

Thermo-Mechanical, Rheological, Dynamic Mechanical And Tensile Properties Of Poly(3-Octylthiophene)/Polystyrene (P3OT/PS)

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Abstract— A series of poly(3-octylthiophene)/polystyrene (P3OT/PS) blends with different weight ratios of P3OT were prepared using casting technique. The tensile properties of these blends was studied and the elastic modulus, tensile strength and strain at break found to decrease with increasing P3OT content. In addition, the rheological properties including the dependence of complex viscosity, storage modulus on the oscillating frequency as well as the weight content of P3OT was also studied and both variables found to decrease with increasing P3OT. Dynamic mechanical properties were measured for the prepared blends. The results indicate that the storage modulus changed steadily, and the loss modulus peaks are broader and less intense for high P3OT content. The coefficient of linear expansion was calculated from thermo-mechanical analyzer curves and its values proved less interaction between the two polymers. This was also improved from the values of the glass transition temperature obtained from the loss modulus and $\tan\delta$ peaks beside those obtained from the thermo-mechanical analyzer curves.

Keywords— P3OT, PS, Mechanical properties

I. INTRODUCTION

Because of their wide applications in solid state devices such as field effect transistors, organic light emitting diodes, solar cells, batteries, super capacitors, biological and chemical sensors; conjugated polymers and their composites have stimulated much interest among physicists, chemists and material scientists. [1–4].

Since the discovery of the processing possibility of polyalkylthiophene, some studies have involved mixing poly (3-alkylthiophenes) P3ATs with various matrix or polymer. Jaczewska et al. [5] studied humidity and solvent effects in spin-coated poly (3-butylthiophene-2, 5-diyl) and polystyrene (PS) blends in chloroform, cyclohexanone and THF. Also they investigated PS blends with P3DDT, P3HT and P3BT spin-coated onto oxidized silicon surfaces from various common solvents [6]. Monedero et al. studied binary system P3OT+EVA by calorimetric and

dielectric study [7]. Kanemoto et al. studied the photoluminescence characteristics of poly (3-octylthiophene) P3OT diluted in a solid matrix of polypropylene [8]. Masegosa et al [9] investigated the thermal behavior of unsaturated polyester resins/P3OT blends. P3OT has a polar backbone (thiophene ring) and nonpolar side chain (octyl group) which is proved to be soluble in polar (chloroform, THF, etc.) or nonpolar (toluene, hexane, decalin, etc.) solvents providing a more flexible choice of matrix polymers that are either polar or nonpolar [10]. Nicho et al [11] studied the morphological and physicochemical properties of spin-coated P3OT/PS composite thin films and they found a classical percolation phenomenon in the electrical properties of these blends at concentrations of P3OT smaller than 5%. They, also, studied the surface morphology variation as a function of different P3OT concentration in PS and phase segregation was observed, and PS is shown to segregate to the surface of the films.

The study of the mechanical properties of polymer blends has made it desirable to choose these materials over traditional materials for numerous types of applications, such as binder constituents in explosives, load-bearing components, and jet engine modules. As the uses of polymer blends increase, an understanding of the mechanical behavior of these materials becomes vital for creating innovative and economical designs for various components. Polymer blends have more complicated properties as they display elastic and viscous responses at different strain rates and temperatures [12 – 15]. Stress transfer between the polymer matrix constituents strongly affects the strength of the blends [16]. However, for poorly bonded constituents, strength reductions occur [17].

The rheological behavior of polymer blends is of great importance in polymer processing, particularly for the analysis and design of processing operations, as well as understanding structure property relationships. The rheological behavior can realize the full potential of blends for application in thermoplastic matrix based polymer blends and to optimize the processing conditions for achieving high performance polymer blends [18].

This work aims to study the effect of blending poly (3-octylthiophene) with polystyrene with different weight ratio on the tensile, rheological, dynamical mechanical as well as the thermo-mechanical properties. In this respect, the effect of P3OT on the elastic modulus, storage modulus, complex viscosity, glass transition temperature and the coefficient of linear thermal expansion will be studied.

2. Experimental

2.1. Materials and preparation

Polystyrene (PS) grade EPS 452, density 250 kg/cm³ and average molecular weight 2500 was supplied from Sabic, Saudi Arabia in the form of beads with diameter ranging from 0.4 to 0.8 mm and was used in this work as a host polymer. Poly (3-octylthiophene-2, 5-diyl) (P3OT) was purchased from Aldrich (Ref. No. 445711). It has greater than 98.5% head-to-tail region-specific conformation, molecular weight 34,000, and melting point 190 °C. Both polymers were used as received.

Initially, desired amount of P3OT was dissolved in chloroform. The desired amount of PS was also dissolved in chloroform. The weight ratio of P3OT in PS was determined as follows. A fixed weights, w_p , of PS and w_c of P3OT were chosen such that the total weight was $w = w_p + w_c = \text{constant}$. Then, the required ratio, w_c/w_p , was determined by varying both w_c and w_p . Both solutions were mixed together using a magnetic stirrer for 24 h to form P3OT/PS solution blend. The final mixture was further sonicated to achieve best miscibility. The resulting solution was poured to petri dishes with fixed diameter of 7 cm and dried at room temperature. A series of blend films were prepared at different weight ratios (0, 1.6, 7.4, 10.7 and 16.7 wt %) of P3OT/PS. The film weight has to be very close to the fixed weight w to ensure the correct wt % of P3OT. The sample thickness was in the range of 50 – 60 μm .

2.2. Testing

All the mechanical tests in this study were carried out on a Dynamic Mechanical Analyzer DMA Q800 (TA Instruments LLC, Delaware, USA) instrument with film clamps. All the tests were repeated five times to ensure the reproducibility of the results.

Tensile tests

The tensile tests were carried out on film specimens. The measurements were done at constant temperatures of 27 °C and at a force rate of 3N/min. After clamping the sample the temperature was equilibrated at 27 °C, the instrument left isothermal for further 5 min and then the test was started.

Rheological and Dynamic Mechanical tests

The mentioned DMA Q800 instrument was used through the rheological and dynamic mechanical studies. For these tests a film-clamp was used in dry mode. A slow heating rate of 1 °C/min was employed

throughout to ensure that the sample was in thermal equilibrium with the instrument. The oscillating frequency was changed from 0.1 to 200 Hz.

Rheological tests experiments were performed with the film under tension while the frequency is changed. A static pre-load force (0.01 N) was applied to the sample prior the dynamic oscillating force to prevent film buckling.

During measurements, the instrument was programmed to maintain the static load at 125% of the force required to oscillate the sample. It is important that the film remained in its linear viscoelastic region during measurement (to ensure that the properties observed were independent of the deformation applied and truly reflected molecular motions), and so experiments were recorded maintaining constant strain. Generally, for thin polymer films, linear viscoelastic behavior can be assured with a strain less than 0.1%, and so this limit was used.

Coefficient of thermal expansion tests

These tests were carried out on film specimens. The measurements were done at constant applied force of 0.5 N. Change in sample length (strain is calculated automatically) was recorded while the temperature ramped at a constant rate of 3 °C/min in the temperature range from 30 to 150 °C using the DMA described above.

3. Results and discussions

3.1. Tensile properties

Figure 1 presents the stress strain curves for polystyrene loaded with 0, 1.6, 7.4, 10.7 and 16.7 wt % of P3OT at constant temperature of 27 °C. All blends show straight lines relations between stress and strain in the low strain range (shown in the insert). This reflects the Hookean behavior of these composites at these low strains. This linear Hookean regions at low strains are used to calculate the elastic modulus, E , for all composites. Figure 2 plots a relation between the calculated values of the elastic modulus and the weight percent of P3OT content at 27 °C. The elastic modulus is found to decrease exponentially with increasing P3OT content according to the empirical equation (1). Both the tensile strength and the strain at break, shown in Figure 3, are deduced from the stress strain curves for all blends and found to decrease exponentially according to equation 1, too, with increasing P3OT content.

$$\psi = \psi_o + \psi'_o \exp(-KC) \quad (1)$$

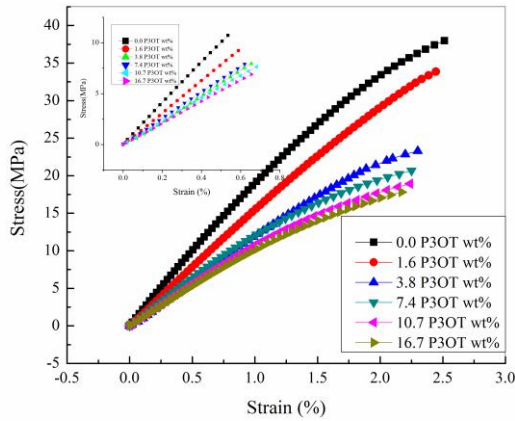


Figure 1: Stress–strain curves for PS loaded with different concentrations of P3OT at 27°C.

where C is the concentration of P3OT. When the equation describes the elastic modulus $\psi = E$ and $\psi_o = 11.31 \text{ MPa}$, $\psi'_o = 8.99 \text{ MPa}$, $K = 2.03$ are constants. For the tensile strength, $\psi = \sigma_b$ and $\psi_o = 17.43 \text{ MPa}$, $\psi'_o = 21.40 \text{ MPa}$, $K = 3.81$, while for the strain at break, $\psi = \varepsilon_b$ and $\psi_o = 2.81 \%$, $\psi'_o = 0.34\%$ and $K = 4.99$.

The blends can be considered to be consisting of hard phase (PS) and soft one (P3OT) [19]. At low strains, both of these phases are coiled like springs and entangled in two different poly-domain structures with PS poly-domains harder than P3OT ones. This appears in the high values of the elastic modulus, tensile strength and strain at break of pure polystyrene sample. As the P3OT content increased, its poly-domain volume increased over those of PS and results in a decrease in the elastic modulus, tensile strength and strain at break values.

3.2. Rheological properties

The complex viscosity, η^* , for PS loaded with 0, 1.6, 7.4, 10.7 and 16.7 wt% of P3OT as a function of frequency at constant temperature of 27 °C is shown in

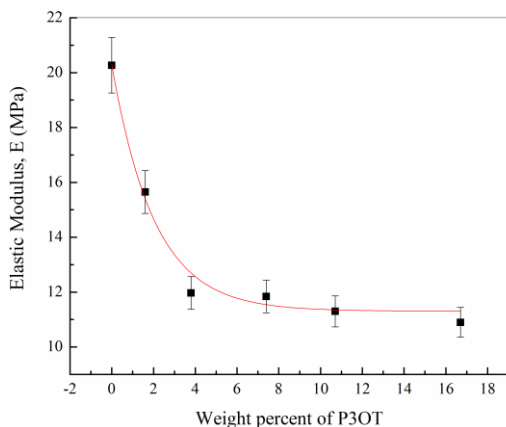


Figure 2: The dependence of the elastic modulus of PS on P3OT content at temperature 27°C.

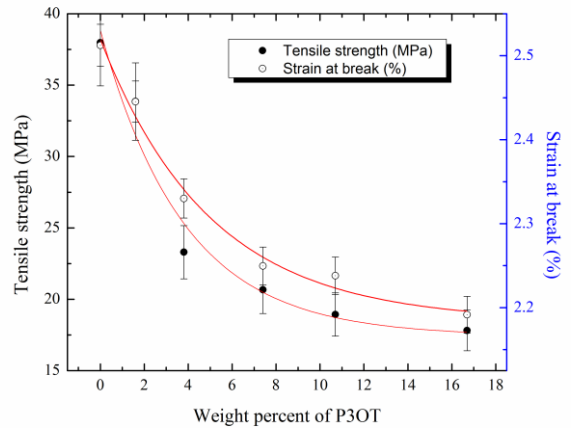


Figure 3: The dependence of the tensile strength and the strain at break of PS on P3OT content at 27 °C.

Figure 4. Apparently, P3OT have a crucial effect on the rheological behavior of the composites. The complex viscosity decreases with increasing P3OT content in the entire frequency range, but is more pronounced at low frequencies. At high frequencies the impact of the P3OT on the rheological properties is definitely weaker, which suggests that the soft phase (P3OT) do not significantly influence the short-range dynamics of the polymer chains. Generally, P3OT do affect polymer chain relaxation but with little effect on the local motion at short ranges [20]. The decrease in the complex viscosity with increasing frequency indicates a non-Newtonian behavior over the frequency range investigated. The complex viscosity can be expressed by an equation of the form

$$\log \eta^* = A - \alpha \log f \quad (2)$$

where A is a constant and α is the slope with values 0.989, 0.987, 0.984, 0.981, 0.98 and 0.88 for 0, 1.6, 7.4, 10.7 and 16.7 wt% of P3OT, respectively.

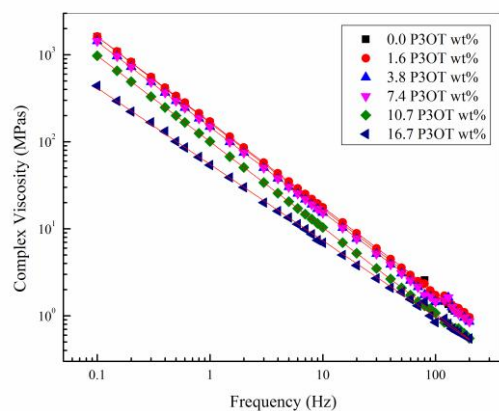


Figure 4: The complex viscosity as a function of frequency for different weight percent of P3OT in PS.

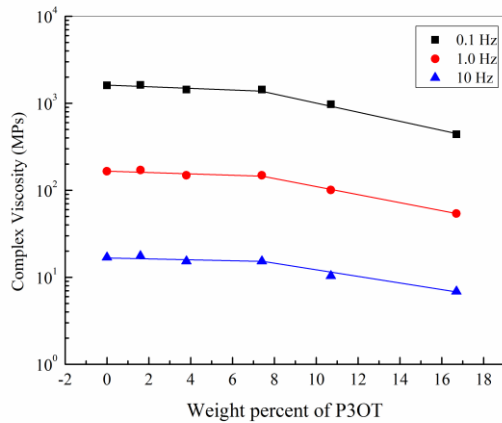


Figure 5: The complex viscosity as a function of P3OT content at different frequencies.

The dependence of η^* of the P3OT/PS blends with the P3OT content at different frequencies of 0.1, 1.0 and 10 Hz is shown in Figure 5. It can be seen that η^* of the blends has decreased with increasing P3OT content over the frequency range investigated. In addition, the extent of decrease in η^* with increasing P3OT content was more pronounced at both low frequency compared with that at high frequency and at concentrations of P3OT above 7.4 wt%. This decrease in η^* with increasing P3OT content may be attributed to the formation of new arrangements of blend chains. The decrease in the complex viscosity with increasing P3OT content shown in Figure 5 can be described by two mechanisms which represented by two straight lines with different negative slopes at same frequency. The first one appears below 7.4 wt% of P3OT at which polystyrene predominates with slopes -0.00782 , -0.00776 and -0.00765 at frequencies 0.1, 1.0 and 10 Hz, respectively. The second one appears above 7.4 wt% of P3OT with slopes -0.0556 , 0.0472 and -0.0364 at same frequency order. The first mechanism may reflect the behavior of polystyrene while the second one may reflect the behavior of P3OT alone or both polymers. This may be due to the incomplete miscibility of both polymers [11].

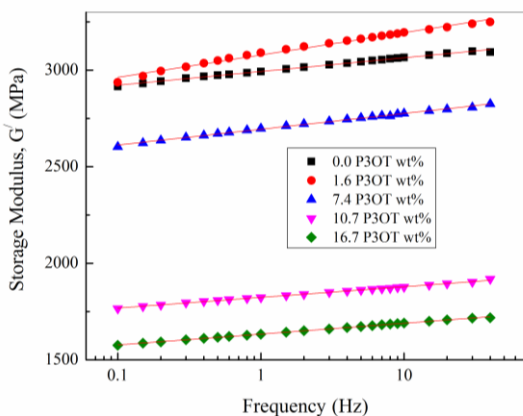


Figure 6: The storage modulus as a function of frequency for different weight percent of P3OT in PS.

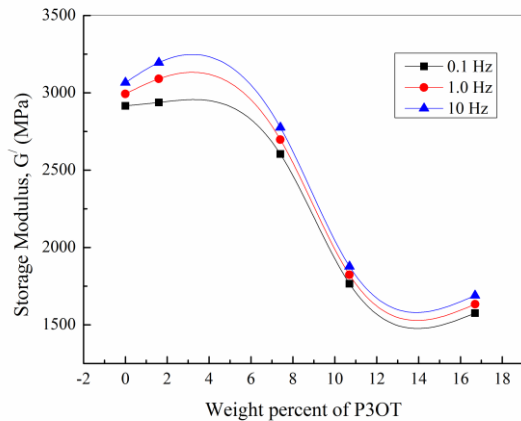


Figure 7: The storage modulus as a function of P3OT content at different frequencies.

The storage modulus G' of the P3OT/PS blends as a function of frequency at constant temperature of 27°C is shown in Figure 6. The figure shows an increase in G' with increasing frequency while a decrease is observed with increasing P3OT content except for the 1.6 wt % blend. This rheological response is similar to the relaxation behavior of the typical polymer composite systems [21]. It is known that the polymer chains are fully relaxed and exhibit characteristic homopolymer-like terminal flow behavior, resulting in that the flow curves of polymers being expressed by the power law $G' \propto \omega^2$ with ω being the angular frequency [22–24]. The dependence of the storage on the frequency can be described well by straight line relation of the form

$$G' = A' + B \log f \quad (3)$$

where A' is a constant and B is the slope with values 71.2, 115.5, 82, 55.3 and 56.2 for composite blends 0, 1.6, 7.4, 10.7 and 16.7 wt% of P3OT, respectively.

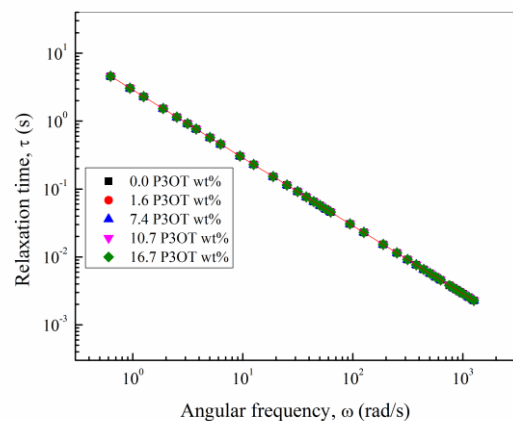


Figure 8: The relaxation time as a function of the angular frequency for different weight percent of P3OT in PS.

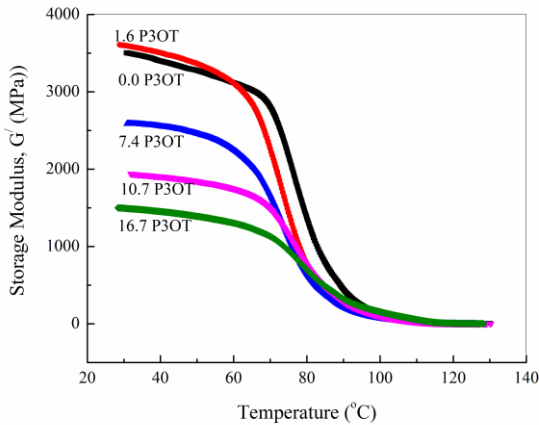


Figure 9: The dependence of the storage modulus on temperature for P3OT/PS at a frequency of 5.0 Hz.

The variations of the storage modulus for P3OT/PS composites blends with the P3OT content at different frequencies of 0.1, 1.0 and 10 Hz are shown in Figure 7. It can be seen that the incorporation of small quantity of P3OT (1.6 wt%) into PS matrix slightly increases the values of G' for the blend composites over the different frequencies used. This phenomenon may be attributed to some rearrangement of polymer chains making more coiling and entanglements. Beyond this concentration, significant decrease is observed for the G' values for all investigated frequencies in accordance with the decrease in the complex viscosity described before as well as the tensile elastic modulus E . This may be attributed to the formation of the growth of weak P3OT poly-domains over those of PS making it easier for flow to occur.

The relaxation time τ under dynamic shear in the polymeric systems that involved the pseudo-structures can be calculated from the following equation [25, 26]

$$J' = \frac{G'}{(\eta^* \omega)^2} = \frac{\tau}{\eta^*} \quad (4)$$

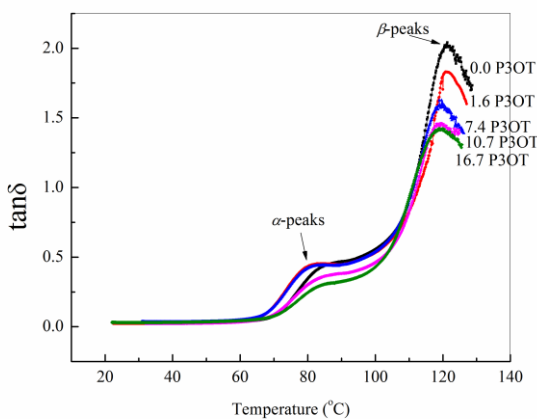


Figure 10: The dependence of the loss factor $\tan \delta$ on temperature for P3OT/PS at a frequency of 5.0 Hz.

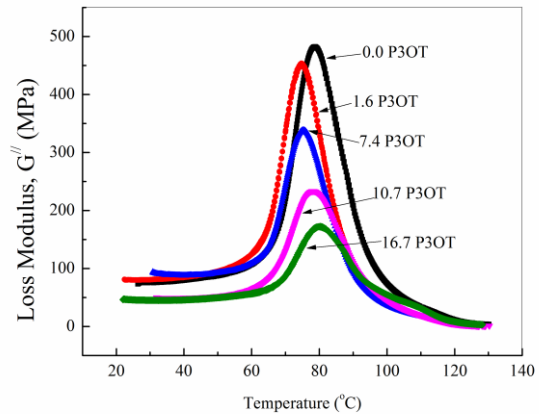


Figure 11: The dependence of the loss modulus on temperature for P3OT/PS at a frequency of 5.0 Hz.

Where J' is the compliance. It is expected that the presence of some molecular order or the physical structure leads to a much longer relaxation time [27]. As shown in Figure 8, the relaxation time of P3OT/PS blend composites decreases with increasing frequency according to an equation of the form

$$\log \tau = a + b \log \omega \quad (5)$$

where a is a constant and b is the slope of the log-log plot between τ and ω and equal unity. Interestingly, it is observed that the incorporation of PTO3 into PS host polymer does not affect the relaxation time at all.

3.3. Dynamic mechanical properties

Figure 9 plots the storage modulus G' of P3OT/PS blends, obtained by DMA measurement, as a function of temperature at a constant frequency of 5 Hz. A single mechanical transition has been observed in the temperature range studied and is recognized as the α -relaxation or the glass transition peak. The α -relaxation is related to the Brownian motion of the main chain associated with the glass transition and the relaxation of segments associated with it. Onset of glass transition is marked by a sharp decrease in its storage modulus, as shown in Figure 9.

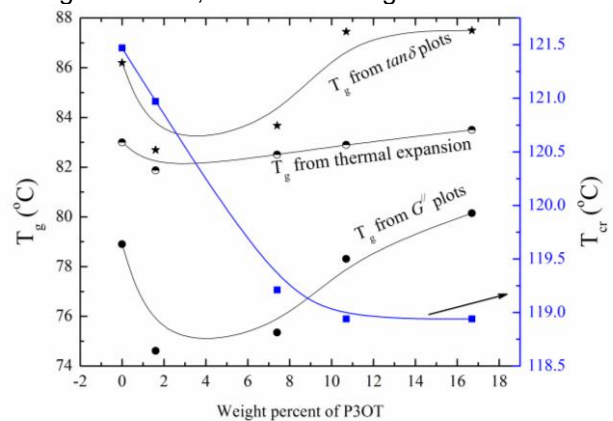


Figure 12: The dependence of the glass transition temperature obtained from different tests and the relaxation of the crystalline blend domains temperature on weight percent of P3OT in PS.

The glass transition temperature, T_g , of a polymer is usually taken from the peak position of loss modulus or $\tan \delta$ versus temperature curves. Peaks of $\tan \delta$ are typically found at somewhat higher temperatures depending on the intensity and/or width of the transition. Figure 10 presents the DMA thermograms of PS loaded with 0, 1.6, 7.4, 10.7 and 16.7 wt % of P3OT as mechanical loss tangent $\tan \delta$, against temperature in the range of 20 to 140 °C. All the thermograms exhibit two peaks designated as α and β peaks in the temperature ranges from 80 to 85 °C and 119 to 122 °C, respectively. The α - peak represents the glass transition temperature while the β - peak represents the relaxation of the crystalline blend domains for crystalline or semi-crystalline polymers [28]. In fact polystyrene haven't any crystallinity, so the β - peak may represent a relaxation of chains from one configuration to another. The figure detects a shift in the α - peak toward lower temperatures for 1.6 wt % of P3OT and then shifted to higher temperature. On the other hand, the β - relaxation peak shifts toward lower temperatures as P3OT content increased. The positions of the α - relaxation peaks are not defined correctly since these peaks are not sharp enough to identify the glass transition temperature. Thus, a plot between the loss modulus, G'' , versus temperature for all studied blends at a frequency of 5 Hz, shown in Figure 11 are used to define T_g with some little shifts to lower temperatures. Again, as mentioned in Figure 10, adding 1.6 wt % of P3OT to the PS matrix shifts the G'' peak from 78.90 °C for pure to 74.61 °C. The increase of P3OT content beyond this concentration increases the G'' peak position up to 80.15 °C for 16.7 wt% of P3OT implying mechanical restriction on the mobility of the PS chains. Increasing the P3OT content also broadens the G'' peak, and lowers its intensity. The breadth of the peak is related to the relaxation of the polymer chain. The constraints on the mobility of the polymer chains due to their being tethered to the P3OT widen the range of temperatures covered by the G'' peak. The height of the G'' peak measures the energy-damping characteristics of a material. P3OT which acts as a soft phase easily rearrange PS chains the G'' peak thus declines as P3OT content increases. The change in the glass transition temperature detected from both $\tan \delta$ and G'' plots and the crystalline relaxation temperature, T_{cr} , as a function of P3OT content are shown in Figure 12.

3.4. Coefficient of thermal expansion

The coefficient of thermal expansion, CTE, quantified the thermal expansion of the solid. The coefficient can be volumetric or linear according to the fact that the measurement is determined by the change of volume or length of the sample. The expression to calculate the linear coefficient of thermal expansion, α , by [29]

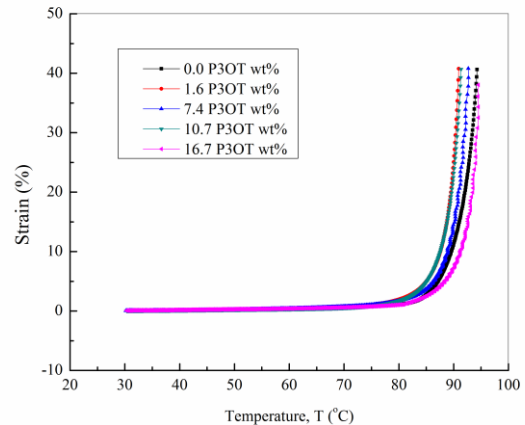


Figure 13: Thermal mechanical analyzer test curves of the P3OT/PS.

$$\alpha = \frac{1}{L} \left(\frac{\partial L}{\partial T} \right)_p \quad (6)$$

where L is the length, T is the temperature, and the derivative is at constant pressure P . The CTE of all produced blend samples can be calculated based on the thermal-expansion curves shown in Figure (13). For polymers the CTE values can be divided into two parts: below and above the T_g . The most useful CTE concerns the temperature below T_g , since polymers or composites lose most of their mechanical properties above T_g . The values of α below T_g are 9.7×10^{-3} , 8.3×10^{-3} , 10.5×10^{-3} , 9.2×10^{-3} and 9.4×10^{-3} °C⁻¹ for blends 0, 0, 1.6, 7.4, 10.7 and 16.7 wt% of P3OT, respectively. On the other hand, the values of α above T_g are 7.46, 9.13, 7.37, 7.74 and 5.79 °C⁻¹ for same order of blends.

The magnitude of the CTE depends on the structure of the materials. For single-phase materials, CTE is determined from atomic bonding, molecular structure, and molecular assembly. An elevated temperature would increase thermal energy and lead to an increase in the atomic movement. Weak atomic bonding with a low bonding energy would show large CTE due to the increase in the interatomic distance. For multiphase materials, such as blends, the CTE are dependent on each component phase and also the interactions between each phase. Weak interface bonding between phases could not effectively incorporate the contributions of each component, while strong interface bonding could compromise each ingredient for thermal expansion properties. The slight change of the CTE in the investigated blends below or above T_g indicates possible poor interaction in the blends.

Conclusions

Poly(3-octylthiophene)/polystyrene (P3OT/PS) blends with different weight ratios of P3OT were prepared using casting technique. Tensile properties

of these blends detect a decrease in the elastic modulus, tensile strength and strain at break with increasing P3OT content. Rheological properties, also, detect a decrease in the complex viscosity and storage modulus as the P3OT content increases. The results of the dynamic mechanical properties indicate that the storage modulus changed steadily, and the loss modulus peaks are broader and less intense for high P3OT content. Less interaction between the two blend phases is proved from the detected values of the coefficient of linear expansion and the glass transition temperature.

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