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Mechanical, thermal and morphological behaviours of polybutylene Terephthalate/polyethylene Terephthalate blend nanotube composites

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ABSTRACT

Polybutylene Terephthalate (PBT) / Polyethylene Terephthalate (PET) nanotube composite blend was fabricated using melt blending technique in a twin extruder. The blend composition was optimized at PBT-PET weight ratio of 80-20. The effect of incorporation of MWCNT on the mechanical, electrical, thermal and morphological properties of the nanotube composites blend has been investigated. Mechanical properties show marginal improvement in impact strength, but considerable improvement in tensile strength and flexural strength. Changes in electrical properties observed. Increase in crystallisation temperature noted from DSC thermograms. DMA add the evidence for DSC and mechanical results. PBT - PET blend with CNT exhibited featureless XRD indicative of exfoliated structure. TEM micrographs also confirmed the same that the CNT has exfoliated into individual tubes.

Introduction

Blending of two or more polymers has emerged as an established route to design tailor made polymeric materials with desired attributes for various high performance applications [1-4]. Different approaches such as use of compatibilising agents, copolymers, grafting agents, reactive extrusion etc., have been the commonly used techniques to modify the interfacial region between the blends and increase the compatibility [5, 6].

More recently, PLS nanocomposites have attracted great interest both in industry and in academic, because they often exhibit remarkable improvements in properties when compared with virgin polymers. Layered silicates, with its inherent high aspect ratio ranging from 100 to 2000 offers more surface contact per unit filler within the polymer matrix resulting in enhanced performance characteristics with a minimum loading of 3-5 % [7].

An intercalated structure results when the polymer penetrates into the galleries of the layered structure resulting in a highly ordered arrangement of alternating clay platelet and polymer layers. An exfoliated structure is formed when the layered silicates are delaminated. Large improvement in the mechanical properties is observed when clay platelets are well dispersBlending of two or more polymers has emerged as an established route to design tailor made polymeric materials with desired attributes for various high performance applications [1-4]. Different approaches such as use of compatibilising agents, copolymers, grafting agents, reactive extrusion etc., have been the commonly used techniques to modify the interfacial region between the blends and increase the compatibility [5, 6].

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An intercalated structure results when the polymer penetrates into the galleries of the layered structure resulting in a highly ordered arrangement of alternating clay platelet and polymer layers. An exfoliated structure is formed when the layered silicates are delaminated. Large improvement in the mechanical properties is observed when clay platelets are well dispers and exhibit an exfoliated morphology within the polymer matrix.

PBT is one of the engineering plastics which have good combination of properties such as rigidity, hardness, abrasion, solvent resistance, electrical insulation and high rates of cyrstallisation that allow short cycle times in injection moulding [8-10]. However, PBT is strongly notch sensitive, give low notched Izod impact strength [11, 12] and break in a brittle fashion when standard notched specimens are tested. The strong notch sensitivity of PBT can be eliminated by the incorporation of impact modifiers [13] such as, nanoclays or in general nanomaterials which increase the surface area which in turn increases the mechanical properties. Thus, polymer nanocomposites, at loading levels of 2-3 % of nanomaterials exhibit enhanced mechanical properties, improved thermal properties when compared with neat polymers or their blends [14]. The cost difference between the neat matrix and its polymer nanocomposites is about 10-15 %.

PET possesses good tensile strength, stiffness, excellent dimensional stability, excellent melt strength with slower crystallisation rate and high tear strength. PET also has good Izod impact strength even at low temperature. Heat deflection temperature HDT, of PET and PBT are same at 1.8 MPa. The

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Tg of PET is 80°C and that of PBT is 25°C. PET belongs to the polyester group as PBT. Both PBT and PET form a well uniform phase on mixing.

Carbon nanotube is allotropes of carbon with a cylindrical nano structure. Nanotube name is derived from their size, since the diameter of a nanotube is in the order of a few nanometers [15]. A nanotube may consist of one tube of graphite called single walled nanotube, SWNT or a number of concentric tubes, called multi walled nanotube, MWNT. MWNT looks like a rope made of bundles of concentric SWNTs. The C-C bond length is 0.14 nm which is shorter than diamond indicating greater strength. This strength results from the covalent sp^2 bonds formed between the individual carbon atoms [16]. Diamond is considered to be the hardest material. Now CNT are on par with diamonds in hardness [17]. MWCNT can be excellent conductor [18, 19].

CNT has variety of applications. Researchers and companies are working to use carbon nanotube in various fields. Attracted by the properties of CNT, efforts were taken in the present work to add nanotube with PBT-PET blend and to prepare exfoliated nanotube composites. Mechanical properties, electrical properties, thermal properties and morphological studies were carried out for the samples prepared with 0.15 %, 0.30 % and 0.45 % weight ratios of carbon multi wall nanotube.

Experiemental

Materials

The polymer matrix used in this research is a commercial PBT (DUPONT ™ CRASTIN® S610SF NC010). PET was supplied by GE plastics. The CNT used was Sun nano ® MWCNT with diameter ranging between 10-30 nm and appear as black powder. PBT was blended with PET in different ratios like 90-10, 80-20 and 70-30. From the experimental results, 80% weight of PBT gives better results with 20% weight of PET and it was considered as an optimized ratio. MWCNT is incorporated in the weight ratio of 0.15 %, 0.30 % and 0.45 % with 80 % PBT toughened by 20 % PET.

Preparation of Blends

Initially PBT and PET were blended without filler, to get 90-10, 80-20 and 70-30 weight ratio to establish optimized blend ratio. Based on the tensile and impact strength, the optimized percentage of PET is 20% by weight. Then PBT/PET 80:20 blend mixture was mixed with 0.15 %, 0.30 % and 0.45 % weight ratios of carbon multi wall nanotube. PBT was dried at 100°C in an air circulated oven for 8 hours prior to blending. The blend was prepared via melt compounding method using twin screw extruder (Bersfort FRG Germany) at temperature range of 220°C with a screw speed of 150 rpm. After the extrusion, the extrudate was cooled in water bath and palletized. Finally these granules were injection molded as per ASTM using SP130 injection molding machine (Windsor, India) having clamping force 100T fitted with dehumidifier at a temperature range of 250 - 285°C.

Mechanical properties

The tensile tests were performed according to ASTM D 638 using SHIMADZU AUTOGRAPH (model AG 50 RNISD MS) at room temperature of 23 ± 1°C. The gauge length was set as 50 mm and the cross head speed was 50mm/min.Tensile strength, tensile modulus and elongation at break were recorded. The flexural properties of all the composites were measured with a Lloyd instruments Ltd, LR 100 KN, UK machine according to ASTM D 790 with a cross head rate of 2.82 mm/min. Izod impact strength was measured with a (ATS FAAR, Italy) impact tester according to ASTM D 256, method-A with notched samples. Five replicate specimens were used for each test and the data reported are the average of five tests.

MFI, as per ASTM D 1238 was carried out for all the PBT blend nanocomposite samples. As per ASTM D 257, the volume resistivity was measured for all the samples prepared. The dielectric strength experiment was carried out as per ASTM D 149 on all the PBT blend nanotube composite samples.

Differential Scanning Calorimetry (DSC)

The DSC scans were carried out by using a Perkin Elmer (Diamond DSC) calorimeter in a nitrogen atmosphere. The sample was first heated from 50°C to 300 °C at 10°C/min and cooling rate was controlled at 10°C/min from 300°C to 50°C. In order to measure the energies of melting, indium was used as standard.

Dynamic Mechanical Analysis (DMA)

Dynamic Mechanical Analysis (DMA) was performed using Netzsch DMA 242 in three points bending mode at frequency of 1Hz and 120 μm over a temperature range of -50°C to 150°C at a heating rate of 10°C/min.

X- Ray Diffraction Analysis (XRD)

Both for the clay and nanocomposites, XRD was recorded using Philips X' pert MPD, Japan make, which had a graphite monochromator and Cu Kα radiation source and was operated at 40 kV and 30 mA.

TEM Analysis

TEM analysis of the specimens was carried out using JEOL JEM 2100 HRTEM. The HRTEM has LaB6 Filament and acceleration voltage of 200 kV. Ultra thin sections of sample were prepared employing ULTRACUT UCT LEICA MICRO SYSTEM microtome with a diamond knife at temperature of -60 °C at N_2 atmosphere.

Results and Discussion

Effect of loading PET on mechanical properties of PBT

The mechanical properties, among all the properties of plastic materials, are often the most important properties because virtually all service conditions and the majority of end-use applications involve mechanical loading [(20)]. Impact strength is toughness and the property of plastics probably most useful to consider [(21)]. While it is possible to perform impact tests and to rank a series of plastic materials, it is impossible to predict whether the material will serve satisfactorily under the working conditions. The factors which may influence are additives, impurities, temperature, geometry, orientation and morphology, surface condition, energy and speed of any impacting blow, the environment and the strains due to external loads. PET was added in small weight proportions like 10 %, 20 % and 30 % to PBT to watch out the changes in mechanical properties of PBT.

The impact of PET on mechanical properties of PBT when added in different weight ratios are given in Table 1.

It is evident from the Table 1 that Izod impact strength increases up to 80-20 weight ratio of PBT-PET and the increase is nearly 3.6 folds that of virgin PBT. Normally, PBT and PET are immiscible with each other as both belong to the same ester group and both are semi crystalline in nature. But without any added additives, blend of PBT and PET show improved mechanical property.

The values for tensile strength and flexural strength increases up to 80-20 weight ratio and then it decreases. Tensile modulus increases up to 80-20 weight ratio but 70-30 weight ratio shows only a slight variation from that of 80-20. Flexural modulus values also show the same trend as that of Izod impact strength, tensile strength and flexural strength. Thus, it is concluded that PBT-PET blends show optimised values for 80-20 weight ratio and this blended polymer exhibits better and improved mechanical properties.

Effect of loading CNT with PBT-PET 80-20 blend

Table 2 gives the mechanical property values obtained on loading CNT with PBT-PET 80-20 weight ratio. 0.15 %, 0.30 % and 0.45 % weight ratios of CNT were added to PBT-PET 80-20 blend. The results show that the impact strength decreases on the addition of CNT to PBT-PET 80-20 blend. Both tensile strength and flexural strength are enhanced on the addition of CNT to PBT-PET 80-20 blend. Among the three nanotube composites, PBT-PET 80-20 blend loaded with 0.30 % CNT exhibits the maximum tensile strength and flexural strength.

The addition of CNT to PBT-PET 80-20 blend offers very little variation in the tensile modulus except 0.30 % CNT nanotube composite which shows increase in the tensile modulus value. On contrast, addition of CNT to PBT-PET 80-20 blend increases the flexural modulus value for all the three nanotube composites. For both tensile modulus and flexural modulus, 0.30 % CNT exhibits the maximum value. The samples with CNT, have shown better results with tensile and flexural properties and compensated the loss due to impact strength. Ultimately, the sample with CNT should be regarded as tough as PBT-PET 80-20 blend. Study of thermal properties and morphology would reveal the inner structure of nanotube composites.

Effect of loading CNT with PBT-PET 80-20 blend on MFI

MFI measures the rate of extrusion of thermoplastic material through an orifice of specific length and diameter under prescribed conditions of temperature and pressure. MFI is primarily used as a means of measuring the uniformity of the flow rate of the materials. MFI is an inverse measure of molecular weight [22]. Table 3 gives the measured MFI values for the addition of CNT to PBT-PET 80-20 blend. MFI decreases for every addition of weight ratio of CNT. Since MFI is inversely related to molecular weight, carbon multi wall nanotube with high molecular weight reduces the viscosity as well as MFI.

Electrical properties of PBT-PET 80-20 blend with CNT loading

Carbon nanotubes are excellent conductors [18, 19]. The electric current carrying capacity of nanotube is 1000 times higher than copper wire. Electrical properties were studied for the samples prepared with 0.15 %, 0.30 % and 0.45 % weight ratios of carbon multi wall nanotubes. The Table 3 shows that on incorporating CNT, the value of dielectric strength decreases emphasising that carbon nanotubes are good conductors. The weight percentage ratio of carbon nanotubes is too small to impart any adverse changes. Nevertheless, the inclusion of carbon nanotubes has made its imprint by reducing the values of dielectric strength from the pure PBT or PBT-PET 80-20 blend.

From Table 3, it is observed that incorporation of CNT decreases the value of volume resistivity when compared with the pure PBT or PBT-PET 80-20 blend. This confirms the fact that the character of CNT has been imparted to the polymer blend taken. Thus, it is clear that CNT with good conducting properties can alter the electrical properties of the polymer sample incorporated to it. The sample with 0.45 % of CNT shows marked decrease in the value of volume resistivity for the PBT-PET nanotube composites.

Thermal studies

Thermal analysis plays a vital role in the characterisation of polymer [23]. Knowledge of thermal behaviour is not only characterisation of materials especially for thermal stability and for selection of appropriate end users.

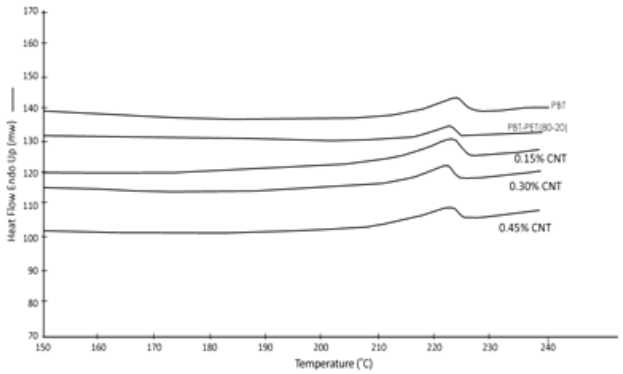
Differential scanning calorimetry

DSC is one of the most important tools used to investigate the thermal properties of the polymers. Melting temperature Tm, crystalline temperature Tc, enthalpy of melting ΔHm and percentage of crystallinity Xc has been detected from DSC thermograms. Percentage of crystallinity, Xc was calculated, taking into consideration that 100 % PBT has a heat of fusion value of 142 J/g [24].

Effect of loading MWCNT with PBT-PET 80-20 blend

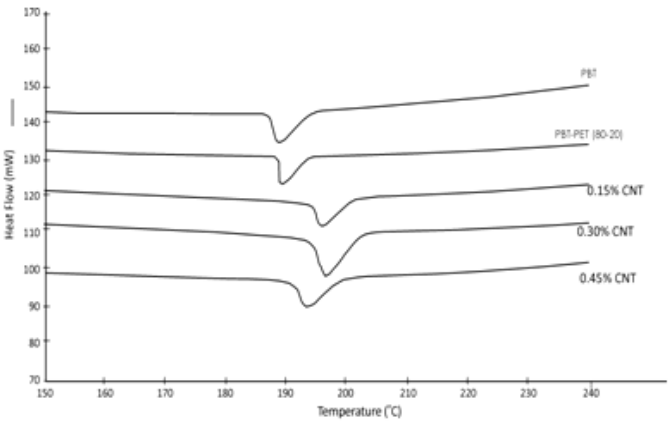
MWCNT in the weight ratio of 0.15 %, 0.30 % and 0.45 % were added to PBT-PET 80-20 blend and DSC analysis was carried out. The results are given in Table 4 and the DSC thermograms are depicted in Fig. 1 and Fig 2. It is evident from the Table 4 that the Tm decreases for PBT-PET 80-20 blend and its nanotube composites when compared to pure PBT. 0.15 % CNT and 0.45 % CNT composites show a very marginal increase in Tm values from PBT-PET 80-20 blend. Nanotube composites have higher Tc values than pure PBT and PBT-PET 80-20 blend. PBT-PET CNT composites show decrease in Xc value when compared to pure PBT and PBT-PET 80-20 blend. These observations are in agreement with the literature. A decrease in crystallinity upon the addition of MWCNT has been observed in PEO nanocomposites [25]. PA 6-ABS blend exhibited a decrease in Tc but addition of MWCNT showed an increase in Tc considerably [26]. Addition of MWCNT to PE had no effect on the melting temperature of PE. However, the Tc increased by about 8°C with 10 % weight of MWCNT, indicating that MWCNT have a nucleating effect on PE [27].Thus, the observations of the present work confirms that MWCNT act as nucleating agents which is in agreement with the results of the earlier works.

Fig 1 DSC (Tm) of PBT-PET 80-20 blend with their Nanotube composites



DSC(Tm)of PBT-PET 80-20 with carbon nanotubes

Fig 2 DSC (Tc) of PBT-PET 80-20 blend with their Nanotube composites



DSC (Tc) of PBT-PET 80-20 with carbon nanotubes

Dynamic mechanical analysis, DMA

DMA has emerged out as one of the most powerful tools available for the study of the behaviour of plastic materials.

DMA gives the fundamental aspects of morphological structure of polymer. The low storage modulus indicates that the material is easily deformed by an applied load. Loss modulus is the contribution of the viscous component in the polymer, that portion of the material will flow under conditions of stress.

The peak of the loss modulus is conventionally identified as the Tg, even though the DMA plot clearly shows that the transition is a process that spans a temperature range. The loss modulus provides the best agreement with determinations made by other thermal analysis methods and ASTM has recently codified this into D-4065. The lack of shift in the Tg indicates that this is an immiscible blend.

Effect of loading MWCNT with PBT-PET 80-20 blend Nanotube composites

Table 5 provides the DMA results for the addition of CNT to PBT-PC 80-20 blend. Fig 3 and Fig 4 enumerate the effect of addition of CNT to PBT-PC 80-20 blend as diagrams. E' value for 0.15 % CNT weight ratio is lesser than pure PBT and PBT-PC 80-20 blend. 0.30 % and 0.45 % weight ratios of CNT, show higher value of storage modulus than pure PBT as well as PBT-PC 80-20 blend. 0.45 % CNT has the highest E' value among the three nanotube composites prepared. Thus, it is clear that E' values have improved on addition of CNT with PBT-PC 80-20 blend. MWCNT induce an increase of storage modulus E' slightly under Tg and visibly above. Probably, it results from the variations in the molecular mobility of polymer chains resulting from the addition of nano filler as well as the stiffening effect of CNT. In the present work also such an enhancement was seen.

Fig 3 DMA of PBT, PBT - PET 80-20 and their nanotube composites

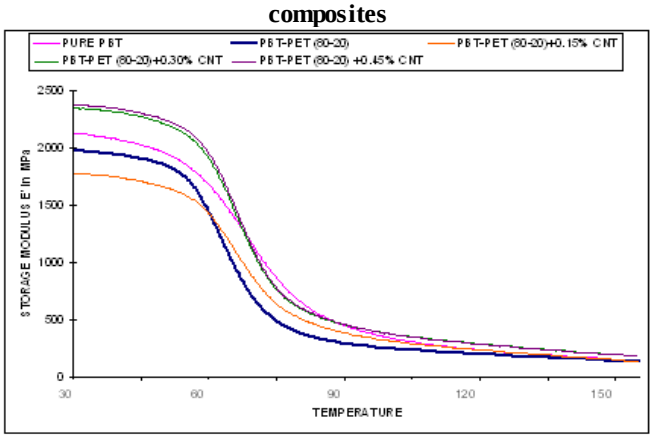


Fig 4 DMA of PBT, PBT - PET 80-20 and their nanotube composites

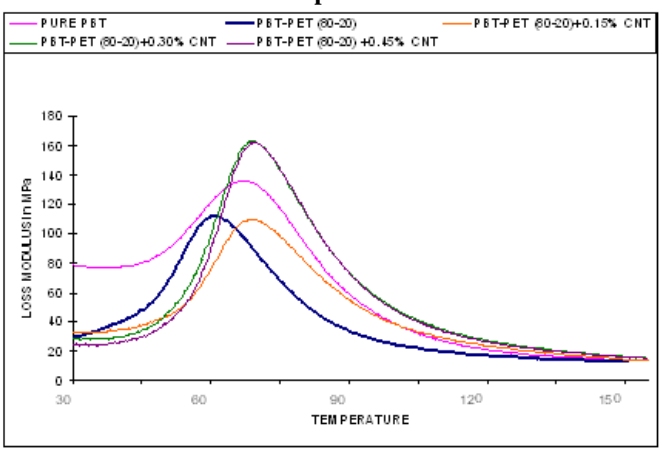
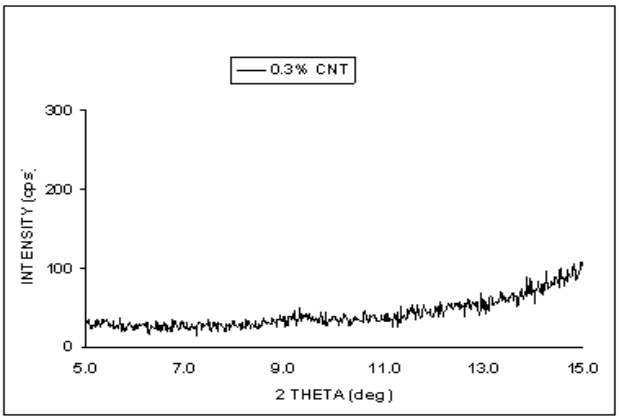


Fig.5 XRD of PBT-PET 80-20 blend with 0.30 % CNT



Loss modulus curve providing Tg values for nanotube composites are lower than pure PBT. Tg values increases for the addition of CNT to PBT-PET 80-20. Tg values correspond to the loss modulus peaks exhibited basically the same trend although the differences between the different materials were marginal. The reason was ascribed to an improved interaction between the PBT-PET and MWCNT and the constraint of PBT-PET chain segment movements by the exfoliated nanotubes in the composite. It has been reported that, the increase in storage modulus and Tg due to clay particles could be attributed to the hindrance of macro molecular mobility of polymer chains caused by the well dispersed silicate layer in the matrix, as proven in other thermoplastic polymer, clay systems [28, 29]. The improvement in Tg suggests an increase in the thermal stability of the nanocomposites [30]. The more pronounced enhancement of mechanical properties should arise from MWCNT was due to the improved interaction between the MWCNT and PBT-PET matrix.

Morphology

Effect of loading CNT with PBT-PET 80-20 blend

From the DMA analysis, carried out for the sample PBT-PET 80-20 blend, it is observed that only one Tg peak is seen in the tan δ curve at a temperature of 71.58°C. This Tg peak corresponds to glass transition temperature. The existence of a single Tg between those of the pure components in polymer blends is evidence for their miscibility [31]. DSC analysis of PET-PBT 30-70 and 70-30 blends carried out by Marcin et al., [31] indicated only one melting point corresponding to the major component present. Results of miscible PET and PBT, below melting temperature, shows the suppression of crystallisation of minor components and possible chemical interaction in the blend [32]. In the present work also, DSC thermogram of PBT-PET 80-20 blend exhibits only one melting point corresponding to the major component PBT has occurred. Immiscible polymer blend of PP/PET was characterised by melting temperatures of the individual components [32].

XRD and TEM have been regarded as complementary in characterising the micro structure of the PLS nanocomposites. XRD of PBT-PET 80-20 blend with 0.30 % CNT is shown in Fig 5. In the study of morphology, XRD exhibits featureless XRD at 2θ less than 10°, showing that PBT-PET-MWCNT nanotube composites may have an exfoliated structure.

It should be noted that a few completely exfoliated PLS nanocomposites exhibit no peak, but instead display a gradual increase in the diffraction intensity towards low diffraction angles [(33, 34)], this is not always the case, however. In fact, it was extensively reported that some PLS nanocomposites show featureless XRD patterns when they exhibit exfoliated or delaminated structures [(35)].

Table 1
Effect of loading PET with PBT on mechanical properties

PBT/PET weight	Izod impact strength	Tensile strength MPa	Flexural strength MPa	Tensile modulus GPa	Flexural modulus GPa
100:0	50	50	71	2.429	2.169
90:10	133	61	79	2.779	3.203
80:20	181	67	84	2.928	3.350
70:30	179	63	81	2.937	3.259

Table 2
Effect of loading CNT with PBT-PET 80-20 blend on mechanical properties

percentage weight ratio of			Izod impact strength J/m	Tensile strength MPa	Flexural strength MPa	Tensile modulus GPa	Flexural modulus GPa
PBT	PET	CNT					
80	20	0	181	67	84	2.9	3.33
80	20	0.15	55	69	94	2.8	4.20
80	20	0.30	51	73	105	3.7	4.85
80	20	0.45	48	70	100	2.9	4.59

Table 3
Effect of loading CNT with PBT-PET 80-20 blend on MFI

PBT percentage weight ratio	PET percentage weight ratio	CNT percentage weight ratio	Melt flow index g/10 min	Volume resistivity Ohm.cm	Dielectric strength KV/mm
100	0	0	43.6	$6.2x10^{16}$	16.00
80	20	0	31.7	$6.4x10^{16}$	19.80
80	20	0.15	28.8	$2.0x10^{14}$	15.31
80	20	0.30	17.3	$8.0x10^{13}$	14.21
80	20	0.45	12.4	$5.0x10^{13}$	12.74

Table 4
DMA data of PBT, PBT-PET 80-20 blend and their nanotube composites

Polymer type	Melting temperature Tm °C	Heat of fusion Hf J/g	Crystallisation temperature Tc °C	Percentage of crystallinity Xc
100% PBT	224.25	21.63	188.04	15.23
80% PBT 20% PET	222.97	26.03	188.67	18.33
80% PBT 20% PET 0.15% CNT	223.50	20.20	195.28	14.23
80% PBT 20% PET 0.30% CNT	222.52	19.43	196.45	13.68
80% PBT 20% PET 0.45% CNT	223.31	15.37	193.55	10.82

Table 5
DMA data of PBT, PBT-PET 80-20 blend and their nanotube composites

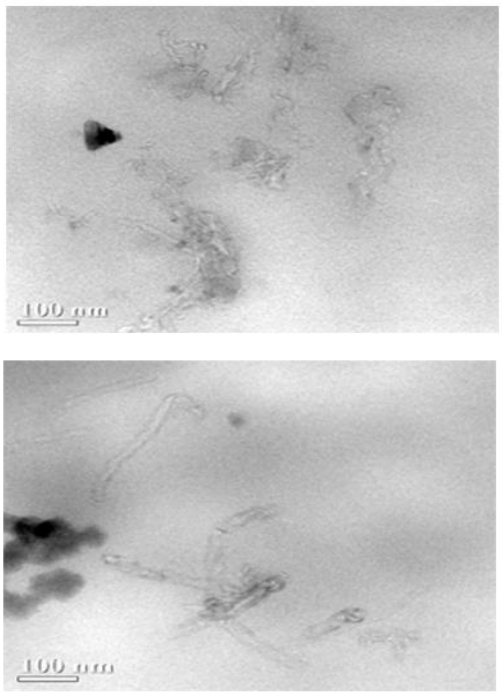
Polymer type	Storage modulus E' MPa	Loss modulus Tg temperature °C	Tan delta peak temperature °C
Pure PBT	2125	68.04	82.16
PBT-PET 80-20	1960	60.43	71.58
PBT-PET 80-20 + 0.15 % CNT	1770	63.86	74.37
PBT-PET 80-20 + 0.30 % CNT	2350	64.29	73.72
PBT-PET 80-20 + 0.45 % CNT	2375	64.72	73.29

Vain et al., [36] and Galgali et al., [37] also observed featureless XRD patterns even for partially exfoliated nano structures.

TEM micrograph of PBT-PET 80-20 blend with 0.30 % CNT is shown in Fig 6. TEM study also clearly reveals a completely exfoliated structure supporting the results of XRD studies. Long nanotubes with relatively well graphitised walls are observed. Microscopic investigations of PBT/PET/CNT nanocomposites confirm rather homogenous distribution of CNT in polymer matrix. The MWCNT can be clearly identified and are uniformly dispersed as single nanotubes and as aggregates of varying dimensions. In some instances, no polymer seems to be present in the inner most tube. Microscopic examination across the length scale would confer that the MWCNT are well distributed and dispersed in PBT-PET matrix. Interestingly, the nanotubes found in majority to be migrated into the matrix materials. No agglomerates could be observed. On studying the effect of loading of MWCNT with PBT-PET 80-20 blend, an exfoliated structure was enumerated with MWCNT acting as

compatibiliser and 0.30% weight ratio of MWCNT has been shown to be the optimised ratio.

Fig 6 TEM micrograph of PBT-PET 80-20 with 0.30 % CNT



Conclusion

PBT/PET blend nanotube composites were prepared by employing melt compounding technique. Incorporation of PET to the PBT matrix increases the impact strength of the virgin matrix and tensile and flexural properties. However, incorporation of MWCNT along with PBT/PET increases tensile and flexural property by sacrificing impact strength in the blend matrix. CNT acts as nucleating agents and influences the rate of crystallisation and the crystallisation temperature of the semi crystalline polymer matrix. In case of PBT/PET blend nanocomposites, the storage modulus increased with the incorporation of the nanotubes. A slight improvement in the thermal stability of the PBT-PET 80-20 was noticed after the incorporation of the nanotube. XRD and TEM clearly show that clay has exfoliated and dispersed in PBT-PET blend matrix.

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