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Thermal and Rheological Properties of Polystyrene Nanocomposites with Carbon Nanotube and Boron Nitride as Dual Nanofiller: Effect of Boron Nitride Content.

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ABSTRACT

This work is focused mainly to understand the effect of the dual fillers such as Boron nitride (BN) and carboxylic acid functionalized multiwall carbon nanotube (CNT) on the thermal and rheological properties of Polystyrene (PS) nanocomposite. The nanocomposite films were produced by keeping the CNT content at 0.3wt% and varying BN content. The interaction between PS and dual fillers (CNT and BN) was analyzed by X-ray diffraction, thermogravimetric analysis and melt rheology. XRD profiles of nanocomposites showed a small increase in d-spacing with an increase in BN concentration. TGA results indicated that, when 20% mass loss taken as reference, the addition of 1wt% BN to the nanocomposites increased the thermal stability by 14 °C. With further increase in BN to 5wt%, it was observed that the thermal stability increased by 19 °C in comparison with PS. Thermal degradation kinetics, degradation mechanism and integral procedural decomposition temperature were evaluated using Coats Redfern method, Criado method and Doyle's method, respectively. The effect of dispersion of varying concentration of nanofillers over Polystyrene matrix can be measured using melt rheological analysis performed on nanocomposites between 1 to 100 rad/s. Addition of BN shifted the cross over frequency to lower angular frequency that reveals the increased stiffness with increasing BN content.

Keywords: PS; CNT; Boron nitride; Nanocomposites;

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INTRODUCTION

Polymer nanocomposite consists of polymer matrix reinforced with nanofiller. Based on the nanofiller reinforced into the polymer matrix, polymer nanocomposites were found to have improved properties like thermal stability, electrical conductivity and thermal conductivity. These properties have drawn keen attention on various industrial application like heat exchangers, packaging, electronic appliances, sensors, machine parts, power electronics, etc., The major factors which affects the preparation of polymer nanocomposites are structure of filler, techniques used in dispersing nanofiller in the polymer matrix, alignment of filler into the polymer matrix and the interaction between nanofiller and the polymer matrix.

There are three basic methodologies in which nanocomposites can be synthesized [1-3]. Solvent blending is the trouble free process used for laboratory scale preparation of polymer nanocomposites. In this method, the nanofiller and polymer are mixed in a suitable solvent in which the polymer has better solubility. Agitation is generally done in the form of magnetic stirring, ultrasonication, shear mixing or reflux or the combination of above. The above method is unsuitable for polymers which are insoluble and commercially large scale handling of the solvent is not possible. Melt processing is an industrially attractive process in large scale production of nanocomposite through extrusion, fiber spinning, compression molding or injection molding. In this procedure, the polymer is softened by heating above a sufficient temperature to form a viscous liquid and the nanofillers were added to the polymer by shear mixing to give better dispersion of filler on the polymer matrix. In-situ polymerization comes handy for the polymers which are either insoluble or thermally unstable. In this method, the nanofiller is dispersed into monomer matrix, either in presence or absence of solvent and then polymerized with conventional polymerization techniques. The advantage of this method is higher nanofiller loading in the preparation of polymer nanocomposites.

The fillers for reinforcement were chosen, based on the properties which we would like to alter in the polymer matrix. Carbon nanotubes are one dimensional nanofillers that brought a revolutionary change in the field of polymer nanocomposites ever since its discovery [4]. There are two types of carbon nanotube: (i) Single graphene sheet rolled to form a single walled carbon nanotube, and (ii) if several graphene layer were stacked and rolled together in concentric fashion giving rise to multiwalled carbon nanotube. Carbon nanotube express a wide range of impressive properties. In terms of mechanical properties, multiwalled carbon nanotubes possess high elastic modulus in the order 2.4 TPa [5, 6] and Young's modulus [7] and tensile strength [8] were found to be between 100-1000 GPa and 11-63 GPa, respectively. Electric conductivity of multiwalled carbon nanotube was reported to be in the range of 10^7 to 10^8 S/m using four probe method with tungsten for lithographic deposition [9]. They possess incredibly high value of thermal conductivity around 6000 W/mK at room temperature [10]. When the carbon nanotube was introduced as nanofiller into Polystyrene matrix using solution mixing and precipitation, it was observed that maximum thermal degradation increased upto 21°C [11]. The polymer nanocomposite prepared using solvent blending method revealed that the thermal stability and melt rheological parameters increased with increasing loading and the hardness increased for about 7.5% with 0.7 wt% loading [12].

Boron nitride (BN) belonging to the category of ceramic filler has gained significant attention in the field of power electronics because of its high thermal conductivity and electrical resistivity properties. Boron nitride is classified into various types based on the structure such as amorphous, cubic, wurtzite, hexagonal and nanotubes. Nanotube form of Boron nitride possess Young's modulus in the range of 0.837 to 0.912 TPa [13] and elastic modulus around 722 GPa [14]. The structure of hexagonal Boron nitride is similar to graphite where carbon atoms in graphite are replaced by alternative Boron and Nitrogen ions [15]. hBN sheets were formed by Boron and Nitrogen atom held together by strong covalent bonds and each hBN layer was connected by weak vander Waals force [16]. Nanocomposite films made out of hBN provided high thermal and mechanical stability [17]. The Boron nitride nanocomposite was prepared using Polystyrene as polymer matrix via solvent blending method accompanied by sonication and precipitation. Amongst various solvents used (Dichloromethane, Toluene and Chlorobenzene), the nanocomposite prepared using toluene as solvent demonstrated high electrical breakdown strength and good dispersion under SEM examination [18].

Recently, the preparation of dual nanofillers based nanocomposites has gained a significant attention in the field of composite processing due to the following reasons: Increased single nanofiller loading may cause agglomeration of nanofiller, increase in viscosity creates difficulties in nanocomposite processing, and increase in nanofiller loading after a critical weight percentage may hamper the mechanical properties. When dual

nanofillers nanocomposites were prepared using Polyphenylenesulfide by melt compounding using a twin screw extruder, it was observed that the thermal conductivity increased with increasing modified BN loading at 25 °C [19]. Similarly, when the Polybutylene Terephthalate composites was prepared by using injection molding with fixed BN loading and various Carbon fiber (CF) loading, it was observed that, electrical and thermal conductivity found to increase with an increase in the CF loading and their values reached a constant at 30 wt% CF loading. Synergetic interaction between two fillers improved the stiffness of the composites [20]. Various observation from the literature stated that the combination of carbon (multiwalled carbon nanotube) and ceramic (Boron nitride) nanofillers has significant impact on thermal, electrical conductivities and mechanical properties on various polymer. Therefore, in our study, we have prepared PS nanocomposites with dual fillers (CNT and BN) by solvent blending method coupled with sonication and the effect of BN content on thermal and rheological properties of Polystyrene nanocomposites was examined.

MATERIALS AND METHODS

Materials used

Polystyrene (PS) was procured from National chemicals Ltd. Gujarat. Toluene, Xylene, Tetrahydrofuran (THF) and N,N-Dimethyl formamide (DMF) were obtained from Merck Specialities Pvt. Ltd. Carboxylic acid (>8%) functionalized multiwalled carbon nanotube (CNT) and Boron nitride (BN) were obtained from Sigma-Aldrich, USA. All the materials were used as it is without further chemical treatment.

Nanocomposite synthesis

PS, CNT, BN and different solvents like Toluene, Xylene, Tetrahydrofuran, N,N-Dimethyl formamide were used in nanocomposite synthesis. The nanocomposite films were synthesized by keeping loading of CNT at 0.3 wt% and varying the loading of BN and the nanocomposites were named as PSCNTnBN (n = 1, 3, 5 wt% of BN) accordingly. Prior to synthesis, Polystyrene and nanofillers were maintained at a temperature of 60 °C for 6 h and then it is placed inside vacuum desiccator to reduce the interference of moisture in nanocomposite synthesis. PS, CNT and BN were taken individually in an appropriate proportion and then dispersed in the solvent. These three solutions were stirred constantly for 12 h and they were sonicated using a bath sonicator (REMI R-24 Ultrasonicator, Mumbai) for 1.5 h to ensure proper dispersion of nanofillers. These solutions were mixed together and the solution was stirred for 12 h, followed by sonication for 1.5 h. Later, the solution was poured uniformly in a petridish and kept undisturbed till all the solvent in the petridish was evaporated under ambient condition. The nanocomposite film was peeled out of the petridish right after complete removal of solvent and the nanocomposite film was subjected to various characterization techniques. Figure 1 briefly explains the process for nanocomposite synthesis.

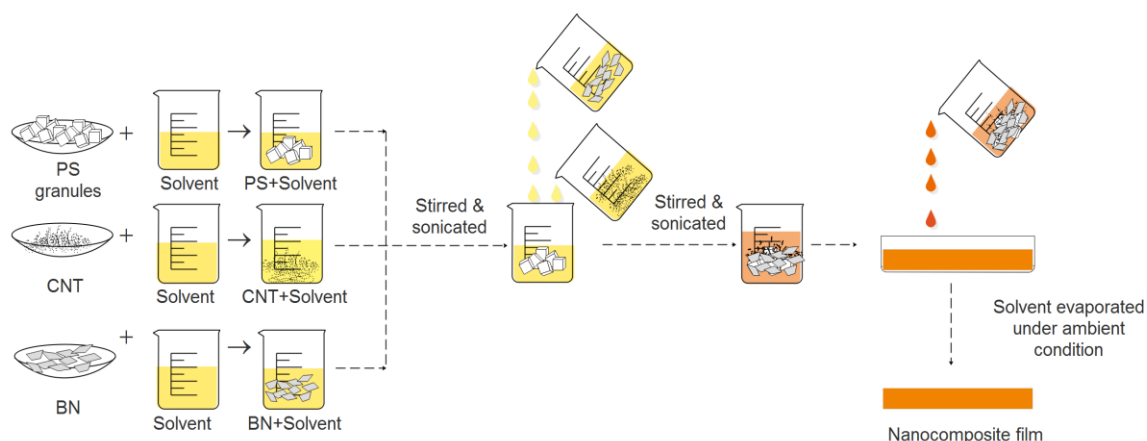


Figure 1: Nanocomposite film synthesis

Solvent screening

Solvents were chosen based on their ability to dissolve Polystyrene. In each of the solvent, 0.3wt% of CNT was added, stirred continuously for 12 h and then sonicated for 1.5 h. Then the degree of dispersion of CNT was observed and the nanocomposite film was prepared based on the procedure discussed above.

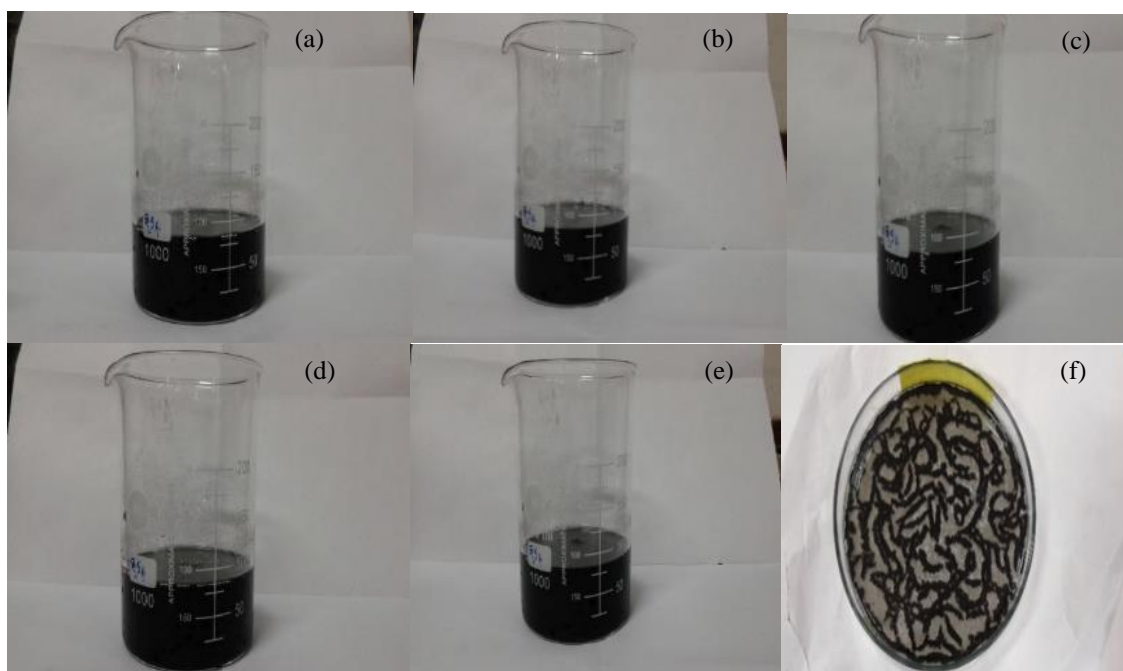
Characterization techniques used on nanocomposites

The XRD patterns were recorded between the 2θ range of 5 to 80° with scanning rate of 3°/min, using Bruker D8 Advanced X-ray Powder diffractometer equipped with Ni-filtered Cu K α radiation ($\lambda=0.15418$ nm) at room temperature. TEM analysis was conducted using JEOL, model number JEM 2100 to investigate the dispersion of BN and CNT in the PS matrix. The thermo-gravimetric analysis (TGA) was performed under Nitrogen atmosphere with a flow rate of 40 ml/min on a TG 209 F1, Libra Analyser, with a heating rate of 10 °C/min, for a temperature range between 30-700 °C. The sample weight was maintained around 6.5 mg. The rheological properties were measured using Anton Paar rheometer MCR 301 series at a temperature of 190 °C using a parallel plate setup with plate diameter of 50 mm.

RESULTS AND DISCUSSION

Solvent screening

Solvent screening was done to find out suitable solvent in which CNT could disperse effectively with minimum aggregation and with minimum solvent being retained on the nanocomposite after casting and drying of the sample. Dispersion of CNT on each solvent was observed for few minutes after sonication for 1.5 h. When THF and DMF were used as a solvent, a stable dispersion was observed for 5 minutes. When the nanocomposite with the above solvents was casted on the petridish, phase separation was noticed with DMF as solvent and a very high agglomeration of CNT over Polystyrene matrix was observed with Tetrahydrofuran as solvent as seen in Figure 2 (f). The possible reason may be higher rate of evaporation of solvent from the nanocomposite films due to its lower boiling point when experiment is performed under ambient conditions.



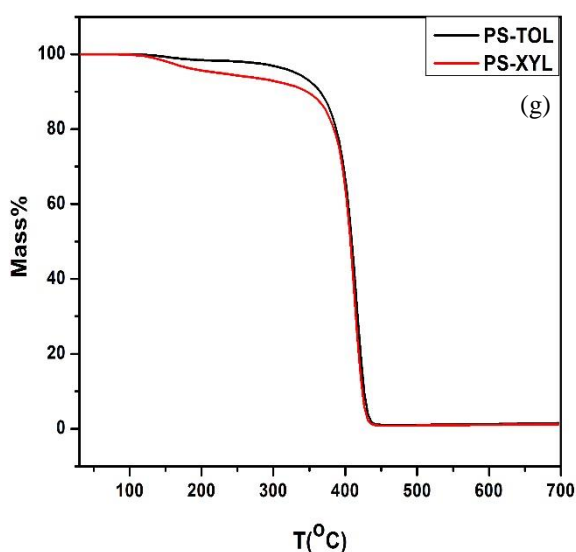


Figure 2: (a-e) shows the dispersion of CNT on Tetrahydrofuran for every 1 minute right after sonication, (f) agglomeration observed when nanocomposite film was synthesized using Tetrahydrofuran as solvent and (g) Comparison between TGA of PS nanocomposites produced using Xylene and Toluene as solvent

However, the nanocomposite films casted using Xylene and Toluene as a solvent, displayed a good dispersion of CNT on Polystyrene matrix. Then the nanocomposites were dried for two weeks prior to characterization using TGA for further screening of solvent.

Figure 2(g) depicts the TGA data of Polystyrene films produced using Toluene (PS-TOL) and Xylene (PS-XYL) as solvent. Results clearly indicated that the xylene was not completely removed from the nanocomposite during the drying process of the sample. Hence, slightly higher weight loss was noticed when the sample (PS-XYL) was heated beyond 130 °C (boiling point of xylene), which is clearly seen as a small dip in the TGA curve. From this analysis, it can be concluded that toluene is a right solvent for the preparation of PS nanocomposite.

X-ray diffraction (XRD) analysis

The XRD results of individual compounds shown in Figure 3 indicates that for pure PS, the broad peaks occurs at 2θ value of 19.4° and 10.9° represent the amorphous structure of polystyrene [12]. The characteristic peak of CNT around 25.9° and a short peak around 43.2° related to the interlayer spacing of nanotubes (d_{002} and d_{100} reflections of C atoms) [21]. The obtained XRD pattern of Boron nitride is in good agreement with the literature [22], confirming that the Boron nitride is of the hexagonal form (See Figure 3 (c)). The sharp peak at 26.7° corresponds to the characteristic (002) plane, which represents the interlayer distance between the two sheets of BN. The Scherrer equation gives us information regarding the mean size of crystals and it is described as follows:

$$\tau = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

Where, τ is the mean size of crystalline domains (nm), k is the shape factor (=1), λ is the wavelength of X-ray (nm), β is the line broadening at half the maximum intensity (rad). Using the above equation, mean crystal size of BN was estimated to be 24.105 nm. When the BN was added along with CNT into Polystyrene, it was observed that the peaks for CNT did not appear, which may be due to dispersion of CNT in the PS matrix. However, the BN's (002) peak did appear with even 1 wt% loading of BN. This may be due to intercalated form of BN on PS matrix. Bragg's law was used on (002) peak of BN to find the interlayer distance of BN layers and was presented in Table 1. The equation for Bragg's law was described as follows:

$$d = \frac{n\lambda}{2\sin\theta} \tag{2}$$

Where, d is the interlayer distance, n is the positive integer ($=1$), λ is the wavelength of X-ray (nm), θ is the scattering angle (degree).

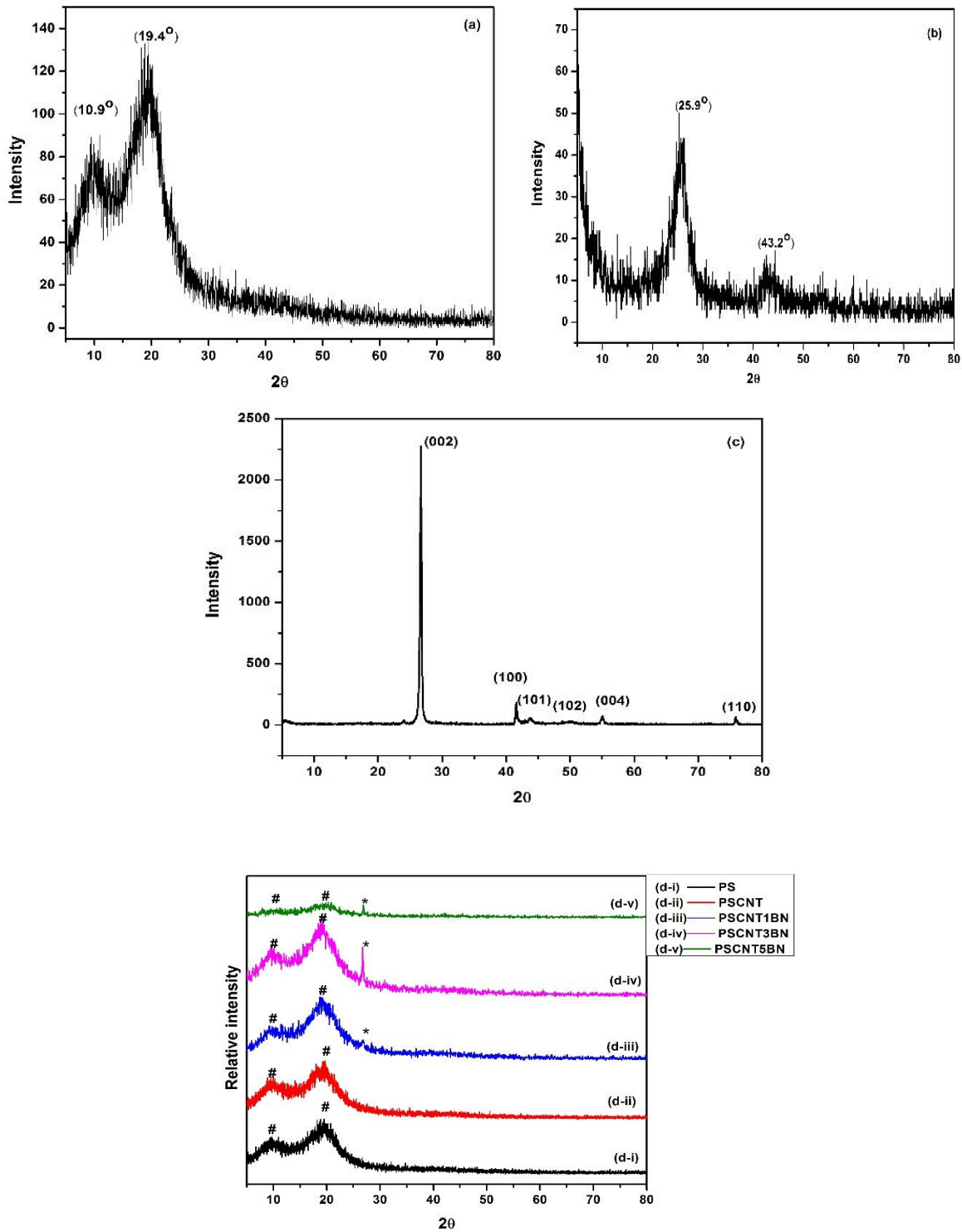


Figure 3: XRD of (a) PS, (b) CNT , (c) BN and (d-(i-v)) nanocomposites (*- peaks of BN, #-peaks of PS)

Table 1: Interlayer spacing of BN on nanocomposites with increasing content

Sample	2 θ	θ	d ₀₀₂ of BN (nm)
BN	26.7	12.925	0.336
PSCNT1BN	25.85	12.825	0.338
PSCNT3BN	25.65	12.825	0.338
PSCNT5BN	25.65	12.725	0.341

TEM analysis

The TEM images of PSCNT3BN under two different magnifications are shown in Figure 4. There was quite a significant interaction between irregular shaped BN and thread like CNT observed. The TEM results were similar to that as reported in literature [22]. TEM image also confirmed that the nanocomposite produced using solvent blending assisted sonication possessed the following: (i) CNT was dispersed in the PS matrix, (ii) layers of BN was observed and the results obtained from XRD matches the images proving the intercalated nature of BN with CNT and PS.

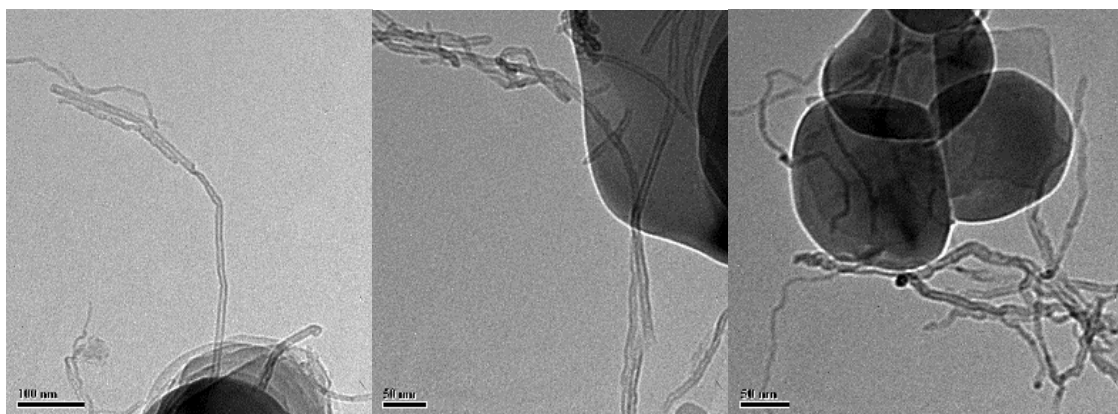


Figure 4: TEM images of PSCNT3BN

Thermo-gravimetric analysis

The thermo-gravimetric analysis is a method in which the change in mass of materials was measured as a function of temperature and heating rate. TGA data of the nanocomposite films are depicted in Figure 5. The TGA findings clearly revealed the effect of fillers on the thermal stability of nanocomposite. As expected, the addition of filler into PS matrix has shifted the onset of thermal degradation to a higher temperature. TGA results indicated that, when 20% mass loss was taken as reference, the addition of 1wt% BN to the nanocomposites increased the thermal decomposition temperature by 14°C. With further increase in BN to 5wt%, the improvement of thermal degradation temperature was observed by 19°C in comparison with PS (see Table 2). When BN and CNT added individually as filler, the onset of thermal degradation increased in comparison to PS. An increase of BN content revealed that the complex interaction between the fillers and nanocomposites was in such a way that, the addition of BN caused a mass transfer barrier for volatile matter to dissociate from the nanocomposite, thereby increasing the thermal stability of the nanocomposites.

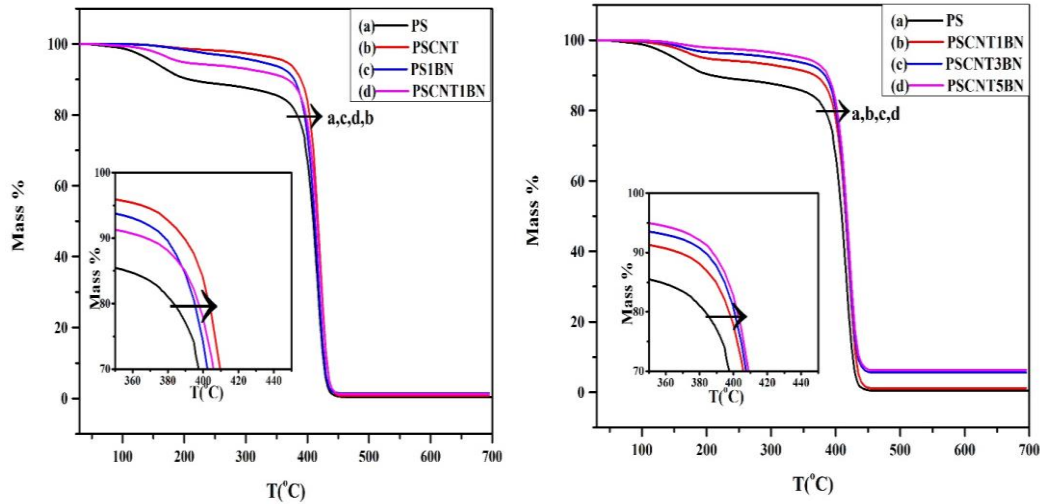


Figure 5: TGA of nanocomposites

Table 2: Thermal stability of nanocomposites

Sample	Temperature at 20% weight loss (°C)	Temperature at 50% weight loss (°C)
PS	383	409
PSCNT	403	417
PS1BN	395	412
PSCNT1BN	397	415
PSCNT3BN	400	416.
PSCNT5BN	402	418

Analysis of thermal degradation kinetics for nanocomposites

The effect of single filler and dual filler on the thermal degradation of nanocomposites can be observed from the kinetic parameters like order of reaction (n), pre exponential factor (A), activation energy (E) and heating rate (β). All these parameters can be determined using Coats-Redfern kinetic model [12].

$$\ln\left(-\frac{\ln(1-\alpha)}{T^2}\right) = \ln\left(\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right) - \frac{E}{RT}, n = 1 \tag{3}$$

$$\ln\left(\frac{1-(1-\alpha)^{1-n}}{(1-n)T^2}\right) = \ln\left(\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right) - \frac{E}{RT}, n \neq 1 \tag{4}$$

Where,

$$\alpha = \frac{W_o - W_t}{W_o - W_\infty} \tag{5}$$

W_o is mass at time $t=0$, W_t is mass at time t , W_∞ is residual mass, R is universal gas constant, T is temperature, β is heating rate. To evaluate the above equation, a plot of LHS of the above equation for various order of thermal degradation was plotted with respect to $1/T$. Suitable order of thermal degradation was found out by applying linear fit and from the resulting equation which gives best correlation of regression was taken into account for the estimation of activation energy and pre exponential factor. The same

procedure was followed for all the nanocomposites and the activation energy and the order of thermal degradation was compared. The plots are displayed in Figure 6 and the obtained parameter values are presented in Table 3. It was observed that activation energy increased with increasing BN loading, which is also good agreement with the increased in thermal stability as in Figure 5.

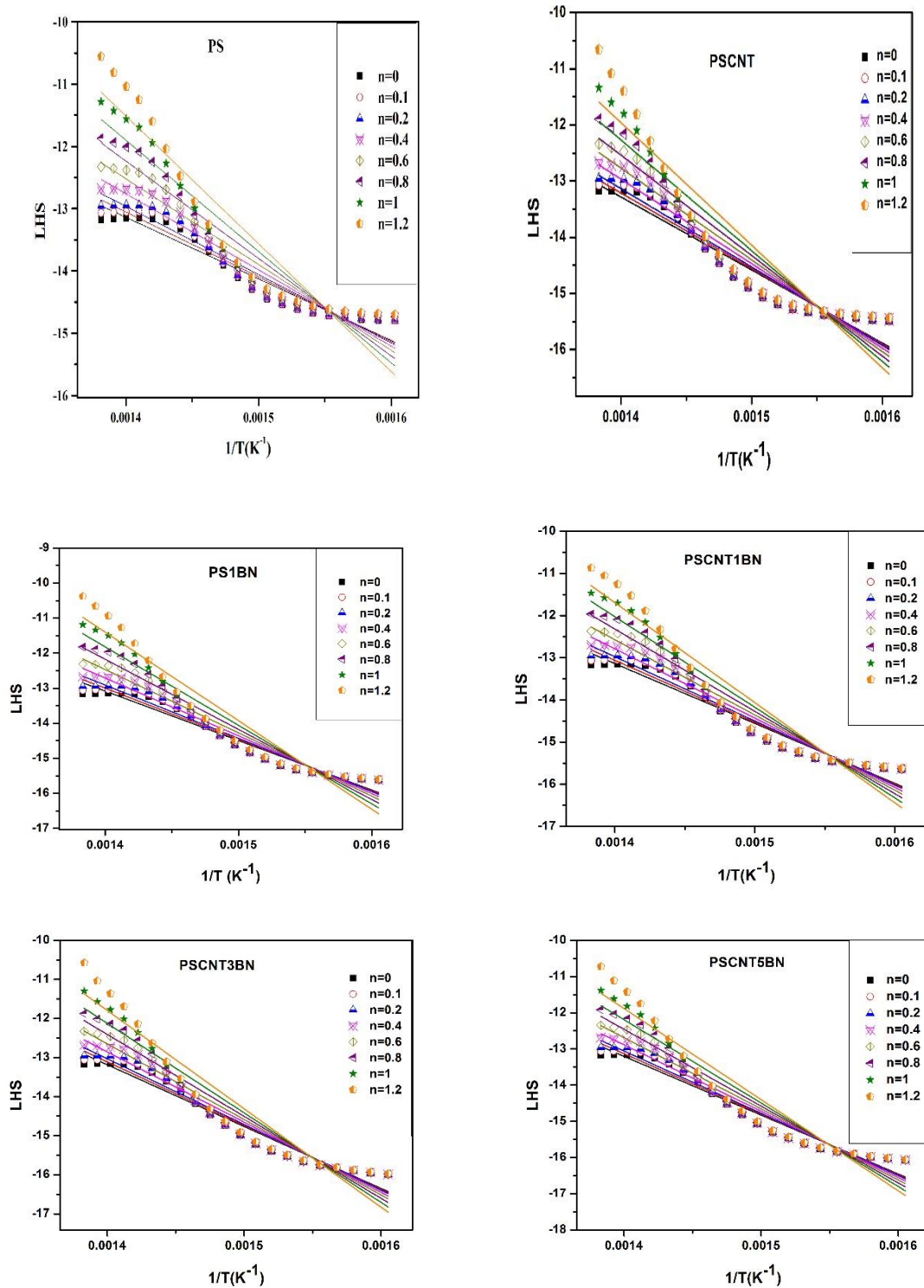


Figure 6: Degradation kinetics of nanocomposites.

Table 3: Kinetic parameters obtained from Coats-Redfern method for nanocomposites.

Sample	Order of reaction, n	Activation energy, E (KJ/mol)	Correlation coefficient, R ²	IPDT (°C)
PS	0.1	86	0.94	382
PSCNT	0	107	0.92	414
PS1BN	0.4	137	0.95	415
PSCNT1BN	0.2	126	0.94	433
PSCNT3BN	0	131	0.95	460
PSCNT5BN	0	135	0.95	470

Analysis of thermal degradation mechanism

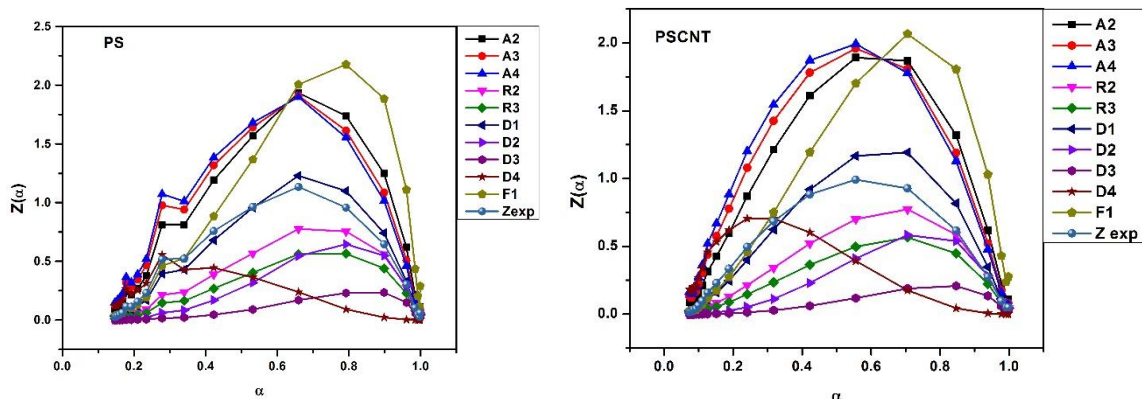
In order to determine the solid state reaction mechanism, which best describes the degradation process, Criado et al., [12] proposed the following governing equations.

$$Z(\alpha) = \frac{\beta}{A} G(\alpha) \frac{d\alpha}{dT} e^{\frac{E}{RT}} \tag{6}$$

$$Z(\alpha) = \frac{d\alpha}{dT} \frac{E}{R} e^{\frac{E}{RT}} \left(\frac{e^{-x}}{x} \frac{x^3 + 18x^2 + 86x + 96}{x^4 + 20x^3 + 120x^2 + 240x + 120} \right) \tag{7}$$

where, $x = \frac{E}{RT}$, $Z(\alpha)$ is the Criado function and $G(\alpha)$ is the solid-state mechanism. In this equation, we substituted the activation energy obtained from Coats-Redfern method. $G(\alpha)$ for different mechanism were utilized from literature [12]. The plot of eq. (6) for different mechanism was compared with eq. (7) with respect to conversion α , to know the reaction mechanism of the nanocomposite as shown in Figure 7.

It was observed from Figure 7 that initially the thermal degradation mechanism follows A4 (Nucleation and growth: Avrami equation3) and after that, the mechanism was shifted to D1 (one-dimensional diffusion) for PS for conversion greater than 0.6. For the entire nanocomposite samples, it was observed that the thermal degradation mechanism followed A3 (Nucleation and growth: Avrami equation 2) and at higher conversion ($\alpha > 0.6$), the mechanism was shifted to D1 (one-dimensional diffusion).



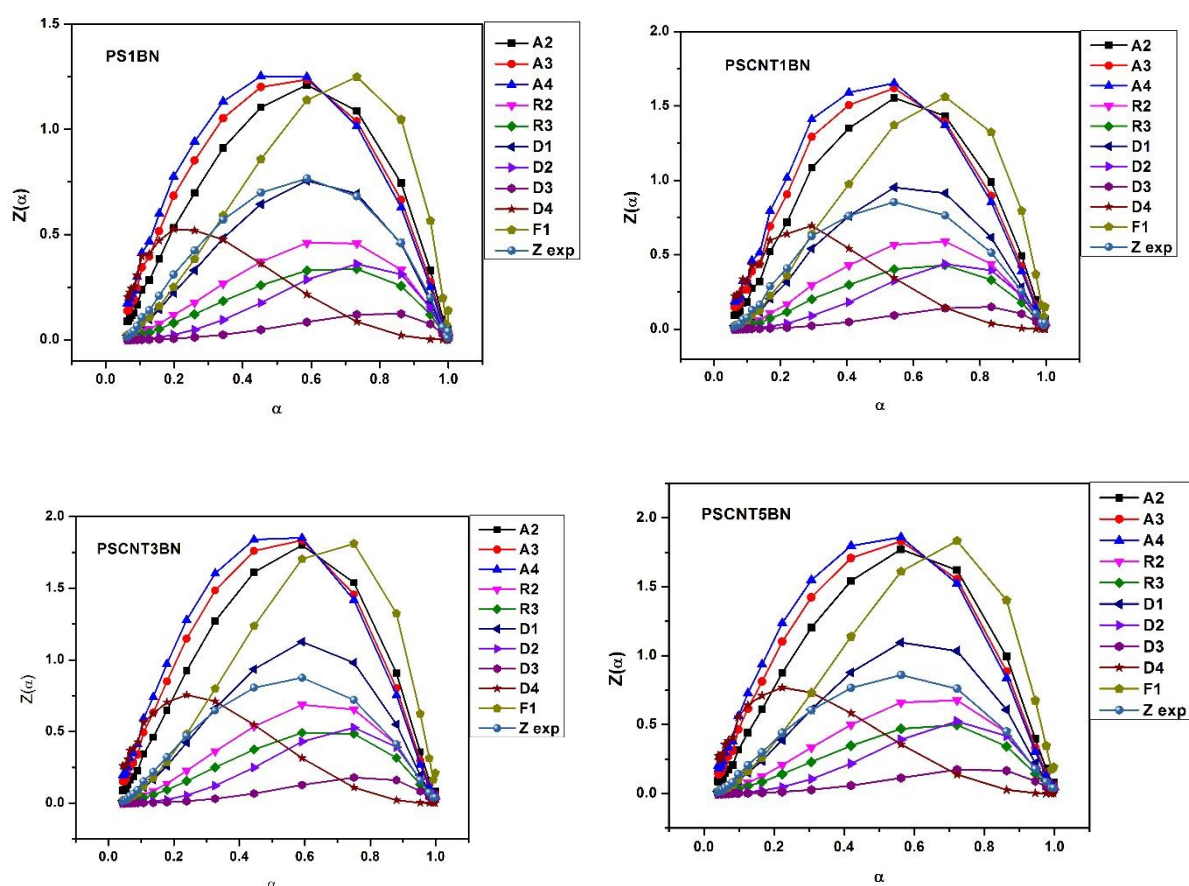


Figure 7: Thermal degradation mechanism using Criado method for nanocomposites.

Integral procedure decomposition temperature (IPDT)

Doyle [12] proposed a method to estimate the inherent thermal stability of polymeric materials by correlating with the volatile parts of polymer nanocomposites. IPDT takes into account the whole shape of the thermogram and places all the materials on an equal procedural basis, the curve was integrated between temperature ranges of 30 to 700 °C for all the nanocomposites. The IPDT was calculated using the procedure reported elsewhere [12] and the values obtained were consistent with that of activation energy calculated using Coats-Redfern method. The IPDT values after incorporation of nanofillers were found to increase. It was found to be highest for PSCNT7BN by 61.41°C in comparison with PS (see Table 3).

Melt rheological analysis

Melt rheological analysis was performed in an attempt to know the impact of interaction of dual fillers on various rheological properties which were not performed for this system using oscillatory test in literature so far. At the beginning of the test, the thermal stability of nanocomposite at 190°C was determined to avoid the effect of degradation during analysis. The parameters that influence the property of melt rheology are strain, angular frequency and temperature. The temperature was maintained at 190°C. Strain sweep was performed to determine a strain from viscoelastic region that has to be kept constant, to perform amplitude sweep [23].

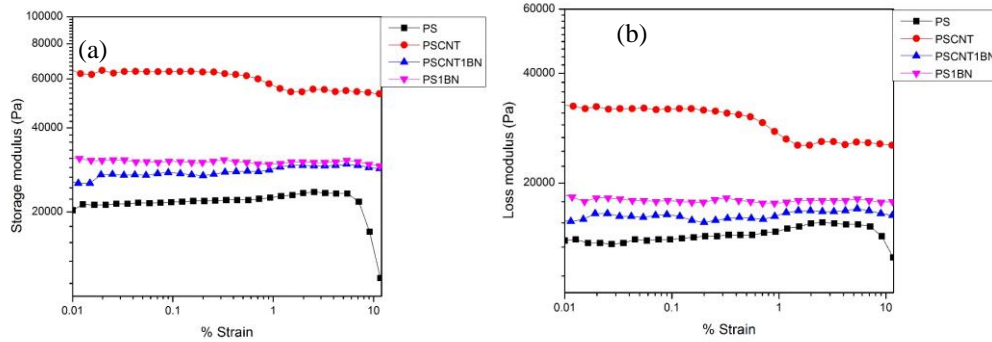


Figure 8: A plot on (a) storage modulus and (b) loss modulus Vs % Strain.

Strain sweeps were performed at various strains, keeping the angular frequency and the temperature constant. Here, the strain sweep was conducted at 190 °C and 100 rad/s and the values of storage and loss modulus were analyzed. To choose the appropriate strain from the viscoelastic region, the linear region of the plot was considered as shown in Figure 8, and it was observed that around 0.1% strain, both storage and loss moduli were constant.

Frequency sweeps were conducted at 0.1% strain and 190 °C, to know the viscoelastic behavior of the nanocomposite i.e., sample exhibits gel character if $G' > G''$ and liquid character if $G'' > G'$. Figure 9 shows the variation of rheological parameters with angular frequency from 1-100 rad/s for dual filler nanocomposites only. Storage modulus G' [Pa] and loss modulus G'' [Pa] give us information regarding the deformation energy stored and lost by the sample. With an increase in BN loading, it was observed that, the cross over frequency ($G' = G''$) shifted to lower frequency, which symbolizes that BN imparts stiffness to the nanocomposites. It was also observed that increasing BN loading increases the viscosity of the nanocomposites as BN's presence hinders the movement of Polystyrene.

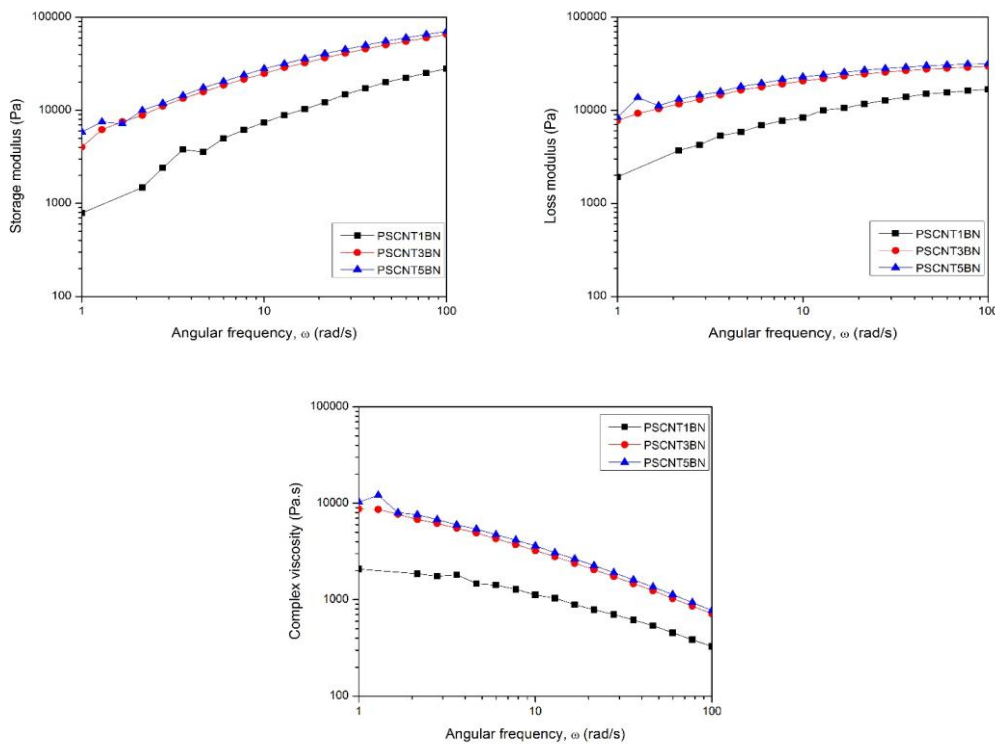


Figure 9: A plot on storage modulus, loss modulus and complex viscosity Vs Angular frequency.

CONCLUSION

The present work discussed the impact on BN loading on the thermal and rheological properties of nanocomposites, which were prepared using solvent blending method assisted with sonication. The XRD and TEM analysis gave us a clear picture of BN nature on the nanocomposites. The addition of BN to nanocomposites increased the activation energy of thermal degradation, which was consistent with the IPDT values. Rheological study revealed that addition of BN increased the stiffness and complex viscosity of nanocomposites.

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