

Mechanical, Dynamic Mechanical, Morphological, Thermal Behavior and Processability of Polyaniline and Ethylene 1-Octene based Semi-conducting Composites

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ABSTRACT: The polyaniline (PAni) was prepared by the oxidative emulsion polymerization of aniline. Different semi-conducting composites were prepared by mixing this PAni with a polyolefinic thermoplastic elastomer namely ethylene 1-octene copolymer (EN) with different ratio. The effect of composition on different mechanical, dynamic mechanical, and thermal properties of these composites and their processability were investigated. The results revealed that the increased incorporation of PAni in EN caused increase in the modulus, hardness, glass transition temperature (T_g), and apparent shear viscosity, but caused decrease in the tensile strength, elongation at rupture, damping character, and die-

swell during injection molding. The base matrix is thermoplastic elastomer for which vulcanization is not required. However, it is found that if such composites are subjected to electron beam (EB) radiation, they can undergo some cross-linking. The introduction of crosslinks to such system through EB radiation improves mechanical properties. The flow behavior shows the material is pseudo-plastic in nature. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 107: 2486–2493, 2008

Key words: activation energy; composites; conducting polymers; electron beam irradiation; thermogravimetric analysis (TGA)

INTRODUCTION

The main drawbacks of effective utilization of intrinsically conducting polymers alone lie in the fact that these materials are difficult to process because of their thermosetting nature, poor solubility and also for poor thermal stability. The composites of intrinsically conducting polymers, where they are used as conducting filler with other insulating polymer (matrixes) have received attention because such system can find practical applications as conducting composites. These composites may be used for different electronic or electrical applications like devices for electrostatic charge dissipation, touch control switches, electromagnetic interference shielding material, pressure sensitive sensors, solar cells.^{1–5} Among the intrinsically conducting polymers, extensive study on polyaniline (PAni) has been carried out because of its ease of synthesis, novel properties, and potential applications.^{6–14} While using PAni particles as a conducting filler in insulating rubber matrix, these are mixed with rubber along with other ingredients like curatives, anti-degradants, colorants and finally vulcanized to achieve good mechanical properties. But dopant (usually

acids) used in PAni adversely affects sulphur or peroxide curing of rubber matrix.⁵ If a thermoplastic elastomer (TPE) is used as an insulating matrix instead of a rubber, then the need for curing may be avoided and processability will be easier compared with that of conventional elastomers. TPEs are polymers that behave like an elastomer but can be processed and again reprocessed like a plastic. These materials in general are two phased (hard and soft segments) system. The hard segment is responsible for processability (melt process) and the soft segment is responsible for elastomeric properties. The flow behavior of TPE depends on the melting point of the hard segment. Thus upper and lower usable temperature limits depends on the melting point of hard segment and glass transition temperature of soft segment. Recently commercialized ethylene 1-octene copolymers (EN) are interesting polyolefinic elastomers. They can be elastomers or TPEs depending on the 1-octene content. Engage 8150 is one of the commercial grades of EN having a melting point 58 C. It can be easily processed over the temperature range of 60–70 C, and which is safe for PAni processing. In this temperature range, PAni remains almost unaffected. If one chooses TPE having very high softening temperature, then the PAni may undergo some chemical change during melt processing of TPE, which may adversely affect its conductivity. Literature dealing with the use of

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TPE specially Engage 8150 as the insulating matrix for the conducting composite with PAni is very scarce.

The present work is based on the usage of TPE (Engage 8150, abbreviated here as EN) as the insulating matrix for the semi-conducting composite, where PAni was used as conducting filler. Different properties of these composites were measured as such as the matrix is thermoplastic in nature. However, to check the effect of cross-linking on different properties of such composites, only composite having the highest PAni concentration (60 phr) was subjected to EB curing and properties were measured for this composite.

This article includes mainly mechanical, dynamic mechanical properties, and thermal degradation behavior of different PAni filled EN composites designated as EN-P in general. For use as an electrical and electronic material, composites should have adequate mechanical properties and thermal stability. Processability of these composites was also studied, which is important for product fabrication. Morphological studies were made through scanning electron microscopy (SEM) to understand how different properties are affected through filler-matrix interaction.

EXPERIMENTAL

Materials

Aniline, ammonium peroxy disulphate (APS), ammonia solution (S.D. Fine Chem., Mumbai, India), sodium dodecyl sulphate (SDS), cetyl alcohol (Loba Chemie, Mumbai, India), methanol (SISCO Research Laboratories, Bombay, India), hydrochloric acid (Ranbaxy, India), EN with 25% 1-octene content, commercial name Engage 8150 (Dupont Dow Elastomer, USA), abbreviated in this report as EN. All are GR grade.

Synthesis of PAni

In a typical reaction procedure 3.0 g SDS, 10.0 g cetyl alcohol, and 1.0 L distilled water were taken in a beaker, stirred for 1 h with high-speed mechanical stirrer to form the mini-emulsion. Emulsion formed by the use of surfactant (SDS) and cosurfactant (cetyl alcohol) in the molar ratio of 1 : 4 (M/M) is more stable and reduces the size of the emulsion droplets compared with that of normal emulsion.^{6,15} To this miniemulsion, 25.0 mL aniline (freshly distilled) and 25.0 mL HCl were taken and polymerization reaction was initiated by dropwise addition of aqueous solution of APS (50.0 g APS in 500 mL distilled water) and carried out for 6 h at room temperature with constant stirring. The polymerization reaction was terminated through the addition of 50 mL methanol. The mass was then filtered, washed and finally dried

in a vacuum oven at room temperature. The DC conductivity of this PAni is 1.3×10^{-3} S/cm.

Preparation of EN and PAni composites

The PAni was mixed with EN in different ratio in a Brabender Plastograph (D-47055 Duisburg, Germany) at 70 C at a speed of 30 rpm for 10 min. The composites were converted into thin sheet using Moore press (George E Moore & Sons, Birmingham) at 70°C under pressure of 8 psi. The composite containing the maximum amount of PAni was cured by 15 Mrad of electron beam (EB) irradiation (Dynamitron, available in NICCO Cable Corporation, Shyamnagar, India). The composition of the composites and their designation are presented in Table I.

Properties measurement

The morphological study was carried out using SEM, JSM 5800, JEOL, JAPAN. All samples were gold-coated prior to scan.

The mechanical properties of different samples were evaluated using Hounsfield - H10KS, UK as per ASTM D 412.

The hardness was measured as per ASTM D 2240 by Durometer type A (Shore Instrument & MFG., USA).

The dynamic mechanical analysis (DMA) was carried out using a TA Instruments model DMA 2980 Dynamic Mechanical Analyzer over the temperature range from -100 to 40°C at the heating rate $3^\circ\text{C}/\text{min}$ at the frequency 1 Hz with amplitude $20 \mu\text{m}$.

The thermogravimetric analysis (TGA) was carried out under N_2 atmosphere from room temperature to 700°C at the heating rate $20^\circ\text{C}/\text{min}$ (TGA, Q50, TA Instruments, USA).

The differential scanning calorimetric (DSC) study was carried out under N_2 atmosphere from -80°C to 350°C at the heating rate $10^\circ\text{C}/\text{min}$ (DSC, Q100, TA Instruments, USA).

The melt flow behavior of pure EN and EN-PAni composites were examined using Monsanto Processability Tester (MPT-85077, USA). The flow behavior was carried out at four different temperatures (90,

TABLE I
The Composition and Designation of Different Engage-PAni Composites

Ethylene 1-octene copolymer (EN) in wt %	PAni in wt %	Vulcanized by EB radiation	Composite designation
100	0	No	EN
100	20	No	EN-P (20)
100	40	No	EN-P (40)
100	60	No	EN-P (60)
100	60	Yes	EN-P (60)-EBR

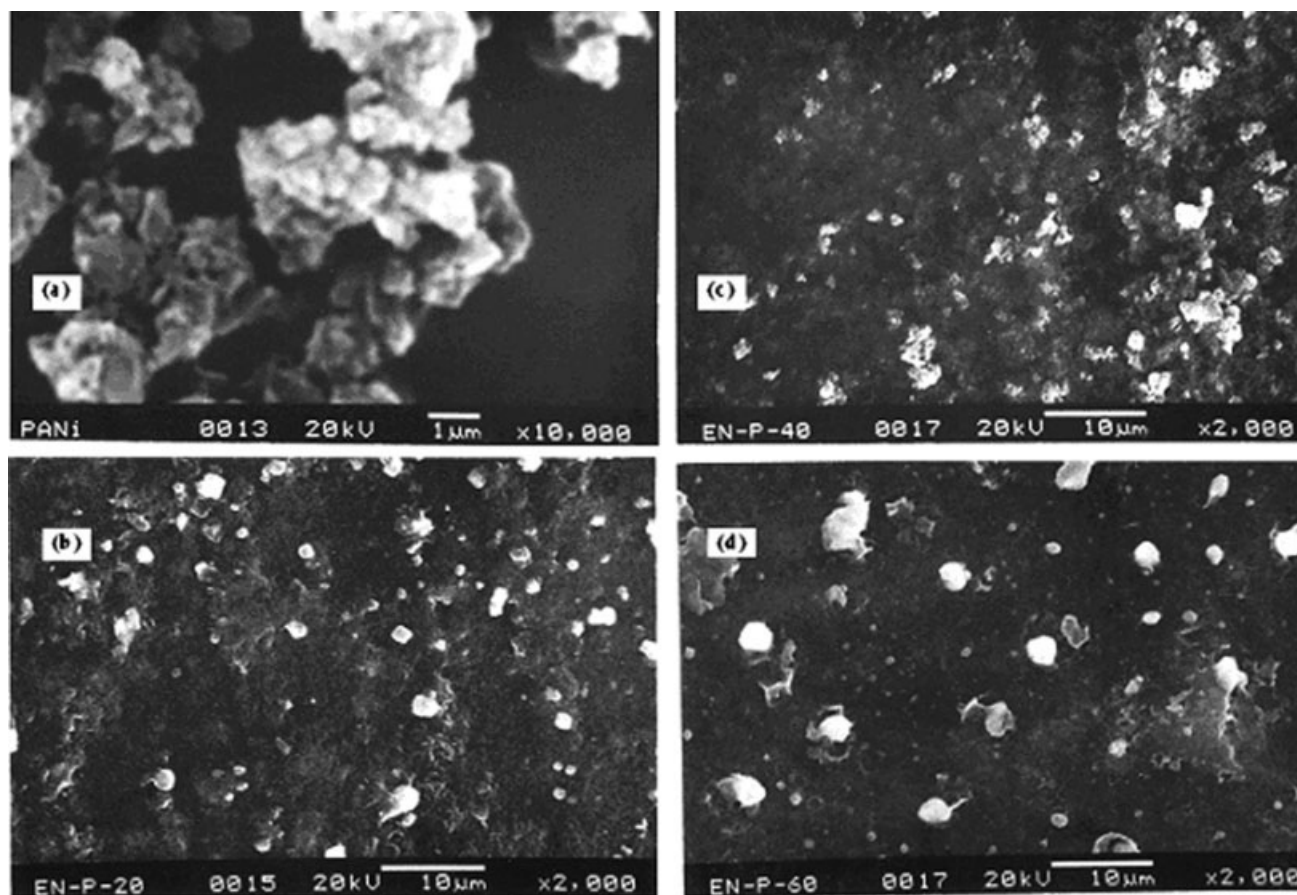


Figure 1 SEM picture of (a) PANi, (b) EN-P (20), (c) EN-P (40), (d) EN-P (60).

100, 110, and 120°C) and at four different shear rates (61.3, 122.6, 306.5, and 490.4 s^{-1}) in the instrument limitation range.

RESULTS AND DISCUSSION

Morphology

The morphology for any composite is very important to understand the filler (dispersed phase) distribution in the matrix (continuous phase) because it is the most important aspect, which governs different physical and mechanical properties.^{16,17} SEM pictures of pure PANi and engage-PANi (EN-P) composites are presented in Figure 1. The Figure 1(a) (top left) for neat PANi reveals that PANi particles exist in clustered or aggregated form. The Figure 1(b) is for EN-P (20) composite (top right) where PANi particle distribution is found to be fairly good, however a few lumps are found to exist in matrix polymer. The Figure 1(c) is EN-P (40) composite (bottom left) where PANi particles have tendency to form some scattered agglomerate and the Figure 1(d) is EN-P (60) composite (bottom right) where agglomeration tendency of PANi particles is more and agglomerated particle size is also greater compared with previous

composites. With the increase in PANi concentration in EN, the tendency of agglomeration is increased with increased agglomerate size. This may be due to the difference in polarity (PANi being highly polar and EN being nonpolar in nature) between EN and PANi as well as poor filler-polymer interaction.^{18,19}

Mechanical properties

The stress-strain curves for different composites and neat polymer are presented in Figure 2. With the increase in PANi concentration both stress (tensile strength) and strain (elongation) at rupture decreases slowly. This indicates PANi is not reinforcing filler for matrix EN. Although not acting as reinforcing filler for the matrix, PANi does not deteriorates different mechanical properties like other nonreinforcing fillers.

The progressive addition of PANi to some extent increases the modulus of the composites as PANi has higher modulus compared with the matrix polymer, EN (Table II). The marginal decrease in elongation at break and increase in modulus with increase of PANi concentration is also due to the restricted mobility of the polymer chain imposed by the filler

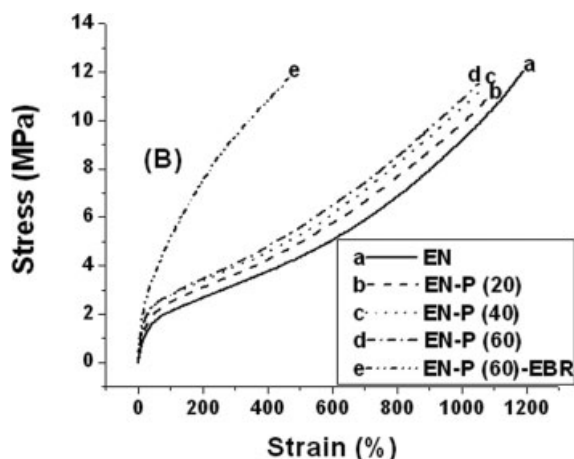


Figure 2 Stress-strain curve of EN-PANI composites.

(PANI particles).²⁰ With the increase in modulus, hardness (very low strain modulus) is also expected to increase. The polymer matrix (EN) is thermoplastic-elastomeric in nature. As such thermoplastic elastomeric materials are self reinforced, common fillers rarely improves their strength properties. In the present case, PANi does not have good adhesion with the matrix polymer due to polarity difference. Consequently when stress is applied, there is hardly any effective stress transfer between the matrix and the filler.¹⁹ However, there is an increase in modulus, hardness and tensile strength after EB radiation treatment but large decrease in elongation at break. This may be caused by introduction of some crosslinks in the polymer matrix.²¹

DMA

The DMA of neat EN and its composites filled with different concentration of PANi are presented in Figure 3. At any particular temperature, the storage modulus increases with the increase in PANi concentration in EN. A sharp $\tan \delta$ peak is obtained at around -40°C for pure EN, which gets shifted towards higher temperature with increase in PANi concentration. These peak temperatures may be considered as the glass transition temperature, T_g (Table III) for pure matrix polymer, EN and filled composites.^{17,21} The peak height also reduces with the

increase in PANi concentration, which signifies that the damping characteristic decreases with increased PANi concentration. This is mainly because PANi is harder than matrix polymer EN. The effect of EB radiation is further increase of storage modulus and T_g and reduction of damping behavior, due to restriction of chain mobility after crosslink formation. This increase in storage modulus (very low strain modulus) and T_g and decrease in damping characteristics is mainly due to the restriction of EN chain mobility due to increase of rigid filler (PANi) concentration and formation of crosslinks after EB radiation.

TGA

The weight loss vs. temperature plots (Fig. 4) show that pure polymer is thermally stable up to the temperature 380°C , from 380°C decomposition starts and is completed at around 480°C , when the weight loss is 100%. This shows that the polymer is completely decomposed without leaving any residue at 480°C . This is expected as EN is purely a hydrocarbon based polymer. But the composites containing PANi show gradual weight loss starting from 100°C up to 380°C , and beyond 380°C there is an abrupt loss of weight due to the decomposition of the total composite.²²⁻²⁴ However, there is almost no change in the decomposition temperature of EN when PANi is added to it. But a residual weight of the composites is observed at 480°C , which does not change even on further heating up to 700°C . This residual weight increases with the increase in PANi concentration in the composite. So, it clearly shows that this residual weight is entirely due to PANi. PANi shows some residual weight even after heating to 1000°C in nitrogen atmosphere. These residual materials are mainly thermally stable carbonized materials.²⁵ The initial small weight loss up to 380°C for the composites (not found for pure EN) is mainly due to moisture, bound water and HCl as PANi is hygroscopic and HCl is used as initiator and dopant during synthesis.^{7-9,26,27} So, with the increase in PANi concentration as the relative quantity of PANi is increased, the initial weight loss is also increased, as well as left over residue at the end of experiment at 700°C .

TABLE II
Hardness, Modulus, Tensile Strength (TS), and Elongation at Break (EB) of EN and Its Composites

Sample	Hardness (Shore A)	Mod at 100% elongation (MPa)	Mod at 200% elongation (MPa)	Mod at 300% elongation (MPa)	TS (MPa)	EB (%)
EN	57	2.1	2.7	3.2	12.0	1190
EN-P (20)	61	2.5	3.1	3.7	10.9	1080
EN-P (40)	65	2.8	3.4	4.0	11.4	1065
EN-P (60)	67	2.8	3.5	4.1	11.5	1050
EN-P (60)-EBR	70	5.2	7.5	9.3	11.7	460

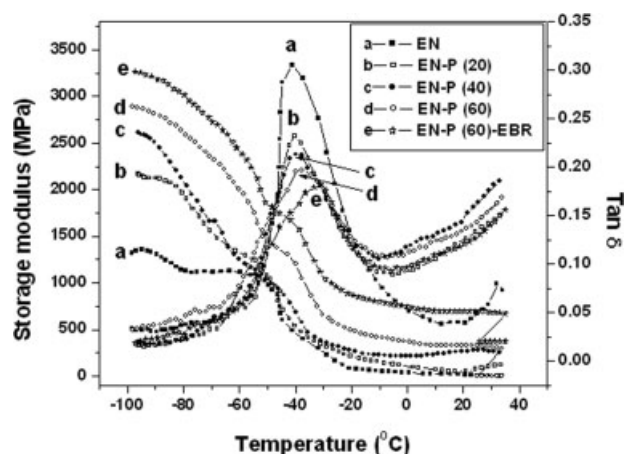


Figure 3 Storage modulus and $\tan \delta$ as a function of temperature for EN-P composites.

DSC study

Figure 5 represents the DSC curve of EN and its composites. EN shows one endothermic peak, en1 [Fig. 5(a)] is due to melting of the hard crystalline phase of EN,¹⁶ which acts as physical crosslink in TPE. However, all uncured composites show three endothermic peaks (en1, en2, and en3). The endothermic peak en2 may be for the evaporation of moisture or HCl (dopant). It is stated earlier, PANi is highly polar and absorbs some moisture during storage.^{28,29} This peak is absent for pure EN, which is purely a hydrocarbon and consequently hydrophobic in nature. The peak en3 may be for crystalline melting of PANi. The changes in peak area also support our argument. With the increase in PANi concentration the peak area that means enthalpy of en1 is found to decrease as the relative proportion of EN is decreased in the composite. But the peak areas for en2, en3, are found to increase as the relative concentration of PANi is increased in the composite. The en2 and en3 peaks are found to be very weak in EB radiation cured sample [Fig. 5(e)] indicating evaporation of moisture and/or HCl as well as melting of some PANi crystals during EB radiation treatment.

Some observations about transient heat flow behavior through different composites may be made from their DSC plots using almost identical sample weight.

TABLE III
Tan δ Peak Temperature or Glass Transition Temperature (T_g) of Engage and Its PANi Composites Obtained from DMA Results

Sample	Tan δ peak/ T_g (°C)
EN	-41
EN-P (20)	-40
EN-P (40)	-40
EN-P (60)	-38
EN-P (60)-EBR	-33

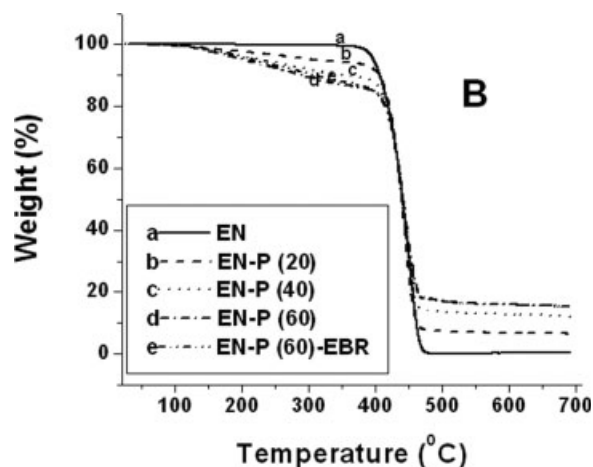


Figure 4 TGA analysis of EN-P composites.

The relative value of heat flow at different temperature through different composites may be considered as their temperature dependent or transient thermal conductivity (TDTC). The Figure 5 shows that the heat flow value is almost linear over the temperature range from -70 to 20°C . So, the TDTC of the composites in this temperature range may be compared. But the values of heat flow in Figure 5 is in arbitrary scale, all curves were arranged sequentially for better visualization and understanding. The true value of heat flow or TDTC at the temperature range from -70 to 20°C is shown in Figure 6. Curves for different composites show that with the increase of PANi concentration TDTC value is increased. However, it is expected because better electrical conductors, are also better thermal conductor like metals when compared with ceramics. The PANi is conductive in nature and hence with the increase in concentration of PANi, the TDTC of composites is increased. Some increase in TDTC after EB curing is also observed, this may be

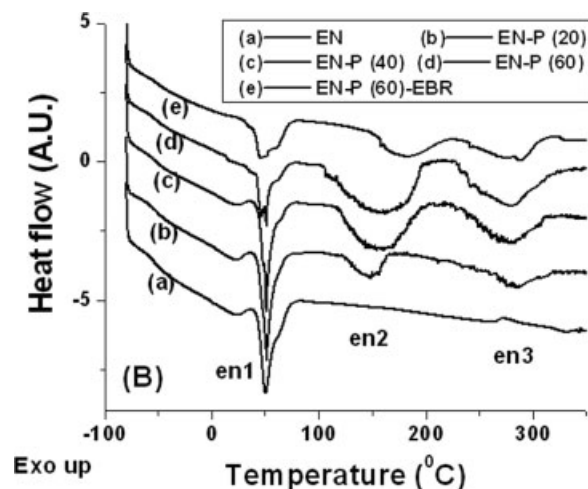


Figure 5 Heat flow as a function of temperature for EN-P composites.

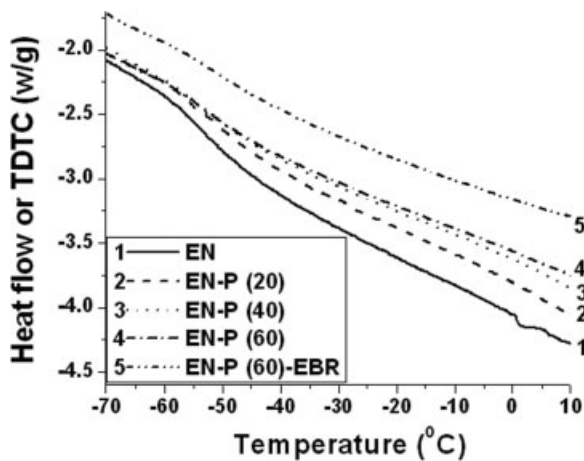


Figure 6 Transient heat flow value as a function of temperature for different composites.

due to some arrangement of PAni particles in polymer matrix during curing reaction.

Processability and flow behavior of EN-PAni composites

In Monsanto processability tester sample is placed in the electrically heated cylindrical barrel capillary having different L/D ratio, a definite force is applied by a piston to move the heated sample through a capillary. The processability parameters are as follows.^{30–32}

$$\gamma_a = \frac{32\pi R_b^2}{\pi d_0^3} \times S \quad (1)$$

$$\tau_a = \frac{d_0 P}{4L_0} \quad (2)$$

$$\eta_a = \frac{\tau_a}{\gamma_a} \quad (3)$$

$$\% \text{ Die Swell} = \frac{E - 0}{0} \times 100 \quad (4)$$

Where γ_a is the apparent shear rate in s^{-1} , τ_a is the apparent shear stress in Pascal, η_a is the apparent shear viscosity in Pa s, R_b is the barrel radius, d_0 is capillary diameter, S is the linear piston speed, P is the barrel pressure, L_0 is the capillary length, E is the diameter of the extrudate, and 0 is the diameter of the capillary. During the experimentation, temperature, applied stress, L/D ratio of the capillary can be changed. The change in dimension of extruded sample just after coming out of the barrel gives an idea about the die-swell.

The detailed discussion on rheological properties, their influence on different flow behavior, relationship among these rheological properties, their calculation and also the calculation of different constant defining the flow characteristics were discussed elsewhere.^{30–32}

Figure 7 represents the change in apparent shear viscosity (η_a) and running die-swell as a function of apparent shear rate (γ_a) for EN and its composite at 90°C . The effect of temperature on these two parameters was studied for EN-P (60) containing the highest PAni concentration. All the results show that with the increase in γ_a , η_a is decreased indicating that EN and its composites exhibit pseudo-plastic or shear thickening behavior and are expected to follow the Power Law model.³⁰ At a particular temperature and at any particular shear rate, with the increase in PAni concentration η_a is increased marginally [Fig. 7(a)] but the decrease of running die-swell is very high [Fig. 7(b)]. This increase in viscosity and decrease in running die-swell with loading may be due to occlusion of EN in PAni aggregates and hence there is increase in the hydrodynamic volume.

The flow behavior index, n and consistency index, k were calculated by using the Power Law equation given below³⁰

$$\begin{aligned} \eta_a &= k\gamma_a^{n-1} \\ \text{or} \\ \log \eta_a &= \log k + (n-1)\log \gamma_a \end{aligned} \quad (5)$$

The values n and k ; were calculated after linear regression analysis of $\log \eta_a$ vs. $\log \gamma_a$ plot. The decrease of consistency index (k) of EN with PAni loading (Table IV) indicates lack of interaction between them and flow behavior index value ($n < 1$) indicates that the EN and its composites show pseudo-plastic behavior.³⁰ With the increase in experimental temperature, viscosity decreases marginally [Fig. 7(c)] but die-swell increases substantially [Fig. 7(d)]. This may be due to the increase in ease of flow due to decrease in EN-P interaction and there is also possibility of decrease in occlusion of EN in PAni aggregates. The decrease in the magnitude of consistency index (k) temperature also corroborates this argument (Table IV).

The activation energy of viscous flow of EN-P (60) was derived from the Arrhenius–Frenkel–Eyring relation as given below³⁰

$$\begin{aligned} \eta_a &= B e^{E_\gamma/RT} \\ \text{or} \\ \log \eta_a &= \log B + \frac{E_\gamma}{RT} \end{aligned} \quad (6)$$

Where, E_γ is the activation energy of the flow at a particular shear rate, R is the universal gas constant,

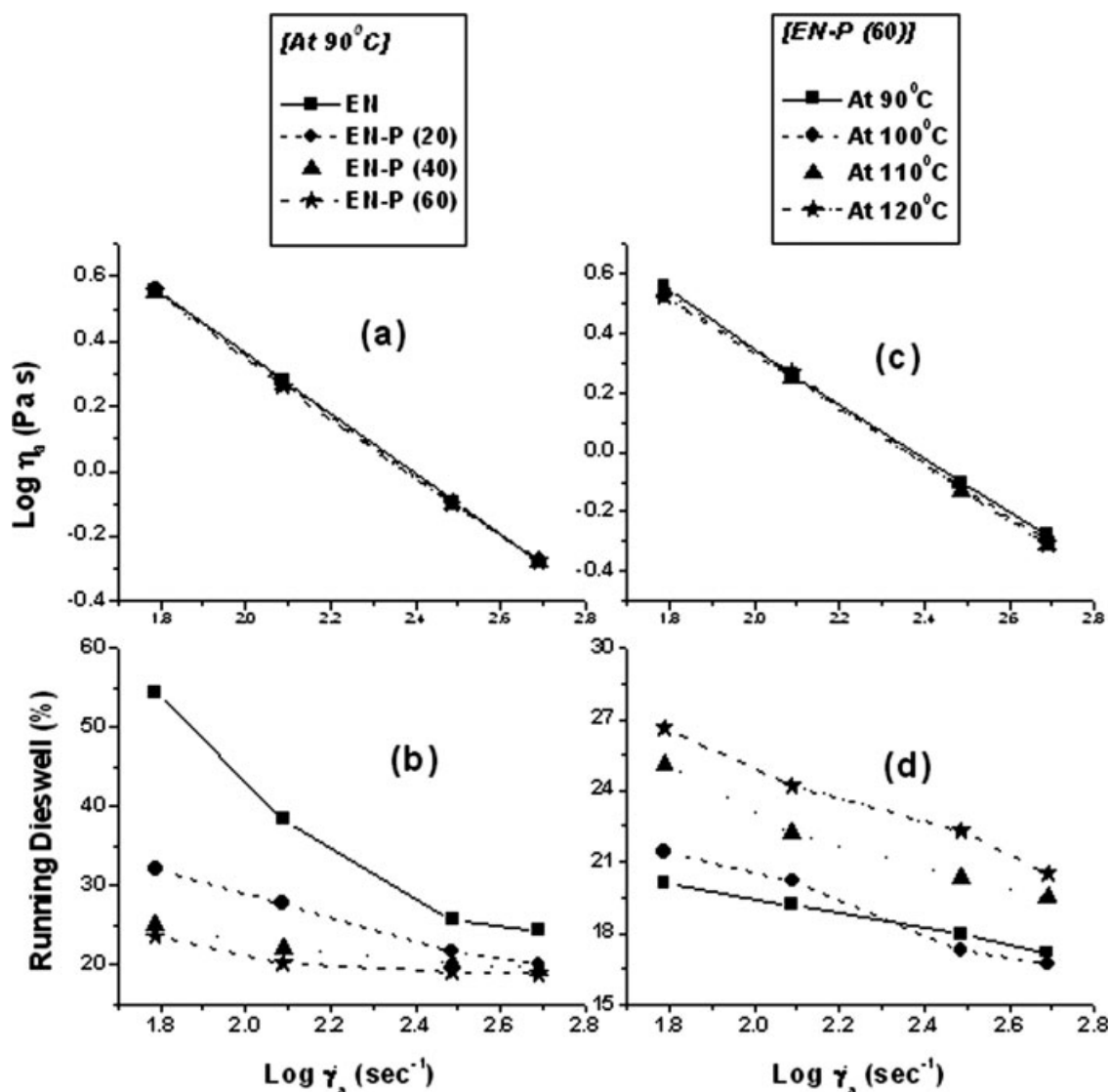


Figure 7 Change in (a) apparent shear viscosity ($\log \eta_a$) in Pascal second (Pa s) and (b) running die-swell in % as a function of apparent shear rate ($\log \dot{\gamma}_a$ in s^{-1}) EN, EN-P (20), EN-P (40), EN-P (60) at constant temperature (90°C), whereas (c) and (d) are the similar parameter changes against apparent shear rate for EN-P (60) at different temperature.

T the temperature in Kelvin, and B is the pre-exponential component. From the slope of the linear plot of $\log \eta_a$ vs. $(1/T)$, values of E_a/R are obtained from which the activation energies can be calculated.

TABLE IV
Flow Behavior Index, n and Consistency Index, k of EN and Its PANi Composites Obtained from the Linear Regression of $\log \eta_a$ vs. $\log \dot{\gamma}_a$ Plots Using Power Law

Sample	Experimental temperature (°C)	k (Pa s n)	n
EN	90	168	0.07
EN-P (20)	90	161	0.08
EN-P (40)	90	156	0.08
EN-P (60)	90	159	0.08
EN-P (60)	100	142	0.07
EN-P (60)	110	144	0.05
EN-P (60)	120	153	0.07

The activation energy of viscous flow of EN-P (60) as a function of shear rate is presented in Figure 8. Lower activation energy indicates better flow behavior. With the increase in shear rate the activation energy is found to decrease and this may be due to the orientation of the molecular segments in the direction of applied stress as well as increased wall slippage.³⁰

CONCLUSION

When PANi is added as conductive filler in EN, there are marginal drops in mechanical strength, elongation at break but increase in hardness and high and low strain modulus. There is also reduction in damping behavior and increase in glass to rubber transition temperature (T_g) compared with base

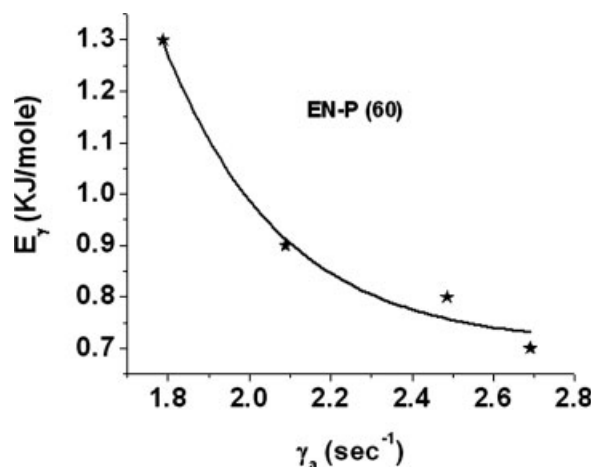


Figure 8 Activation energy of viscous flow (E_γ) of EN-P (60) as a function of shear rate (γ_a).

polymer. All these properties change with the increase in PAni concentration are due to the restricted mobility of the polymer chain imposed by the filler. Although PAni is not acting as reinforcing filler for the matrix, it does not deteriorate mechanical properties like other nonreinforcing fillers. There is a marginal change in thermal decomposition temperature of base polymer due to addition of PAni. However, a part of decomposed PAni remains even after heating up to 700°C. Addition of PAni in Engage (EN) matrix increases viscosity but decreases running die-swell is due to occlusion of EN in PAni aggregates and hence there is increase in the hydrodynamic volume. EN-PAni composites behave like thermoplastic elastomeric composites. If such composites are subjected to EB irradiation, there will be some crosslinking. EB irradiation increases tensile strength, modulus, hardness but decreases elongation at break, T_g and damping behavior.

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