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Thermoplastic Conducting Composites through Facile Melt Mixing: Ideal Material for Flexible Electronics

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The study presents the fabrication of flexible conducting composites of high-density polyethylene (HDPE) and polythiophene (PTH) through a facile melt mixing process and their systematic characterization. A continuous PTH network with limited interfacial adhesion was formed within the HDPE matrix resulting in a composite with improved modulus and lower tensile strength. Thermogravimetric analysis demonstrated enhanced thermal stability, as a function of PTH content. Electrical conductivity exhibited an exponential increase beyond a percolation threshold of 20 phr. The composites exhibited a substantial increase in dielectric constant, indicating their suitability for supercapacitor applications. The composites achieved EMI shielding effectiveness of up to 31 dB in the X-band (8.2–12.4 GHz), predominantly through absorption mechanisms, highlighting their potential as effective flexible EMI shielding materials as well. These results underscore the utility of HDPE/PTH composites in advanced applications requiring flexible, thermally stable, and electrically conductive materials.

Keywords: High-density polyethylene (HDPE), Polythiophene (PTH), Flexible conducting composites, Melt blending, Thermal stability, Dielectric properties, Electromagnetic interference (EMI) shielding, Supercapacitors.

1. Introduction

The development of flexible and electrically active thermoplastic composites has gained substantial research interest, particularly for applications in electronics, sensing, and electromagnetic shielding. One of important parameter that motivates the preparation of thermoplastic conducting composites is the possibility of imparting flexibility to the resulting composites by appropriate selection of the matrix polymer. In flexible electronics and wearable sensors, the deformable nature of composites enables conformal fitting to various surfaces. Sensors and actuators fabricated using conducting composites utilize the flexibility of the composites and the change in conductivity on applying a deformation or the change in dimension on the passage of current. If the flexibility factor can be derived from the use of a general purpose commercial thermoplastic, it will be all the more desirable in terms of economic considerations. On account of all these factors high density polyethylene (HDPE) was selected as the matrix material for the current study. There have been studies on HDPE matrix to develop electrically active composites. Nurazreena et al. studied the effect of metal powders such as aluminium, copper and iron on electrical and mechanical properties of HDPE [1]. Composites with electrical and thermal conductivity were prepared by Krupa et al., through incorporating nickel powder in HDPE [2]. Mechanical and electrical properties of carbon black filled polyethylene composites were investigated by Qiang Yuan et al. [3] Polyethylene conducting composites prepared by Alvarez et al. showed significant electrical conductivity on 10% volume loading of submicron sized copper [4]. Graphene when incorporated in HDPE reduced resistivity and increased modulus [5]. Hybrid effect of carbon black and carbon fiber on polyethylene was investigated by Shen et al. [6] Conductivity and EMI shielding effect imparted by carbon nanotubes to polyethylene has been studied [7]. Intrinsically conducting polymers have also been used to impart electrical conductivity to polyethylene. Polyaniline has been blended with polyethylene in different concentrations to arrive at an expression for the effect of conducting particles on electrical and mechanical properties of the blend [8].

Polythiophene (PTH) is a prominent conducting polymer renowned for its electrical conductivity, which can be systematically tuned by doping to exhibit conductivity varying from insulating to metallic range. Moreover, PTH exhibits exceptional environmental and thermal stability, rendering it a material of considerable interest for a diverse array of advanced applications. These include electrostatic coatings, sensors, light-emitting diodes, photovoltaic cells, rechargeable batteries, electrochromic smart windows, DNA detection systems, electromagnetic interference shields and supercapacitors. [9–17] Composites of polythiophene with different matrices have been made to exploit its electrical properties. [18–21]

In this work polythiophene has been used as the conducting element in HDPE. The effect of polythiophene on electrical, mechanical and thermal properties of the composites is systematically analysed. The dielectric properties of the prepared composites has also been investigated to find their suitability as capacitor materials.

2 Experimental

2.1 Materials

Thiophene, ferric chloride, dichloromethane and acetonitrile were used in this study

2.2 Preparation of composites

Table 1 Formulation of PE/PTH composites

Ingredient	Quantity, phr*				
PE	100	100	100	100	100
PTH	0	10	20	30	40

* parts per hundred rubber

Mixing was done in a Brabender Plastograph at 145 °C. When PE was softened PTH powder was added slowly and the mixing was continued for another 5 min. The mix was taken out from the mixing chamber and pressed between aluminium platens. The pressed sheets were chipped into small pieces and used for compression moulding. Moulding was done in a hydraulic press at a temperature of 150 °C under 200 MPa pressure.

3 Results and discussion

3.1 Mechanical properties

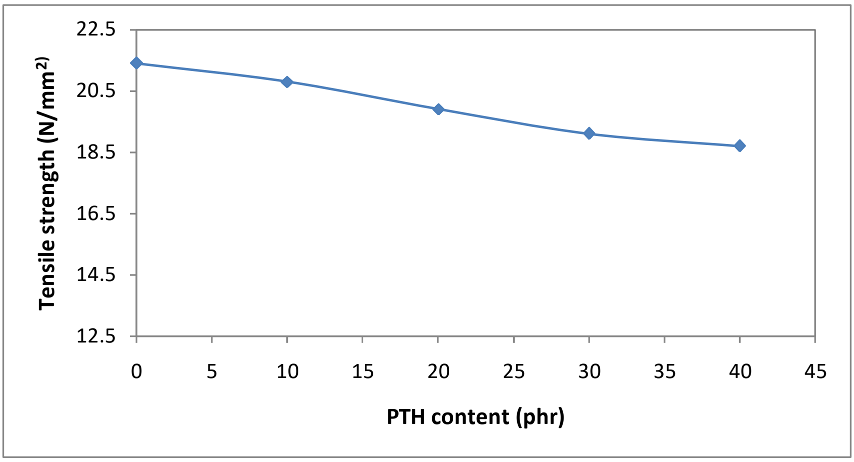

Fig. 1 Variation of tensile strength with PTH content in PTH/PE composites

The effect of PTH loading on tensile strength of PTH/PE composites is shown in Fig. 2. Tensile strength is found to be decreasing with the incorporation of PTH. HDPE being nonpolar is inherently incompatible with the polar PTH. So the interaction between the PE and PTH phases at the interface will be lower. The stress applied on the polymer matrix is to be transferred efficiently to the filler through the interface and partially born by the filler if it is to strengthen the composite. The low interfacial adhesion between PE and PTH affects the stress transfer and results in stress concentration at the interface. This will lead to crack formation and crack growth through the interface on the application of tensile forces eventually leading to premature failure and low tensile strength. With increase in PTH content, the interfaces, that is weak spots prone to easy failure, also increase further decreasing the tensile strength.

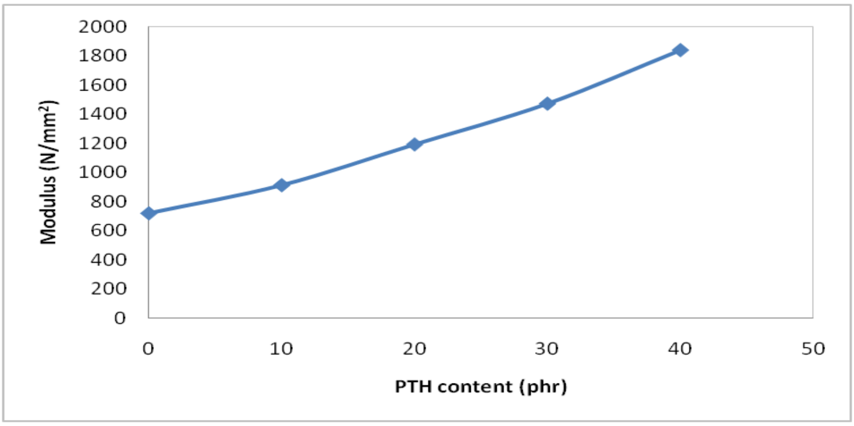

Fig. 2 Variation of modulus with PTH content in PTH/PE composites

Variation of tensile strength with PTH content in PTH/PE composites is depicted in Fig. 3. Unlike the behaviour of tensile strength, modulus increases with PTH content. The PTH particles are distributed uniformly and form a network by the association, as seen in the SEM. Modulus is measured at low strains where strain varies linearly with stress. The network of PTH particles is able to keep itself intact at these low strains and impart rigidity to the composites which translates to higher modulus. The voids and cracks at the interface leading to premature failure appear only at higher strains, surpassing the interfacial adhesion. At higher PTH content the network is strengthened and becomes more rigid which appears as an increase in the modulus of the composites.

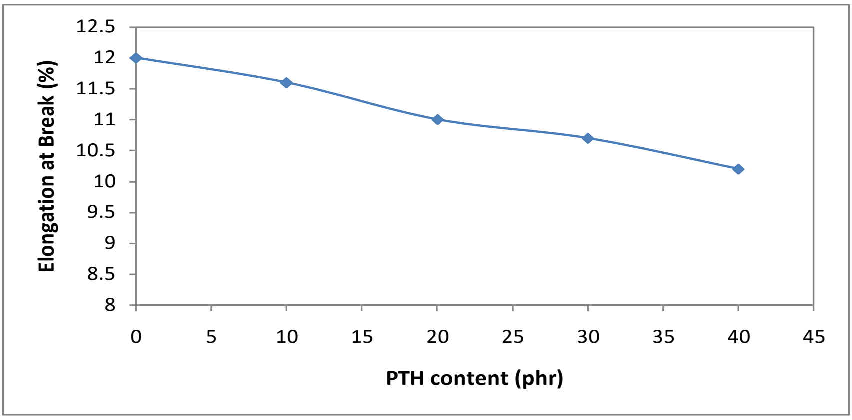

Fig. 3 Variation of elongation at break with PTH content in PTH/PE composites

Influence that the loading of PTH exerts on the elongation at break of the composites is illustrated in Fig. 4. Elongation at break decreases with increasing PTH content. This may be due to the low interaction between HDPE matrix and PTH particles. The low interfacial adhesion between the particles and the matrix may be easily overcome by the applied forces during a tensile test. This leads to the breaking up of contact between the two components resulting in the generation of voids at the interface. These voids will act as stress concentration points eventually ending up as growing cracks. Some of these cracks may grow up to the full width of the sample cross section or may intersect with other similarly propagating cracks so that the combination be spread over the entire width. The resulting premature failure of the composite reduce the elongation at break from that of the pure sample. Increasing the PTH content will result in an increase of the extent of weak interfacial area and subsequently bring about an increase in the possibility of void and crack formation. This escalates the possibility of premature failure further reducing the elongation at break of the composites.

3.2 Morphology studies

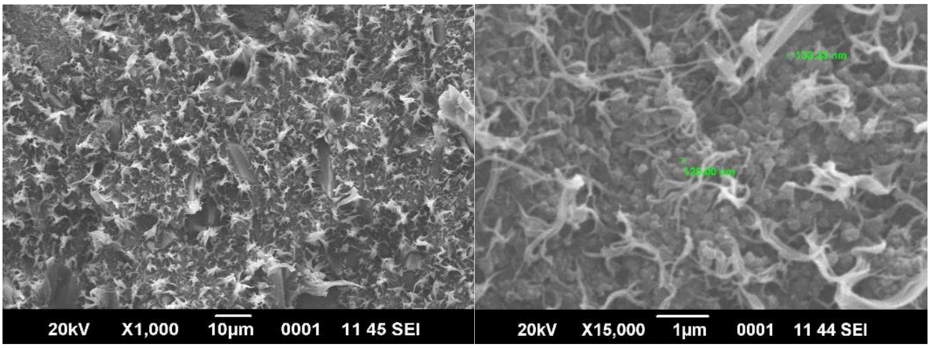

Fig. 4 SEM images of tensile fracture surface of PTH/PE composites

Scanning electron microscopy images of tensile fracture surface PE composites with 40 phr of PTH are shown in Figs. 4a and 4b, where a and b show images of different magnification. The fractured surface shows fibril-like structures formed due to the elongation of polyethylene matrix under the applied tensile force. Due to the nonpolar-polar incompatibility between HDPE and PTH and the ensuing low interfacial interaction, the PTH particles do not impart any reinforcement to the composite. Efficient stress transfer to the PTH particles does not take place resulting in stress concentration and fracture through the interface leading to easier pullout of the PTH particles from the matrix. Only the parts where the matrix (HDPE) continuity is retained through the distributed network of PTH particles, resist the applied forces and elongate giving fibrillar morphology. PTH particles are seen in between these elongated features throughout the matrix. Since the effective area that resists the tensile forces is limited due to the incorporation of PTH, tensile strength of the composites is bound to reduce.

3.3 Thermal studies

Thermogravimetric curves of HDPE and PTH/HDPE composites with varying concentration of PTH are given in Fig. 5. As in the case of PTH/PP composites here also incorporation of PTH increases the thermal stability of HDPE. A more accurate information on the increase in thermal stability can be obtained from the derivative thermogravimetry (Fig. 6). Temperature corresponding to the maximum rate of degradation, expressed by the peak in the derivative curve, shows an increase of more than 15 °C when the PTH content is 10 phr, compared to that of pure HDPE. The increase becomes 20 °C on doubling the PTH content. PTH particles distributed within the HDPE matrix form a continuous network which influence the thermal degradation behaviour of the composites. Compared to pure HDPE, the main chain degradation of PTH starts at a higher temperature. The continuous network of thermally more resistant PTH particles protect the matrix against degradation and delays the weight loss. With the increase in PTH content the thermal protection becomes more ef 0 PTH In pure HDPE the degradation is almost complete with negligible residue. As the P 10 PTH tent increases the residue also increases proportionally. The residue in the case of pur 20 PTH is 40%. Since the PTH was incorporated in phr wise (parts per 100), it should leave around .6% of residue in the case of 10 phr composites. But the fact that almost double the am 30 PTH residue is present indicates that the parts of HDPE matrix embedded inside the PTH r 40 PTH remain protected from complete degradation.

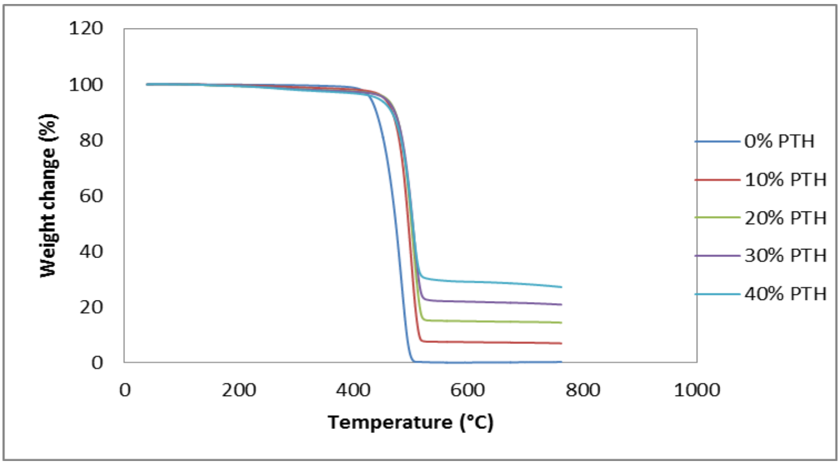

Fig. 5 Thermogravimetric curves of HDPE and PTH/ HDPE composites

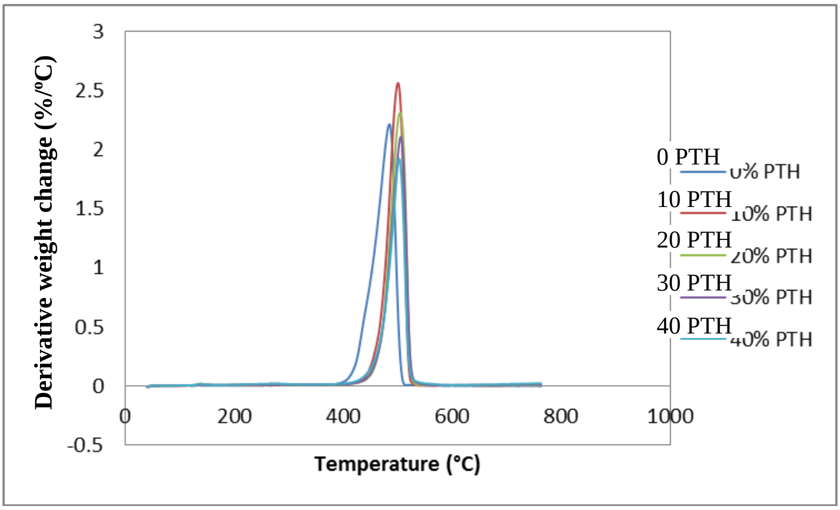

Fig. 6 Derivative thermogravimetric curves of HDPE and PTH/ HDPE composites

3.4 Conductivity studies

Variation of DC conductivity of PTH/HDPE composites with respect to PTH content is shown in Fig. 7. Conductivity shows an exponential increase beyond a loading of 20 phr. Compared to PTH/PP composites conductivity of PTH/PE composites was found to be 4 orders of magnitude higher. This may be due to the higher crystallinity of HDPE compared to PP. Conducting particles incorporated in the matrix will preferably be distributed in the amorphous phase than crystalline phase. By the exclusion of crystalline phase from the distribution it is easier to establish a conducting network with the same concentration of conducting species. Similar result has been reported earlier [22] where a 40% increase in crystallinity produced a 3 order increase in conductivity. HDPE composites are found to attain percolation threshold at a lower PTH content compared to PP, beyond which there is a remarkable increase in conductivity. This is attributed to the concentration of PTH particles predominantly in the amorphous region increasing the efficiency of the continuous conducting network for the same amount of PTH. With increase in PTH content, new conducting pathways are formed preferably within the same restricted region which gives a large increase in conductivity.

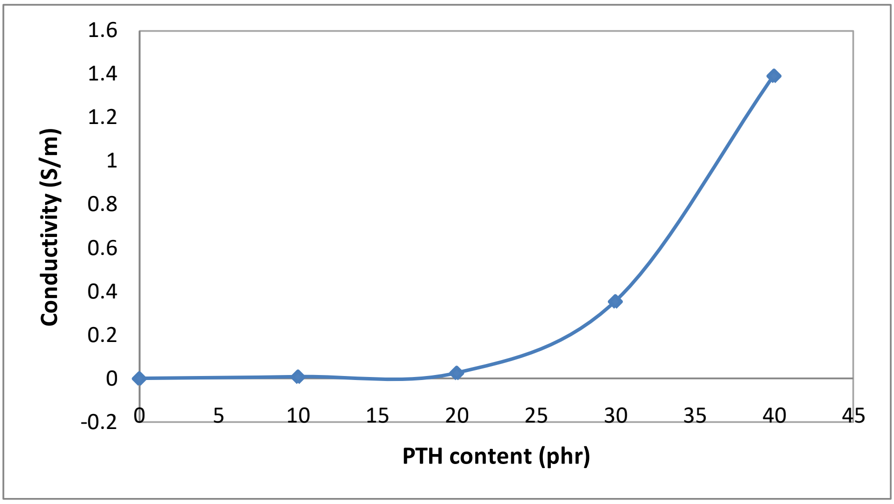

Fig. 7 Variation of conductivity with PTH content of PTH/HDPE composites

3.5 Dynamic mechanical properties

Variation of storage modulus with frequency of PTH/PE composites in dynamic mechanical analysis is given in Fig. 8. Storage modulus increases with both frequency and PTH content. Increase of frequency in dynamic mechanical analysis is equivalent to decrease of temperature. Correspondingly lower frequencies emulate higher temperatures where the polymer goes through a transformation from a glassy solid to a rubbery state. Distribution of PTH in the HDPE matrix forms a continuous network which gives the additional rigidity to the matrix in proportional to the PTH content. At higher temperatures increased molecular motions may disturb the PTH-matrix interfacial interaction and cause detachment of the matrix from the PTH particles. With matrix no longer supporting the PTH particle network the dynamic forces applied during the analysis will be able to breakdown the network and decrease the modulus. This behaviour is mimicked at lower frequency range as low modulii of the composites.

Variation of storage modulus at a fixed frequency of 1 Hz with PTH content is plotted in Fig. 9. Storage modulus increases almost linearly with PTH content of composites. An improvement of 57% can be attained for 40 phr composites compared to pure PE.

Loss modulus variation with frequency is illustrated in Fig. 9. Loss modulus shows an inverse trend with frequency compared to the trend of storage modulus. Loss modulus, which is a measure of energy dissipated, decreases with frequency. The transition region corresponding to high temperatures or low frequencies is visible in the case of loss modulus also, though with an inverted shape. Pure HDPE shows a large change in loss modulus with frequency, corresponding to the increase in flexibility with temperature. At the transition region more energy is absorbed for the increased mobility of molecular chains making the polymer more flexible. This materialize as an increase in loss modulus. As frequency can be considered as an inverse of temperature in dynamic mechanical analysis a decrease of loss modulus can be observed. In the composites due to the rigid network of PTH particles the mobility of the molecular chains is restricted decreasing the change in loss modulus with frequency. As the PTH content increases the interfacial area between PTH particles and HDPE matrix also increases. As there is low interaction between PTH and HDPE there will be energy dissipation at the interface. So with higher PTH content there will be more energy dissipation ensuing higher loss modulus.

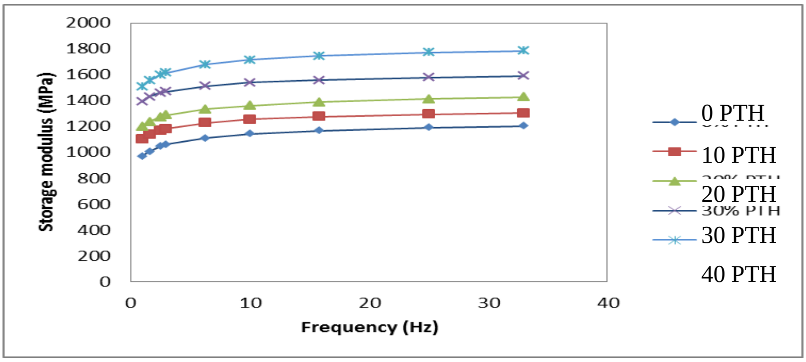

Fig. 8 Variation of storage modulus of PTH/HDPE composites with frequency

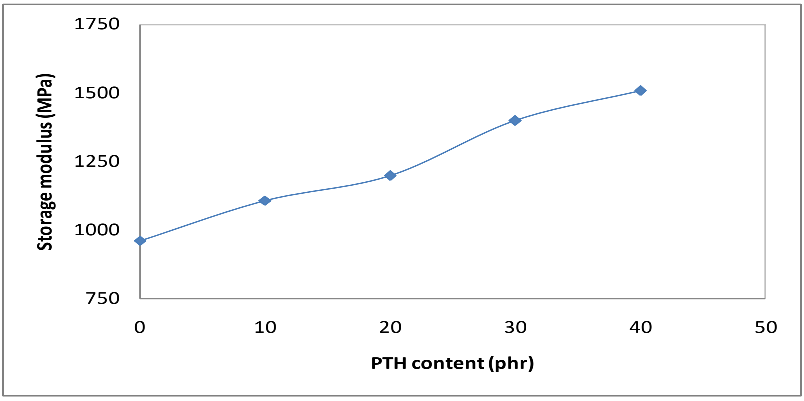

Fig. 9 Variation of storage modulus @ 1 Hz of PTH/HDPE composites with PTH content

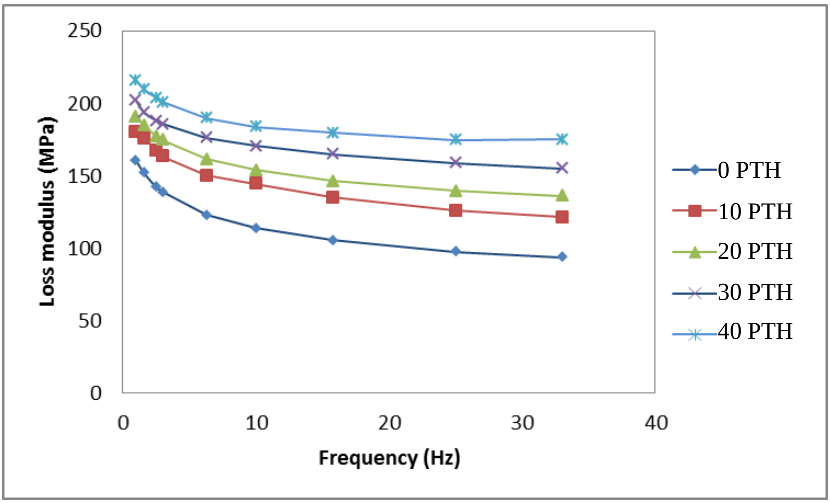

Fig. 10 Variation of loss modulus of PTH/HDPE composites with frequency

3.6 Dielectric properties

Dielectric properties of the composites were evaluated with respect to PTH content and frequency. Variation of dielectric constant with frequency is plotted in Fig. 11 and that of dielectric loss in Fig. 12. Pure PE shows very low dielectric constant being deprived of any dipoles or heterogenity in the material and thereby lacking in dipolar or interfacial polarization. With the introduction of PTH particles a large increase in dielectric constant is observed which increases with PTH content. For 30 and 40 phr composites 5 orders of magnitude increase in values is observed. On incorporation of PTH, dipoles are also introduced giving rise to dipolar polarization. Charge accumulation in the interface between conducting PTH and nonconducting PE matrix results in interfacial polarization. PTH particles being preferentially dispersed in the amorphous phase of the matrix the conducting network possess very high conductivity whereas the crystalline phase remain comparatively devoid of conducting particles. Thus a huge difference in conductivity is created between the phases which gives rise to very large interfacial polarization. This together with dipolar polarization is responsible for the very high dielectric constant of composites. At lower frequencies dielectric constant decreases with increase in frequency. Dipoles change orientation in tandem with alternating electric field. When frequency increases dipoles may not be able to keep up with electric field resulting in a lag in the orientation and resulting polarization leading to a decrease in dielectric constant.

Dielectric loss also shows a similar behaviour as that of dielectric constant with respect to PTH content and frequency. The dipoles orienting with respect to the electric field generate heat by friction between the surrounding molecules. This energy loss as heat is responsible for the dielectric loss in the material. With increase in PTH content, number of dipoles and hence dielectric loss increases. With increase in frequency, the lagging behind in orientation of the dipoles lead to decrease in dielectric loss. Apart from the frequency factor dielectric loss is also depended on DC conductivity of the material. Higher conductivity give rise to higher dielectric loss. Here the high values of loss, especially at higher loadings, can be attributed to the very high DC conductivity of composites.

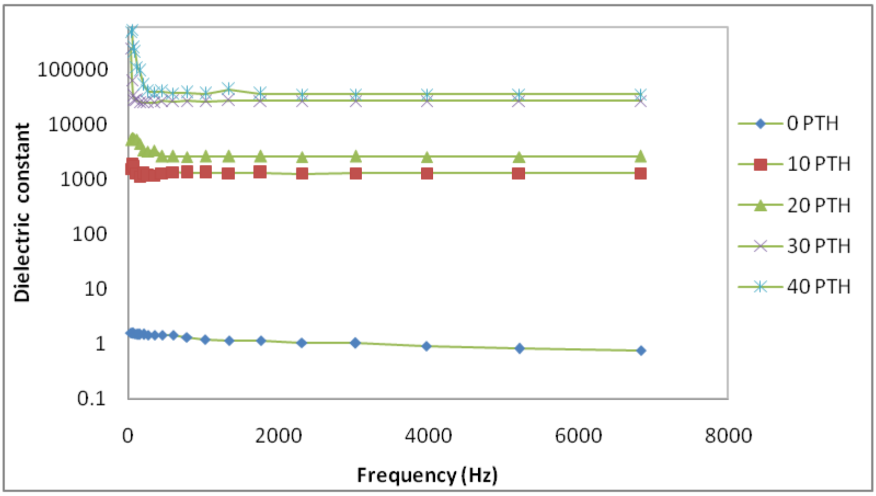

Fig. 11 Variation of dielectric constant of PTH/PE composites with frequency

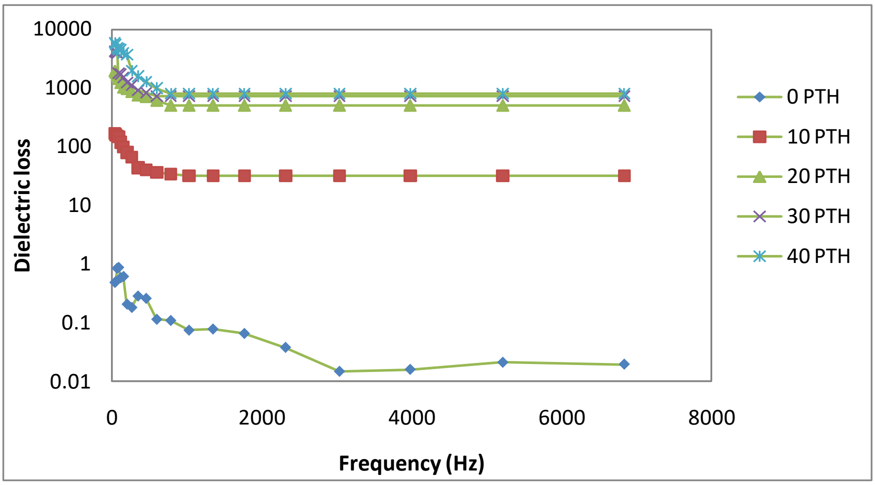

Fig. 12 Variation of dielectric loss of PTH/PE composites with frequency

3.7 Electromagnetic interference shielding

Electromagnetic shielding efficiency of PTH/PE composites is illustrated in Fig, 13. A significant improvement in shielding efficiency could be achieved for PTH/PE composites compared to PTH/PP composites, maximum being 31 dB at a frequency of 8.6 GHz for 40 phr composites. A minimum shielding of 25 dB could be maintained throughout the X band region making the composites suitable materials for commercial applications. Fig. 14, which shows the shielding effectiveness due to absorption, confirms that the mechanism of shielding is almost exclusively absorption loss, reflection loss rather being negligible. This qualifies the composites to be microwave absorbers used in stealth technology which makes aircrafts, missiles, ships and submarines invisible to the radar.

Transmission coefficient of the shielding material is given by the equation,

$$T = e^{-\gamma d} \text{ .... } (1)$$

Where 'γ' is the propagation constant and 'd' is the thickness. [23]

$$\gamma = \gamma_0\sqrt{\varepsilon_r \mu_r} \text{ .... } (2)$$

Where 'γ0' is the propagation constant for free space, ε_r is the relative permittivity and μ_r is the relative permeability. [24]

It is imperative that increased relative permittivity (dielectric constant) of the material will decrease the transmission coefficient. PTH/PE composites possess exceptionally high dielectric constant as a discussed in section which translates into low transmission through the composites. Apart from this the composites is endowed with high DC conductivity which can be credited to the preferential distribution of conducting particles in the amorphous part of the PE matrix. This increases the shielding efficiency due to absorption (SEA) of the material. The combined effect of both these parameters materializes into high shielding efficiency of the composites. With increase in PTH concentration conductance loss and dielectric loss increases rendering the composites with enhanced shielding efficiency.

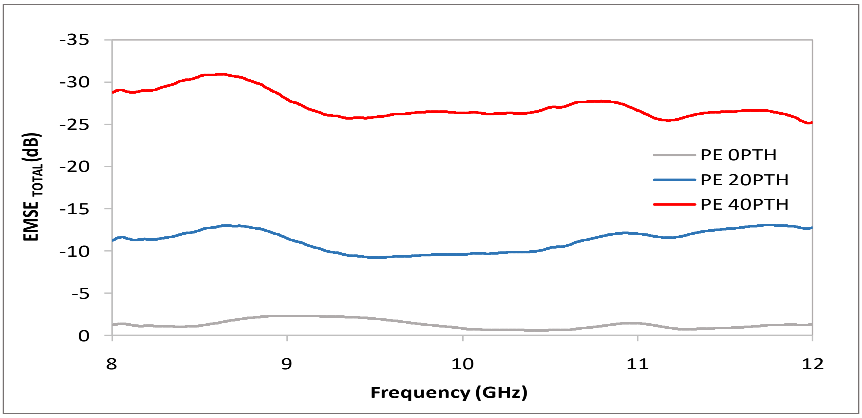

Fig. 13 Total EMSE of PTH/PE composites in X band

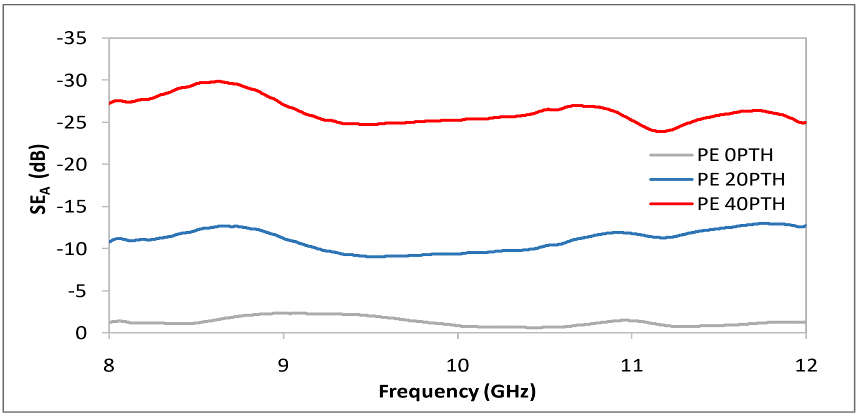

Fig. 14 EMSE due to absorption of PTH/PE composites in X band

4 Conclusions

Conducting composites with increased flexibility could be produced through incorporation of polythiophene in a commercial general purpose thermoplastic polymer, high density polyethylene. Surface morphology reveals low interaction arising from polar-non polar incompatibility between PTH and PE phases in the composite. Mechanical properties of the composites are affected by low interaction between constituent phases. Thermal stability of the composites is improved by the incorporation of PTH. An exceptional increase in conductivity could be achieved by the incorporation of PTH in HDPE which can be attributed to preferential building of conducting network through the amorphous phase of the polymer. Dynamic mechanical analysis shows improved modulus indicating a build-up of PTH network in the matrix. A remarkable increase in dielectric constant of the composites could be achieved by incorporation of PTH making them promising candidates for capacitor fabrication. The composites exhibit excellent absorption of electromagnetic radiation in the X band, which makes them ideal materials for flexible EM shields with potential strategic applications.

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