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A Decisive Study on Dielectric Response of Bi_2O_3/Polystyrene & Bi_2O_3/PVDF Composite as Flexible Electrodes for Energy Storage

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Abstract

In this manuscript a comparative study on Bi_2O_3/polystyrene and Bi_2O_3/PVDF composites has been executed via analysis of structural, bonding, surface morphology and dielectric response of composites for energy storage. The composites have been synthesized using solution cast method by varying concentrations of Bi_2O_3 (BO = 1 - 5 mw%) into polystyrene (PS) and polyvinylidene fluoride (PVDF) polymers respectively. X-ray diffraction confirms the generation of crystallinity, Fourier transform infrared (FT-IR) spectroscopy confirms bonding behavior and scanning electron microscopy (SEM) confirms uniform distribution of Bi_2O_3 (BO) in PS and PVDF polymers. Impedance spectroscopy has been employed for determination of dielectric response of the fabricated composites. The dielectric constant has been found to be increased as 1.4 times of pristine PS to $BO_{5\%}PS_{95\%}$ composites and 1.8 times of pristine PVDF to $BO_{5\%}PVDF_{95\%}$ composites respectively. These high dielectric composite electrodes are useful for flexible energy storage devices.

Keywords

Bismuth Oxide (Bi_2O_3), Polymer Composites, Surface Morphology, Dielectric Constant, Energy Storage

1. Introduction

Flexible supercapacitors are highly attractive for the large number of emerging portable lightweight consumer devices. The novelty of a flexible super capacitor is the incorporation of flexible electrode or substrate material to combine struc-

tural flexibility with the inherently high power density of supercapacitors. Flexible supercapacitors can use non-Faradaic energy storage process as seen in the electric double layer capacitor type or a Faradaic mechanism as seen in the pseudocapacitors (PCs). Materials with high dielectric constant attracted attention of researchers due to their suitability for energy storage applications. [1] [2] Generally, dielectric constant of pristine polymers is low, however these materials have drawn considerable interest for energy storage applications due to their easy processing, low cost, high breakdown strength and light weight. [3] [4] [5] If the permittivity of these polymeric materials could be increased then they might be suitable materials for energy storage applications. The loading of ceramic fillers increases permittivity of polymers. [6] [7] To attain this kind of materials, high dielectric constant ceramic fillers like bismuth oxide (Bi_2O_3) loaded into polymers like polystyrene (PS) and polyvinylidene fluoride (PVDF) because of their extraordinary thermal, chemical, dielectric, pyroelectric, mechanical and piezoelectric properties. PS is naturally transparent, thermoplastic, waterproof, dimensionally stable, rigid chemical inert, relatively cheaper, versatile, good dielectric strength and easy to fabricate. [8] PVDF has incredible properties such as excellent mechanical strength, light weight, heat, radiation and weather resistant, recyclable and easy to process. The polarity of alternating CH_2 and CH_2 groups present in PVDF $(C_2H_2F_2)_n$ chains are responsible for its amazing electrical properties. [9] [10] The dielectric constant of pristine PS and PVDF is 2.5 and 10 respectively at room temperature and at KHz frequencies. [11] [12] [13] Dielectric strength of PS and PVDF is 19.7 MV/m and 420 MV/m respectively. [14] [15] Bi_2O_3 is a metal oxide suitable as filler due to its high dielectric constant as well as good photoelectric, mechanical and electrical properties. The α-Bi_2O_3 phase is most stable and commonly used in ceramic applications [16]. Permittivity of the ceramic/polymer composites increases with filler concentration, however decreases in breakdown strength and mechanical properties which limits high concentration of filler. [17] [18] [19] [20] In present work, a comparative study on structure, surface morphology, bonding and dielectric response has been executed for BO/PS and BO/PVDF composites with varying filler concentration to optimize the best concentration of loading where high dielectric constant is achieved without losing the other responses.

2. Experimental

2.1. Materials

Bismuth oxide (Bi_2O_3 99.99% purity), polystyrene (PS) granules purchased from Sigma Aldrich, polyvinylidene fluoride (PVDF 99.99%) & solvents like dichloromethane, NMP and benzene were purchased from Alfa Aesar respectively.

2.2. Synthesis of Composites

The BO/PS and BO/PVDF composites with varying concentration of BO = 1 to 5 wt% were synthesized by solution casting method. In this method, filler and po-

lymers were individually dispersed in crystallizable benzene using ultrasonicator for one hour. Then both the solutions were mixed and further sonicated till homogeneously dispersion. This mixed solution was poured in the Petri dish floating on mercury surface to get composites of uniform thickness of 100 μm.

2.3. Characterization

X-ray diffractometer (Bruker AXS, ApexII) in the angle (2θ) range 20˚ to 60˚ with step resolution 0.2˚ was used to investigate the structure of the composites. FT-IR spectra of all synthesized composites were collected by using Alpha Bruker FT-IR Spectrometer (ECO-ATR) in wave number range 4000 cm^{-1} to 500 cm^{-1} at room temperature. Scanning electron microscope JSM-7610F Plus, JEOL Ltd Tokyo, was used for surface morphology and energy dispersive x-ray (EDX) spectra for elemental analysis of the fabricated composites. Impedance analyzer WAYNE KERR Electronics, London, UK (6500B series) was used for dielectric response measurements in the frequency range 1 kHz to 3.16 MHz.

3. Results and Discussion

3.1. X-Ray Diffraction

Figure 1 show XRD spectra of pristine PS, PVDF, BO, $BO_{1\%}PS_{99\%}$, $BO_{5\%}PS_{95\%}$, $BO_{1\%}PVDF_{99\%}$ and $BO_{5\%}PS_{95\%}$ composites. The average crystallite size of the order of 50 nm for Bi_2O_3 has been found by Scherer equation.

The diffraction spectrum of bismuth oxide shows peaks at angles 27.3˚, 33.1˚, 46.4˚ and 54.8˚ corresponds to planes (120), (121), (311) and (241) respectively

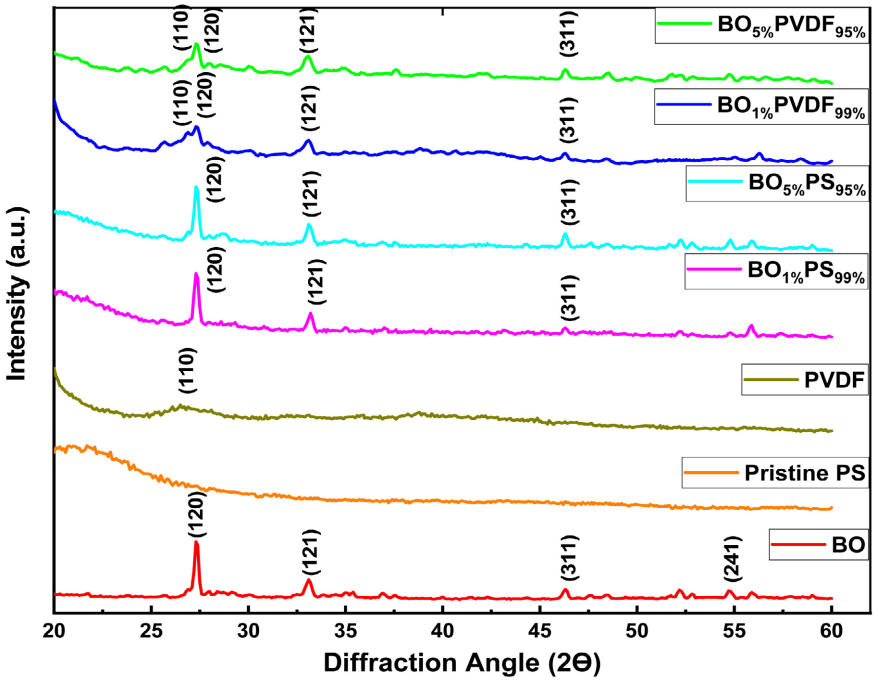


Figure 1. XRD spectra for (a) BO, (b) pristine PS, (c) PVDF, (d) $BO_{1\%}PS_{99\%}$, (e) $BO_{5\%}PS_{95\%}$, (f) $BO_{1\%}PVDF_{99\%}$ and (g) $BO_{5\%}PVDF_{95\%}$. Composites.

matches well with JCPDS file (76 - 1730) belongs to monoclinic phase of Bi_2O_3 [21] [22]. XRD spectrum of pristine PS showed only a hump reflecting amorphous nature of polystyrene. [23] However diffraction peaks at $2\theta = 26.4^{\circ}$, 34˚ and 46˚ are obtained for PVDF, [24] showing the crystalline nature of the polymer. The position of diffraction peaks for composites like $BO_{1\%}PS_{99\%}$, $BO_{5\%}PS_{95\%}$, $BO_{1\%}PVDF_{99\%}$ and $BO_{5\%}PS_{95\%}$ composites remains the same as those of individual only intensities at higher concentration of filler found to be increased indicating the phases of dispersed bismuth oxide.

3.2. SEM and EDX Analysis

Figures 2(a)-(i) show SEM images of pristine PS, PVDF, Bi_2O_3, BO/PS and BO/PVDF composites at different magnifications. The surface morphology of pristine PS as shown in **Figure 2(a)** looks like colloidal structure, however SEM images of PVDF **Figure 2(b)** and **Figure 2(c)** appearing like spherical ball shape connected to each other. The SEM micrographs as shown in **Figure 2(d)** and **Figure 2(e)** appearing like cylindrical needle shape of bismuth oxide (Bi_2O_3) match well with previous reports. [25] [26] The porous structure formation in BO/PS composites as shown in **Figure 2(f)** and **Figure 2(g)**, may help to trap

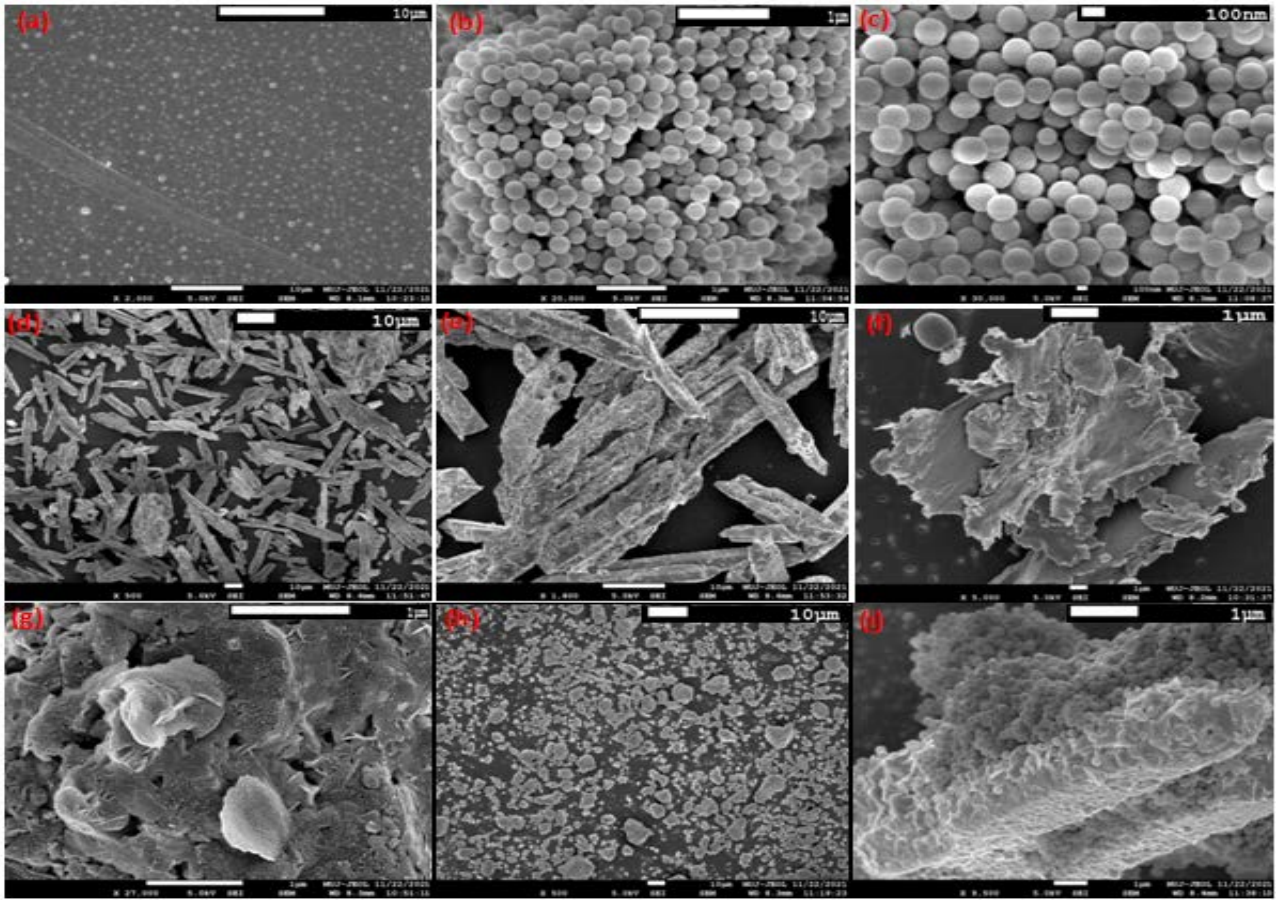


Figure 2. SEM images for (a) pristine PS ((b) & (c)) PVDF ((d) and (e)), Bi_2O_3 ((f) and (g)), BO/PS ((h) and (i)) BO/PVDF composites.

charge carriers during charge discharge process. The rods of BO surrounded by PVDF as shown in **Figure 2(h)** and **Figure 2(i)** represents a significant interaction between filler and polymer which has been also obtained by bonding characteristics as confirmed by FT-IR measurements.

Figure 3(a) and **Figure 3(b)** represents energy dispersive x-ray spectra of BO/PS & BO/PVDF composites, confirms presence of bismuth, oxygen, and carbon in the fabricated composites of PS and PVDF. [27]

3.3. FT-IR Spectroscopy

Figure 4(a) and **Figure 4(b)** show FT-IR spectra for pristine BO, PS, BO/PVDF composites and BO, PS, BO/PS composites. As shown in **Figure 4**, peaks for BO are observed at 563 cm^{-1}, 604 cm^{-1}, 1535 cm^{-1}, 1685 cm^{-1} and around 3600 cm^{-1} corresponds to Bi-O stretch, Bi-O-Bi (metal-oxygen-metal) stretching vibrations, NO_3^- group and O-H (hydroxyl group) stretching respectively. These peaks are reference of monoclinic structure of Bi_2O_3, and matches well with earlier reports [28]. As shown in **Figure 4(a)**, absorption peaks at 604 cm^{-1}, 760 cm^{-1} (CF_2 bending), 871 cm^{-1} (C-C-C), 1173 cm^{-1} (CF_2 stretch), 1395 cm^{-1} (CH_2 wagging) and 1685 cm^{-1} (C = C stretching vibration) are characteristic peaks of α-PVDF. Appearance of absorption peaks at the same wave number in composites and individual is evidence of well orientation and bonding of filler with PVDF. FT-IR spectrum of pristine PS as shown in **Figure 4(b)** showing various peaks at wavenumbers like 684 cm^{-1}, 754 cm^{-1}, 1494 cm^{-1}, and 3026 cm^{-1} related to C-H bending, aromatic C = C stretching and aromatic C-H stretching vibrations and characteristic peak of styrene [29].

3.4. Dielectric Spectroscopy

Figure 5(a) and **Figure 5(b)** show frequency vs dielectric constant of pristine PVDF as well as BO/PVDF composites and PS as well as BO/PS composites. The dielectric constant of pristine PS is obtained 3.687 at freq. of 1 kHz and found to be increased for $BO_{5\%}PS_{95\%}$. up to 5.246, similarly, dielectric constant of PVDF is obtained 9.754 and found to be increased for $BO_{5\%}PVDF_{95\%}$ upto 17.449 attributes

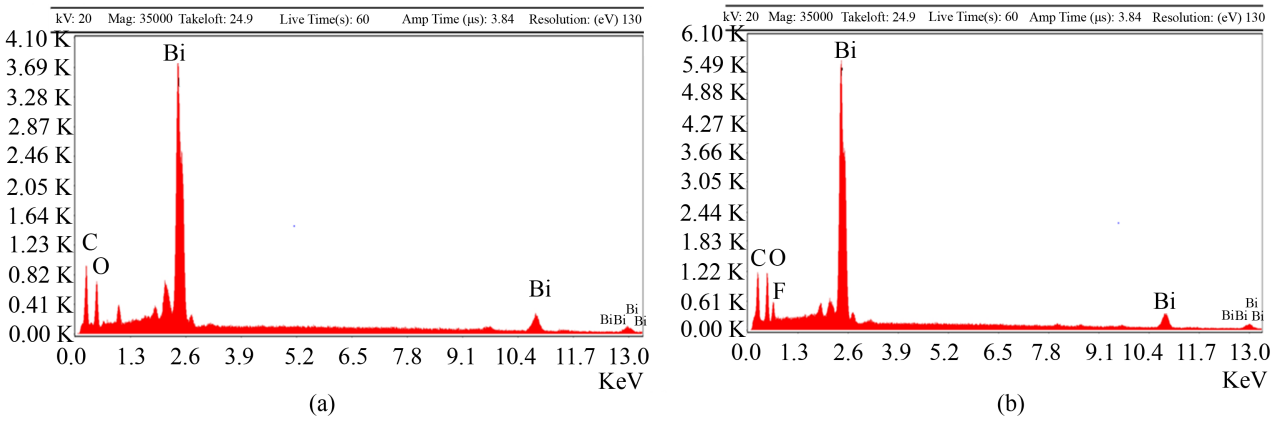
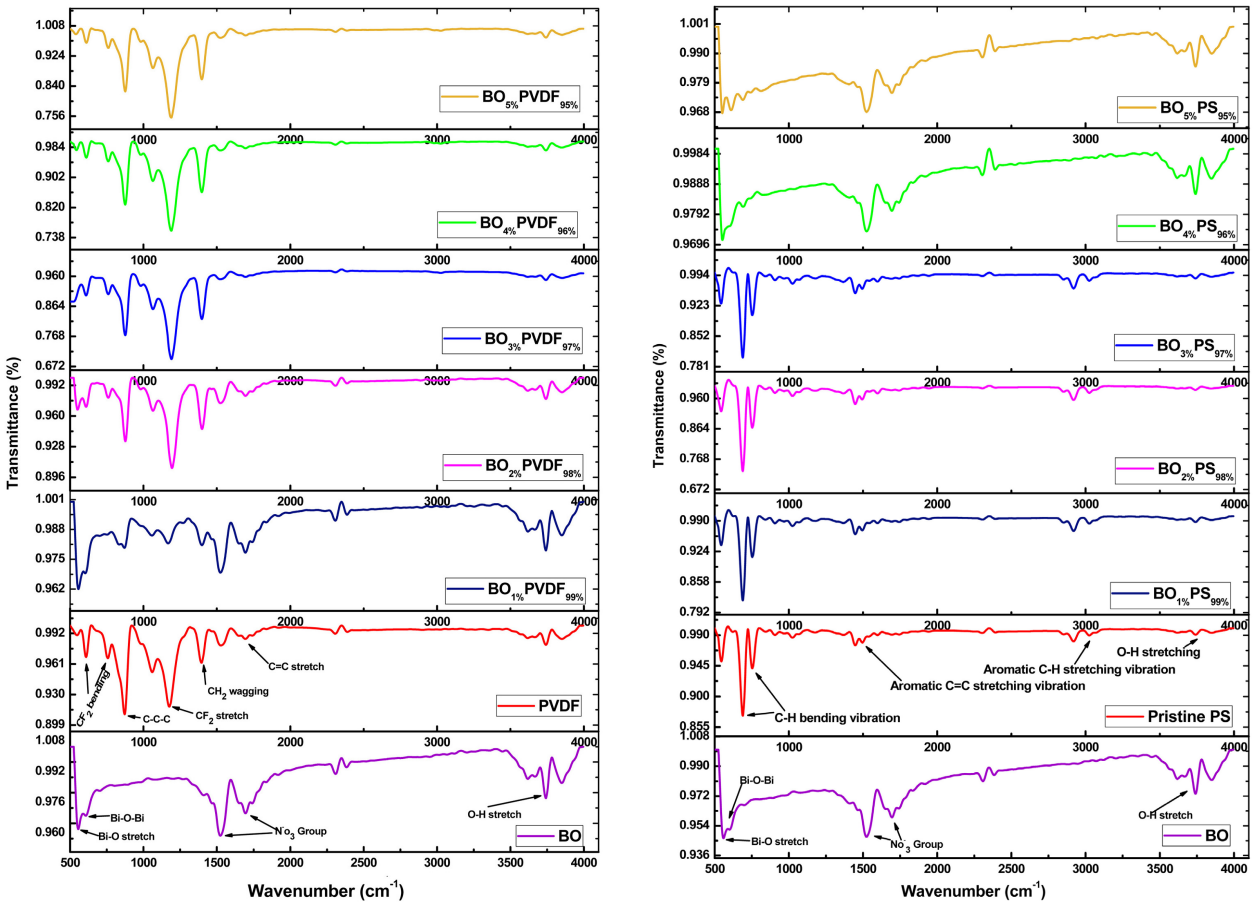


Figure 3. EDX spectra for (a) BO-PS and (b) BO-PVDF composites.



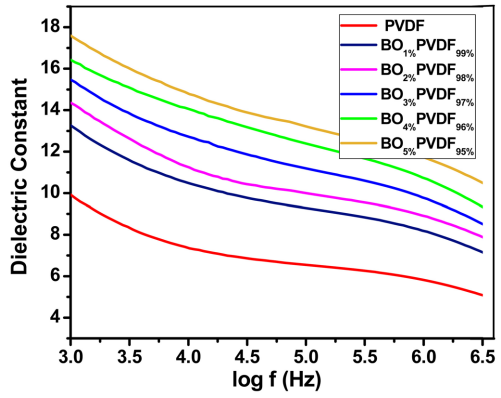
(a) (b)

Figure 4. FT-IR spectra for (a) BO, PVDF & BO/PVDF composites; (b) BO, pristine PS & BO/PS composites.

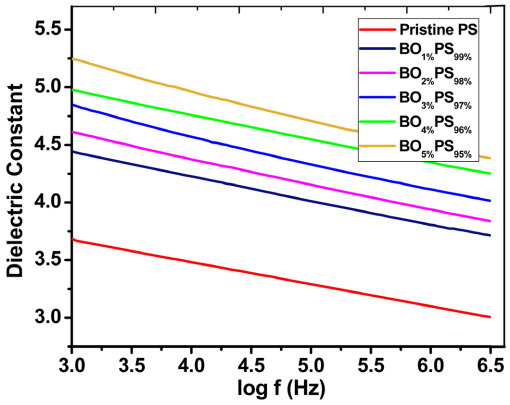
the accumulation of charge at grain boundaries leading to the formation of intrinsic dipoles in composites of BO/PS and BO/PVDF results dielectric constant approx. equivalent to the BO. Thus, on increasing BO concentration within the polymers, there might be maximum possibility of increase in intrinsic dipoles and polarization area of the composites, which results as enhancement in dielectric constant. However, values of dielectric constant decreases with increase of frequency attributes all type of polarization (interfacial, dipolar, vibrational and electronic polarization) contributes in permittivity, but dipoles are unable to flip with field when frequency is increased causing the polarization and dielectric constant to decrease with increasing frequency. [30]

Figure 6(a) and **Figure 6(b)** show dissipation factor (tanδ) vs frequency response of pristine PVDF, BO/PVDF composites as well as PS, and BO/PS composites respectively. It indicates that tanδ increases with increasing BO concentration in both the polymers PS and PVDF. Dissipation factor attains large values for $BO_{5\%}PS_{95\%}$ and $BO_{5\%}PVDF_{95\%}$ composites. As shown in **Figure 6(b)**, PS and BO/PS composites have large tanδ values at smaller frequencies and decreases with frequency up to 500 kHz, after this it increases again with increasing frequency.

However, for PVDF and BO/PVDF composites tanδ decreases with frequency up to 100 kHz and then increases rapidly with increasing frequency, as shown in Figure 6(a). The dielectric constant of $BO_{5\%}PS_{95\%}$ and $BO_{5\%}PVDF_{95\%}$ composites increased 1.4 and 1.8 times as compared to pristine PS and PVDF which is very close to the reports available in the literature [31]. Thus, filler BO improves dielectric performance of composites but dissipation factor limits its concentration. Table 1 showed values of dielectric constant with log f for pristine PS & PVDF as well as their various composites.



(a)

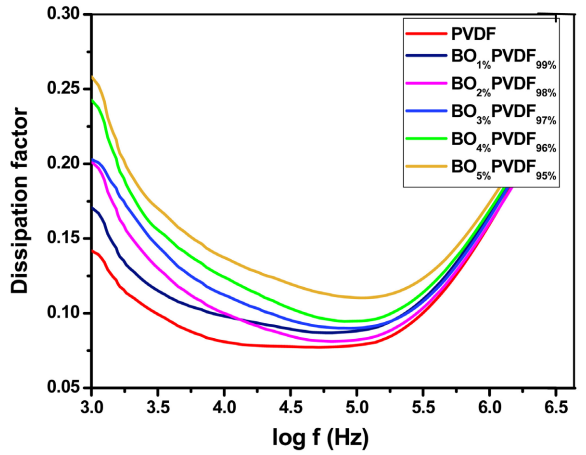


(b)

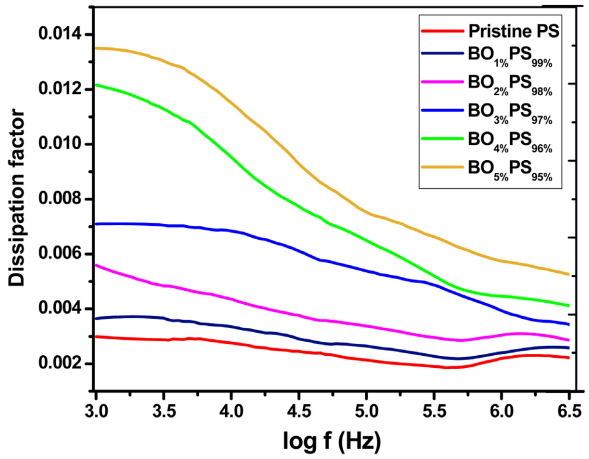
Figure 5. Dielectric constant vs frequency response of (a) pristine PVDF & BO/PVDF composites; (b) pristine PS & BO/PS composites.

Table 1. Values of dielectric constant with log f for pristine PS & PVDF as well as their various composites.

S. No.	log f (Hz)	Dielectric Constant							
		Pristine PS	$BO_{1\%}PS_{99\%}$	$BO_{3\%}PS_{97\%}$	$BO_{5\%}PS_{95\%}$	PVDF	$BO_{1\%}PVDF_{99\%}$	$BO_{3\%}PVDF_{97\%}$	$BO_{5\%}PVDF_{95\%}$
1	3.0000	3.687	4.445	4.851	5.246	9.754	13.118	15.347	17.449
2	4.0101	3.478	4.225	4.567	4.961	7.348	10.469	12.703	14.772
3	5.0202	3.288	4.007	4.326	4.703	6.539	9.265	11.168	13.188
4	6.0303	3.094	3.801	4.108	4.476	5.785	8.140	9.715	11.663
5	6.5000	3.001	3.710	4.012	4.380	5.062	7.120	8.476	10.456



(a)



(b)

Figure 6. Dissipation factor vs frequency response of (a) pristine PVDF & BO/PVDF composites (b) PS & BO/PS composites.

4. Conclusion

In summary, the average crystallite size of bismuth oxide has been found approx. 50 nm which was used as filler in the PS and PVDF matrix. The XRD spectra of bismuth oxide and PVDF are consistent to monoclinic crystal structure, however spectrum for PS indicated its amorphous nature. SEM micrographs showed well orientation of BO in PS and PVDF composites however FT-IR spectra reveal good interaction between filler and polymers in composites. The dielectric constant of $BO_{5\%}PS_{95\%}$ and $BO_{5\%}PVDF_{95\%}$ composites increased 1.4 and 1.8 times to that of pristine PS and PVDF respectively and attributes the accumulation of charge at grain boundaries leading to the formation of intrinsic dipoles in composites of BO/PS and BO/PVDF. These types of enhanced dielectric constant composites are useful in flexible energy storage devices.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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