polymer Electrolytes †

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Abstract: The present study focuses on the electrochemical performance of polyethylene oxide (PEO)-polymethyl methacrylate (PMMA) blended plasticized nanocomposite solid polymer electrolytes (BPNSPEs) amid cadmium bromide ($CdBr_2$) as a dopant salt along with a cerium oxide (CeO_2) nanofiller. Incredibly thin nanofilms of BPNSPE were signalized through distinct methods of working in characterization studies, such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), ultraviolet-visible spectroscopy and scanning electron micrography (SEM). The X-ray diffractograms (XRDs) confirmed the formation of the polymer electrolyte (PE) as well as a decrease in the degree of crystalline characteristics in the BPNSPE sample, and the particle dimension was calculated via the Debye–Scherer equation. The structural changes and formation of complexes were inspected by Fourier-transform infrared spectroscopy (FT-IR), and ocular absorbance scrutiny was accomplished by ultraviolet visible spectroscopy, whereas the morphological structure was interpreted by scanning electron microg-graphical images. The existing work is intended to increase the awareness of the significance of CeO_2 nanofillers with the BPNSPE arrangement, which is suitable for batteries and ionic devices.

Keywords: X-ray diffraction; cerium oxide; UV; SEM

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1. Introduction

The nanotechnology concept features various branches of science, whereas the acquired nanotechnology might encourage various sources of synthesized nanoparticles to procure enormous utilizations in electronics, agriculture, chemical industries, etc. The enduring development and comprehensive use of automobile gadgets are most significant in the field of nanotechnology to increase the requirement for secondary batteries where the electrical energy is stored [1]. Recently, polyethylene oxide polymers, which are ecologically safe, have progressed significantly [2–4]. This field has expanded from the classical period, and, in the modern period, scientists have concentrated on the development of polymer electrolytes [5,6]. There are three types of polymer electrolytes (Pes), namely hydro gel polymer electrolytes, liquid crystal polymers and blended composite polymers. Hydro gel polymer electrolytes and liquid crystal polymers have high ionic conductivity but do not have the ability to mitigate current issues when the positive electrode and negative electrode of the electrolyte sample are interfaced [7]. So, to examine this problem, a blended composite polymer electrolyte was taken into description. Blended composite PE is a form of polymer, which includes salts and inorganic filers that are inert, but the non-conductive constant is high in order to balance the potential by constraining the configuration of ion pairs in the form of polymeric matrices [8].

Polyethylene oxide is established in the theory of electrolyte polymers, which can break down assorted salts by appearing in the interactivity of EO, whatever is considered to be the most excellent electrolyte polymers, which are globally employed in battery studies. Commonly, polyethylene oxide is a semi-crystalline polymer that exhibits superior ionic conductivity and tremendous interfacial stability [9], but the stability of PEO decreases at higher temperatures. Hence, to develop the thermal establishment and crystallinity of PEO, it is blended with PMMA, which is an acrylic, amorphous polymer. In particular, polymethyl methacrylate (PMMA), with its excessive tensile firmness, might be acceptable to upgrade the mechanical solidity of the host polymer, and it is most compatible with PEO in the melting state. In order to enhance the conductivity, new inorganic salt, namely $CdBr_{2,}$ was utilized, in which Cd^{2+} ions interacted with ether oxygen (EO) of PEO in order to increase the mobility of charge carriers. Several researchers attempted different ceramic nanofillers, such as SiO_2, Al_2O_3, ZnO and SnO_2 [10–12], with the intention of increasing the potential and standardized stability of the blended PE. Here, we utilized cerium oxide (CeO_2), which is one of the most favorable metal oxide nanofillers. It behaves as a UV absorber and is globally applied in many fields, such as solar cells, catalysts, UV blockers, photo degradation of organic pollution and electronic devices [13].

This assignment aims to acknowledge the morphological and spectral investigation of BPNSPE by means of the cadmium bromide ($CdBr_2$) compound and its additional assimilation of the CeO_2 nanofiller by increasing the ionic conductivity and firmness, evaluated by using X-ray diffractograms, Fourier-transform infrared spectrum, ultraviolet-visible spectrum, along with scanning electron micro graphical (SEM) investigations.

2. Exploration Characteristics

2.1. Chemicals Acquired

Chemicals like polyethylene oxide (PEO), whose molecular weight is 5×10^5, polymethyl methacrylate (PMMA) whose M_n value is 996,000, cadmium bromide ($CdBr_2$) and cerium oxide (CeO_2) were bought from Sigma Aldrich for synthesis.

2.2. Manufacture of BPNSPE Nanofilm Arrangement

Nanofilms of BPNSPE were fused with 0.2 g of polyethylene oxide (PEO) and 0.1 g of polymethyl methacrylate (PMMA) with 0.024 g of cadmium bromide ($CdBr_2$) by the dissemination of 5 wt. % cerium oxide (CeO_2), whose compound size is less than 100 nm. PMMA polymer was put together by placing them in a vacuum drying oven at 373 Kelvin over a period of 15 min. Behind time, polyethylene oxide (PEO) and polymethyl methacrylate (PMMA) were liquefied in usual solvent acetone (30 mL), succeeded by using unmediated mixing for over an hour to produce a homogeneous fluid. The cadmium bromide ($CdBr_2$) sample was dried out using a vacuum drying oven at 373 Kelvin. Thereafter, the output dissipated towards polymer blends and swirled for an hour. In that moment, 5 wt. % of cerium oxide (CeO_2) inorganic ceramic filler was added to the solid complex and mixed for another 2 h, then poured in a washed Petri dish and parched under liberated air for one day at room temperature. Over time, impulsively secured samples of PEO-PMMA-$CdBr_2$ 5 wt. % of CeO_2 were accomplished and safe-guarded in desiccators for further scrutinization studies. A schematic depiction of the BPNSPE nanofilm is shown in Figure 1.

2.3. Idiosyncrasy Studies

The BPNSPE nanofilms were qualified in the recognition period, which is consummated in the form of emissions through a Bruker X-ray diffraction appliance model in a progressive way with copper kα, whose wavelength is 1.54 Angstrom, where its function appears in 30 milli-ampere at 2 theta between angles of 10° and 80°. Vibrational bands entailed in the prototype were ascertained by collecting the data over a wide region in the method of FTIR, which progressed by using the spectrophotometer model of Bruker Tensor 27. Likewise, the structural explication of the composite is induced by the spectrophotometer apparatus of Perkin Elmer ultraviolet spectrophotometer (Waltham, MA, USA). The morphological characteristics of BPNSPE composites were analyzed through Hitachi-S 3400N, which is illustrative of electron microscopy, and the compound's fragmentation portrayal was accomplished by keeping the specimen in liquid N_2, burnished with gold particles over 30 s and manipulated with the electrical potential of fifteen Kilo Volts.

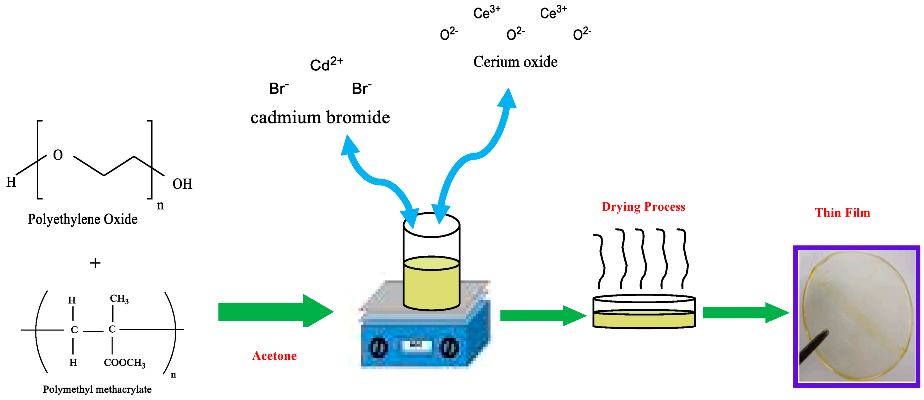

Figure 1. Schematic illustration for the preparation of BPNSPE nanofilm.

3. Results and Discussion

3.1. Microstructure Scrutiny of Blended Plasticized Nanocomposite Solid Polymer Electrolytes (BPNSPE)

The X-ray diffraction outline procured for PEO-PMMA-$CdBr_2$ as well as PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes (BPNSPE) is shown in Figure 2. Highly intense and sharp Bragg reflection angles scrutinized at 19.14° and 23.3° signified the semicrystalline characteristics of pure polyethylene oxide ascribed to (120) and (112) hkl planes [14], whereas the less intense reflection peak at 13.6° [15] resembled the PMMA polymer, expressing its amorphous nature. The diffraction peak at 26.1° harmonized to the (101) hkl plane of cadmium bromide ($CdBr_2$) salt (JCPDS File No. 10-0438). The intensity and elevation of prominent peaks are diminished in PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes (BPNSPEs), thus stipulating that the assimilation of CeO_2 nanoparticles agitates the crystalline zone, thereby increasing the disorderliness of the blended polymer electrolytes. In addition, new reflection peaks were monitored at $2\theta = 28.65°, 33.17°, 47.53°$ and 56.56°, which matched (111), (200), (220) and (311) hkl miller indices ascribing face-centered cubic unit cells of the CeO_2 nanofiller, as substantiated by the JCPDS File No. 81-0792. Thus, the depletion of intensity, emergence of new peaks and disorderliness of the blended polymer electrolytes may lead to more conducting pathways, thus making the polymer shapeless, thereby flourishing the finer transit of ions, as shown by Fourier-transform infrared spectroscopic examinations.

$$L = \frac{k\lambda}{\beta cos\theta} \tag{1}$$

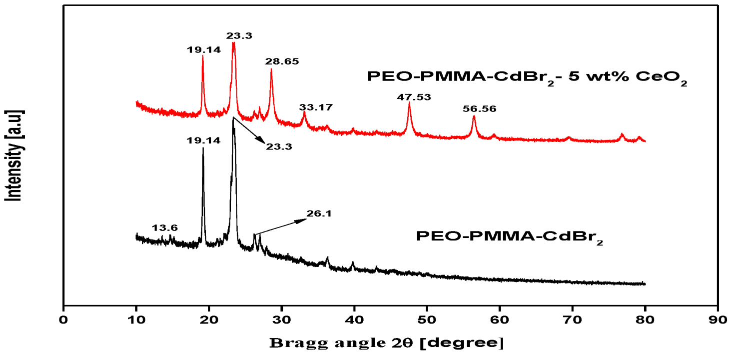

Figure 2. XRD profiles of PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes.

Here, k is the Scherrer shape factor constant ~0.89, λ is the wavelength of light in XRD, β-full width at half maxima, and θ is the Bragg reflection angle.

The size of the particle for the PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 BPNSPE sample was calculated using the Debye–Scherrer formula, utilizing Equation (1) [16] for the most fierce peak at 19.14° by deconvolution fitting of peaks using Origin Pro 8 Software, as delineated in Figure 3. The particle sizes of the samples are approximately 60 nm and 32 nm for the PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended nanocomposite solid polymer electrolyte complex, which points out that a reduction in particle size amplifies the amorphous nature, as exemplified in SEM studies.

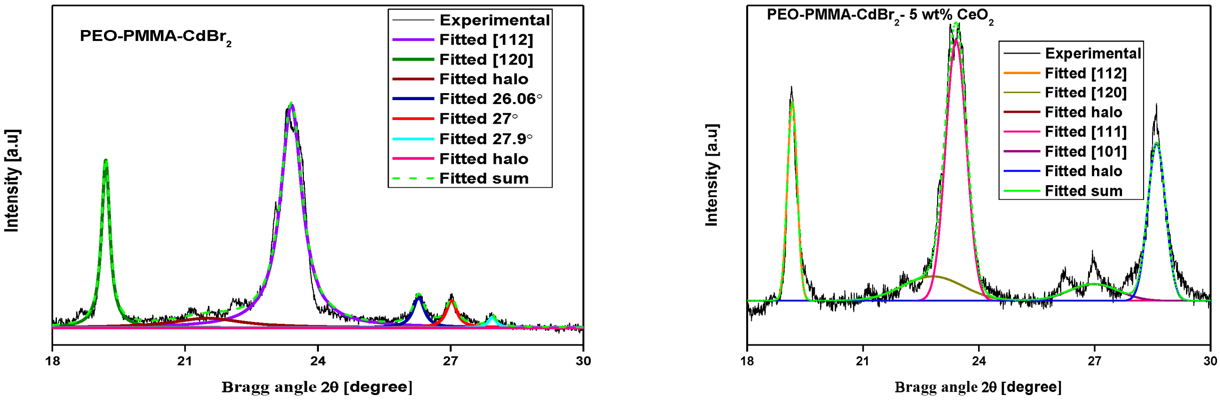

Figure 3. Deconvoluted XRD profiles acquired for PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolyte system.

3.2. Fourier-Transform Infrared Spectroscopic Results

The Fourier-transform infrared spectrum in favor of PEO-PMMA-$CdBr_2$ along with the PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended nanocomposite solid polymer electrolytes (BPNSPE) is shown in Figure 4. It is discerned that significant changes were ascertained after the assimilation of the CeO_2 nanofiller into the solid polymer electrolytes. The wave number noticed at 841 cm^{-1} as well as 961 cm^{-1} correlated to C-H_2 trembling [17] and C-H_2 rotating modes, appropriate for polyethylene oxide [18], and its position changed to 843 cm^{-1} and 954 cm^{-1} subsequent to the merging of the CeO_2 nanofiller into the polymer matrix.

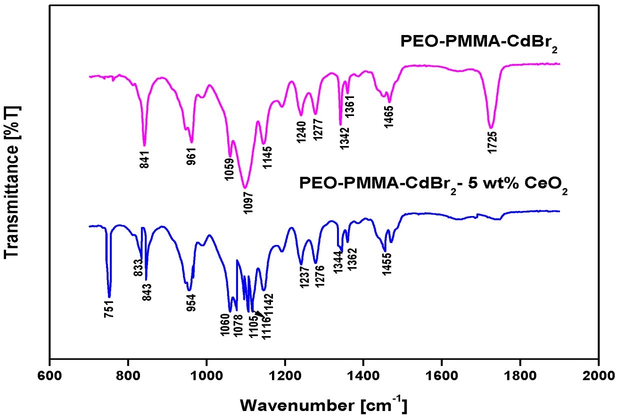

Figure 4. FT-IR transmittance spectrum of PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes.

Polyethylene oxide showed a semiconscious triad vibrational spectrum at 1145 cm^{-1}, 1059 cm^{-1} along with 1097 cm^{-1} comparable to the C-O-C stretching mode of vibration [19], which was relocated to 1142 cm^{-1}, 1060 cm^{-1} and 1105 cm^{-1} in the matrix. Similarly, the absorption peak situated at 1240 cm^{-1} and 1465 cm^{-1}, ascribed to C-H_2 symmetric stretching [20] and the C-H_2 scissoring mode of PEO [18], was dislodged to 1237 cm^{-1} and 1455 cm^{-1} in the ceramic filler complex. Furthermore, twin bands monitored at 1342 cm^{-1} and 1361 cm^{-1} embodied the C-H_2 fluttering form [21,22] transferred to 1344 cm^{-1} and 1362 cm^{-1}. In addition, the spectrum noticed at 1277 cm^{-1}, attributed near the C-C-O stretching mode of vibration of PMMA [23], was shifted to 1276 cm^{-1}, whereas the peak at 1725 cm^{-1} analogous to C=O stretching of PMMA [23] disappeared in the composite. Furthermore, four new spectra were viewed at 833 cm^{-1}, 751 cm^{-1}, 1078 cm^{-1} and 1116 cm^{-1} after the enclosure concerning the CeO_2 nanofiller, while a lower wavenumber vibrational peak was examined at 751 cm^{-1}, similar to Ce-O stretching bond [24] in the complex. The above sequels of spectral transformations may be due to the chemical reaction and structural changes taking place between the EO group of PEO, -CH_3 group of the PMMA polymer with Cd^{2+} cations of $CdBr_2$ and the CeO_2 nanofiller, as evidenced by the present XRD studies.

3.3. Absorption Study of Blended Plasticized Nanocomposite Solid Polymer Electrolytes (BPNSPEs)

The optical response acquired for PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes (BPNSPEs) in the absorption range 200–800 nm is shown in Figure 5. On the whole, the PEO-PMMA-$CdBr_2$ sample showed an absorption peak at 495 nm, which increased to 498 nm after the inclusion of the CeO_2 nanofiller in the polymer matrix, while pure cerium oxide usually shows an absorption peak around 298 nm [25]; however, here, the absorption peak was noticed at 498 nm in the PEO-PMMA-$CdBr_2$ -5 wt. % CeO_2 sample because the CeO_2 filler interacted with the polymer and salt, thus shifting the peak towards the longer wavelength region, which is due to the lower energy level to higher energy level exhilaration of the electrons, thereby increasing the mobility of charge carriers. The absorption peak observed in a higher wavelength range may be due to the transition of electrons from the 2p state valence band of the O^{2-} molecule present in the polymer to the 4f state conduction band of Ce^{4+} of the CeO_2 nanofiller [26].

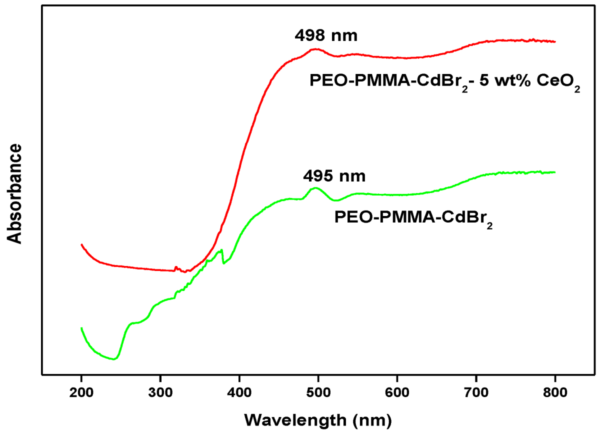

Figure 5. Optical absorption spectra of PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes.

3.4. Surface Morphology of Blended Plasticized Nanocomposite Solid Polymer Electrolytes (BPNSPEs)

SEM micrographs of PEO-PMMA-$CdBr_2$ along with PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended plasticized nanocomposite solid polymer electrolytes (BPNSPEs) are shown in Figure 6. The surface image of PEO-PMMA-$CdBr_2$ reveals the presence of compactly arranged spherical grains of approximately the same size, which indicates that the blend is highly crystalline in nature, whereas the CeO_2-integrated nanocomposite polymer electrolyte complex illustrates an astonishing improvement in terms of a structural alteration from the crystalline phase to the unstructured phase. It is evident that, owing to the accumulation of the CeO_2 nanofiller hooked onto the complex, the presence attributed to spherical grains completely disappeared, which further changed to a deformed shape, thus indicating a reduction in the crystal temperament of the blended thin film. Still, blended polymer, salt as well as nanofiller signify the smoothened region, which symbolizes the progress of ions to move at a faster rate [27]; thereby, the ionic conductivity of BPNSPE plays a major role, as evidenced amongst the above results.

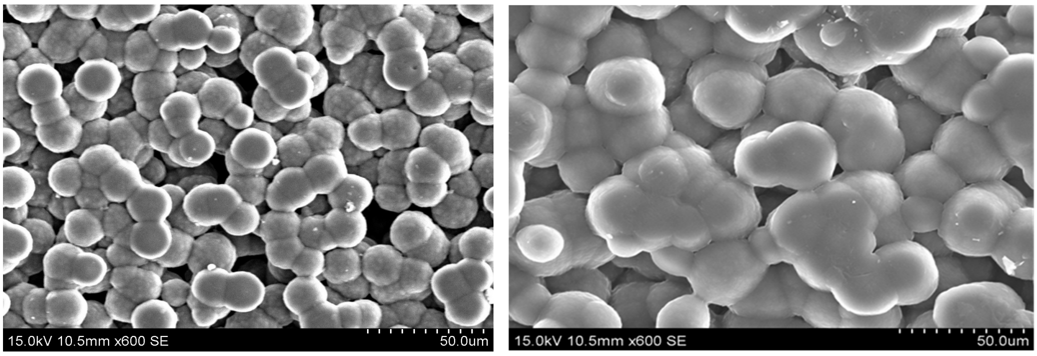

Figure 6. SEM photomicrographs appertaining to PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended nanocomposite solid polymer electrolytes.

4. Conclusions

The electrochemical performance of blended plasticized nanocomposite solid polymer electrolyte (BPNSPE) nano-emaciated films of PEO-PMMA-$CdBr_2$ and PEO-PMMA-$CdBr_2$-5 wt. % CeO_2 blended nanocomposite solid polymer electrolytes were tested through XRD, FTIR, UV and SEM investigations, and the polymer matrix was found to progress with a fall in crystal properties owing to the CeO_2 inorganic nanofiller, analyzed in the same way, through the abovementioned explorations. Thus, the present evaluation highlights the effect of the CeO_2 nanofiller on the PEO-PMMA-$CdBr_2$ complex; thereby, the transportability of conducting electrons accelerates, hence making it a favorable compound in upcoming research.

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