

## **Investigating Rheological, Morphological and Mechanical Properties of PBS/PBAT blends**

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### **Abstract**

Morphological, rheological and mechanical properties of various biodegradable poly(butylene succinate) (PBS)/poly(butylene adipate-co-terephthalate) (PBAT) blends via conventional melt blending technique are studied. The morphology, rheology, and tensile properties of the PBS/PBAT blends were observed by scanning electron microscope (SEM), rotational rheometer, and universal testing machine, respectively. The SEM micrographs of the PBS/PBAT blends reveal an occurrence of phase separation indicating by a presence of spherical particles/cavities for a range of 10 to 30 wt% (i.e. PBS90-PBS70), and 70 to 90 wt% (i.e. PBS30-PBS10) PBT content and co-continuous structure for a range of 40 to 60 wt% (i.e. PBS60-PBS40) PBAT content. The rheological observation reveals that storage modulus ( $G'$ ), loss modulus ( $G''$ ), and complex viscosity ( $\eta^*$ ) for the PBS/PBAT blends exhibit similar tendency. The morphological properties of the blends appear to affect the tensile behavior of the PBS/PBAT blends.

**Keyword:** Biodegradable polymer; Poly(butylene adipate-co-terephthalate); Poly(butylene succinate); Polymer blend; Rheology; Morphology.

### **Introduction**

In the light of global environmental concern<sup>(1-5)</sup> biodegradable polymers, such as poly(lactic acid) (PLA), poly(butylene succinate) (PBS), polyhydroxybutyrate (PHB), poly(butylene adipate-co-terephthalate) (PBAT) and polyvinyl alcohol, have been extensively studied as evidenced by numerous patents and research publications. However, most of biodegradable polymer has some limitation in industrial development owing to lack of some properties<sup>(6)</sup>. To address those drawbacks, numerous modification techniques have been applied. To enhance the properties of polymer by taking the advantages of each component, blending technique is one of the most promising techniques on account of cost effective and less time consuming comparing to other techniques<sup>(7-9)</sup>.

Poly(butylene succinate) (PBS) is a biodegradable aliphatic polyester synthesized via polycondensation reaction of 1,4-butane diol and succinic acid.<sup>(7, 10-12)</sup> Compared with petroleum-based plastics, PBS has an excellent properties such as biodegradability, thermal stability,

thermal and chemical resistance and good processing properties<sup>(13-16)</sup>. However, some properties such as gas barrier properties, melt viscosity for further processing, etc. are insufficient for various end-use applications<sup>(11)</sup>. To improve those properties of PBS with retaining biodegradability, PBS is in general blended with other biodegradable polymers such as polycaprolactone (PCL), polyhydroxybutyrate (PHB) and poly(butylene adipate-co-terephthalate) (PBAT)<sup>(6-7, 16-17)</sup>. John et al.<sup>(7)</sup> reported about an incorporation of PCL 30 to 70 wt% in PBS, and found that PBS/PCL blend was not completely miscible indicating by DMA results showing two Tg values. They found that with increased PCL content the mechanical properties, i.e. tensile strength, and crystallinity decreased due to the phase separation of the blend systems. Surprisingly, PBS with 30 wt% PCL blend showed an improved tensile strength from 33.30 to 43.75 MPa, percentage of elongation from 126 to 238, and crystallinity from 41 to 70. This suggested an occurrence of a synergetic effect in these blend compositions. Qui et al.<sup>(16)</sup> blended PBS with PHB in a ratio

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of 20, 40, 60, and 80 wt%. The result showed that miscibility was simply limited to PBS/PHB 80/20 blend indicated by the change in glass transition temperature from DSC results, the depression of the equilibrium melting point temperature of PHB, and the decrease in the crystallization peak temperature of PBS. Muthuraj *et al.*<sup>(6)</sup> studied on the PBS/PBAT 60/40, 70/30 and 50/50 wt%) blend revealed that the rheological properties of the blends increased with an incorporation of PBAT in the matrix. Muthuraj *et al.*<sup>(17)</sup> also reported that good compatibility achieved in the PBS/PBAT (40/60 wt%) blends evidenced by a uniform dispersion of the PBS phase in the PBAT phase. Tensile strength increased with increasing amount of PBS while elongation at break decreased. The phase morphology of the PBS/PBAT blends showed a two phase structure in which PBAT is the minor phase.

In recent times, PBAT has received considerable attention because of its good biodegradable<sup>(18-20)</sup> excellent toughness<sup>(6)</sup> high flexibility<sup>(21-23)</sup> resistant to water<sup>(9)</sup> better process ability than other biodegradable polyesters<sup>(24)</sup> and also commercial availability<sup>(25)</sup>. Attributable to its high toughness, PBAT is mostly used in film extrusion industries (especially in packaging films, agricultural films and compost bags)<sup>(9, 26)</sup>. Typically, it's apparent that PBAT has been blended with other biodegradable polymers such as poly(lactic acid)<sup>(27)</sup> polycarbonate<sup>(28)</sup> recycled PET (rPET)<sup>(29-30)</sup> poly(hydroxybutyrate-co-valerate)<sup>(31)</sup> and poly(butylene succinate).

As mentioned above, blending PBS and PBAT study has received several interests because both of them are natural biodegradable, unique properties and commercial availability<sup>(17, 25, 32)</sup>. However, a study of PBS/PBAT blends mainly focus on mechanical properties and

rheological properties of low range of PBAT content. So that, in this study, an extensive ratio range of PBS/ PBAT blends from 0 to 100 wt% with an increment of 10 wt% are prepared via conventional melt blending techniques. The effect of blend composition on rheological and morphological and mechanical properties were determined as well as the relations among these properties.

## Materials and methods

### Materials

Poly(butylene succinate) (PBS), GS PLA FZ91PD; Pellet; T<sub>m</sub> = 118°C; specific gravity = 1.26 g/cm<sup>3</sup>) was supplied by Mitsubishi Chemical Corporation. Poly (butylene adipate-co-terephthalate) (PBAT, Ecoflex FBX7011; T<sub>m</sub> = 110°C; specific gravity = 1.25 g/cm<sup>3</sup>) was supplied by BASF. Both materials were dried at 60°C in an oven for 24 h. prior to processing.

### Sample Preparation

The PBS/PBAT blends were firstly mixed using an internal mixer (Brabender WIN/B/S 350E) at 120°C with a rotor speed of 50 rpm. After that, the obtained compound was pelletized by pelletizer (Retsch SM100). Subsequently, the samples were fabricated using a Collin P500PM compression moulding machine at 160°C at a moulding pressure of 1,000 MPa for 10 min heating time and 10 min cooling time in 100x100x1 mm, 64x12.5x3 mm for rheological testing and SEM testing, respectively. The dumbbell shaped mould was used for preparing tensile testing samples. The compositions of the PBS/PBAT blends prepared in this study are summarized in Table 1.

**Table 1.** Compositions of PBS/PBAT blends.

Samples	PBS100	PBS90	PBS80	PBS70	PBS60	PBS50	PBS40	PBS30	PBS20	PBS10	PBS0
PBS (wt%)	100	90	80	70	60	50	40	30	20	10	0
PBAT (wt%)	0	10	20	30	40	50	60	70	80	90	100

### Rheological Studies

The rheological properties of the PBS/PBAT blends were investigated by a rotational rheometer (Malvern Bohlin Gemini HR nano) on a parallel plate (gap = 1 mm) with frequency

sweep mode. Storage modulus ( $G'$ ), loss modulus ( $G''$ ), and complex viscosity ( $\eta^*$ ) were determined using dynamic frequency sweep mode as functions of frequency in a range from 0.01 to 100 rad/s at 160°C.

### Mechanical Studies

Dumbbell shaped type IV specimens of the neat polymers and the PBS/PBAT blends were prepared according to the ASTM D638-15. Young's modulus, tensile strength and elongation at break values at ambient temperature were investigated by Instron 5569 Machine, with 10kN load cell and 50 mm/min crosshead speed.

## Results and Discussion

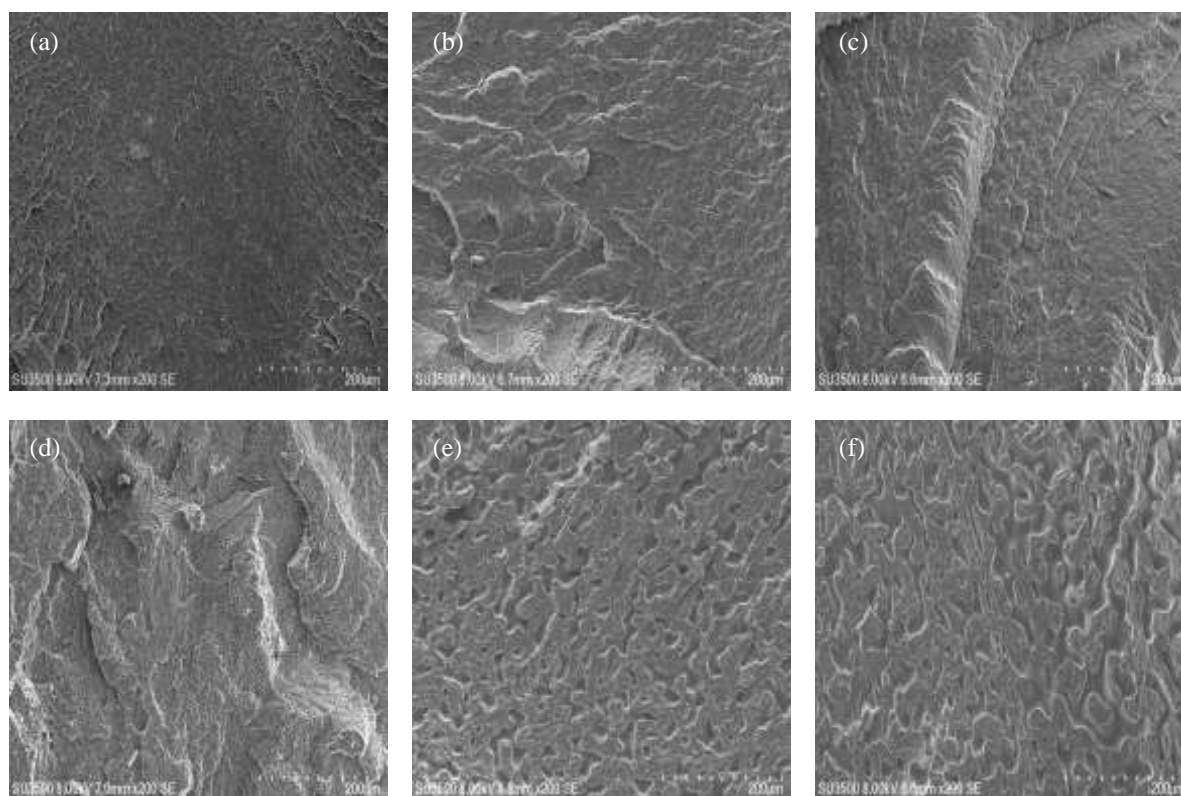
### Morphological Observations

The fracture surface of the samples was examined by scanning electron microscope to study the morphology of the surface. In this study, phase morphology of the blends was identified by the solvent etching method using THF as the etching solvent to dissolve the PBAT phase. SEM micrographs of cryogenically fractured neat polymers and PBS/ PBAT blends with various content of PBS are shown in Figures 1-4. It's apparent that two-phase morphology, i.e. either one phase as spherical

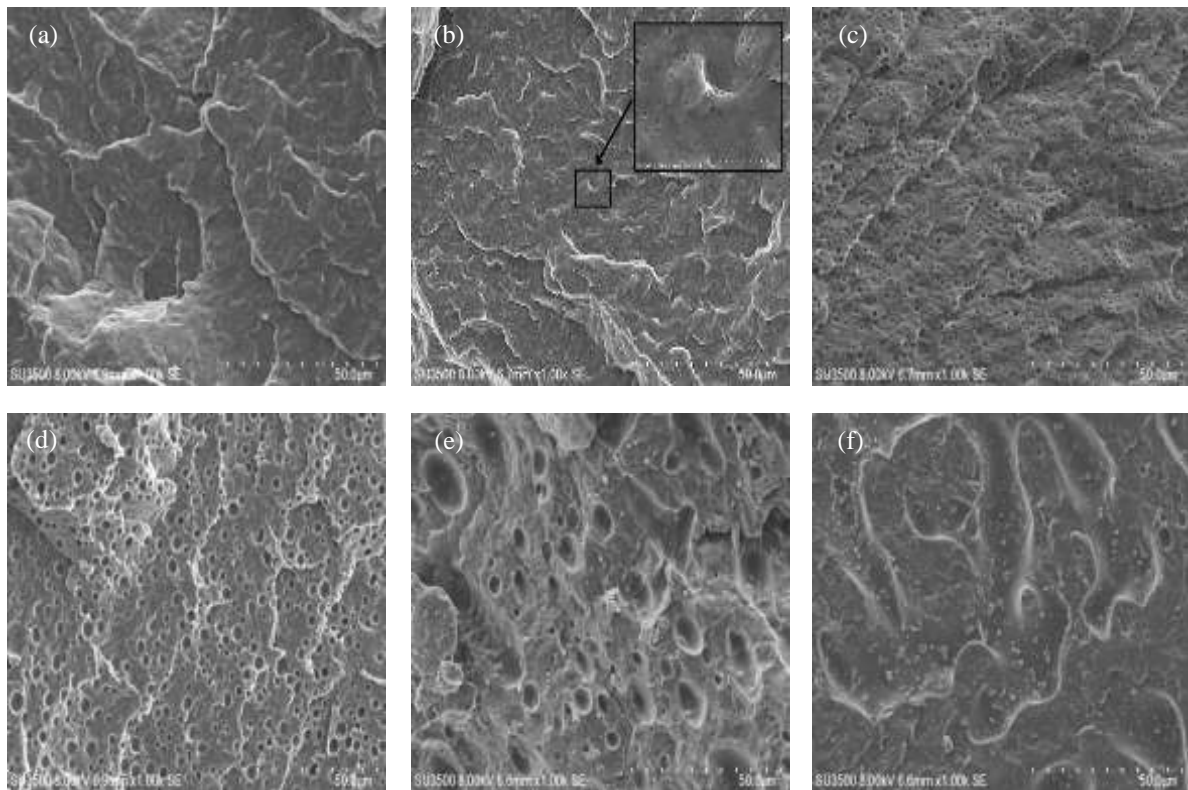
particles/cavities dispersed in another continuous phase or co-continuous phases, is presented for all the blends, except for the neat polymers.

Figures 1 and 2 show the SEM micrographs of cryogenically fractured the neat PBS and the PBS100-PBS50 blends (i.e. PBS major phase) at  $\times 200$  and  $\times 1,000$  magnifications, respectively. For the PBS major phase, the cavities exhibited in the SEM micrographs represent the extracted PBAT phase. As expected with greater PBAT content (i.e. from 40 to 50wt% PBAT), PBS60 and PBS50 clearly exhibit co-continuous structure as depicted in Figures 1(e) and 1(f). The co-continuous phase morphology is always a temporary morphological state through which one mode of dispersed structure is transformed into another dispersed mode<sup>(33)</sup>.

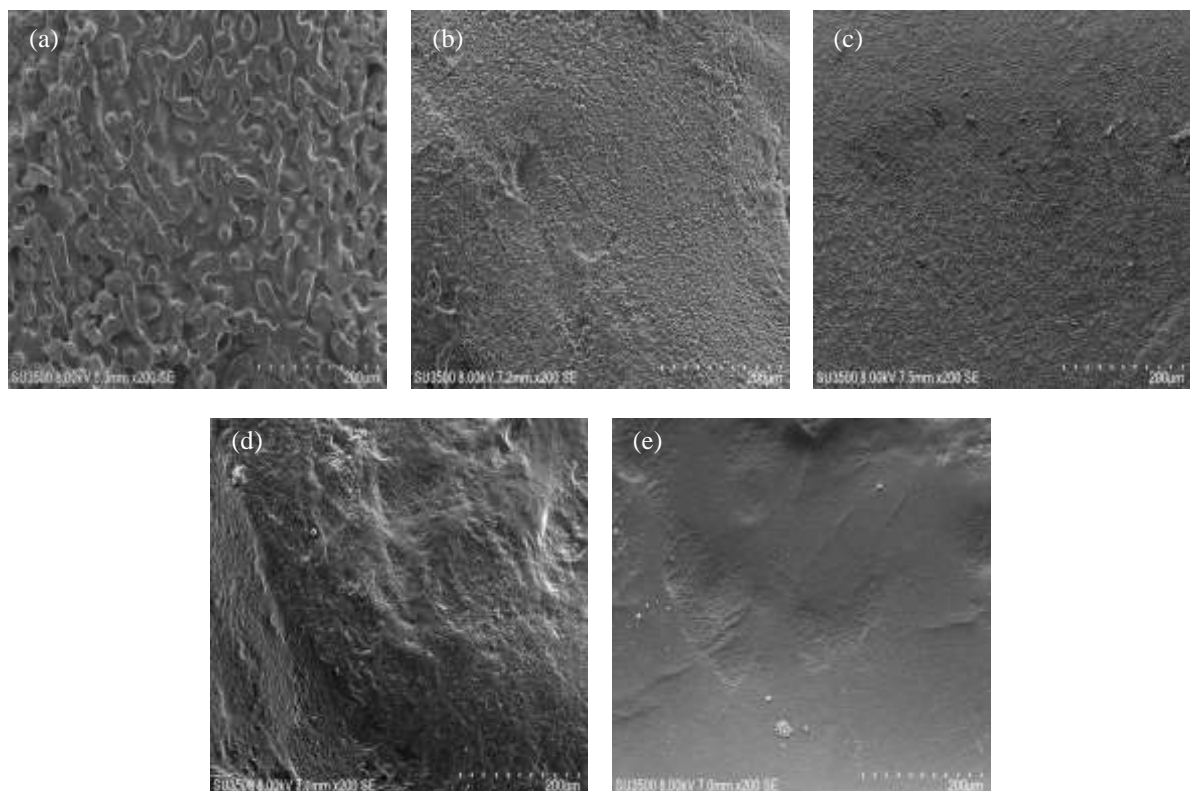
At  $\times 1,000$  magnifications, the dispersed cavities are obviously illustrated for PBS90-PBS70 as seen in Figures 2(b)-2(d). With greater PBAT content, the cavity's dimension became larger and less uniform distribution. Besides, it's apparent that some spherical cavities yet stayed for PBS60, but not for PBS50 indicating a complete co-continuous structure.



**Figure 1.** SEM micrographs at  $\times 200$  magnifications of (a) PBS100, (b) PBS90, (c) PBS80, (d) PBS70, (e) PBS60 and (f) PBS50.



**Figure 2.** SEM micrographs at  $\times 1,000$  magnifications of (a) PBS100, (b) PBS90, (c) PBS80, (d) PBS70, (e) PBS60 and (f) PBS50.



**Figure 3.** SEM micrographs at  $\times 200$  magnifications of (a) PBS40, (b) PBS30, (c) PBS20, (d) PBS10 and (e) PBS.

Figures 3 and 4 show the neat PBAT and the PBS/PBAT blend in a range of 40-10 wt% of PBS (i.e. PBAT major phase) at  $\times 200$  and  $\times 100$  magnification, respectively. With increasing PBAT content exceeding 50 wt%, the PBAT turned from the dispersed phase to the matrix as seen from the SEM micrographs in Figures 3 and 4. It's obvious that the embossed structure representing the PBS since THF dissolved the PBAT. Once more, the co-continuous structure

was detected for PBS40, which is relatively similar to PBS60. This implies that co-continuous phase generally occurs as both polymers having somewhat comparable amount.

Again, at  $\times 1,000$  magnifications, the embossed structure (i.e. referred as "particle") is simply seen in the SEM micrographs as shown in Figure 4 where the particle became finer with increasing PBAT content.

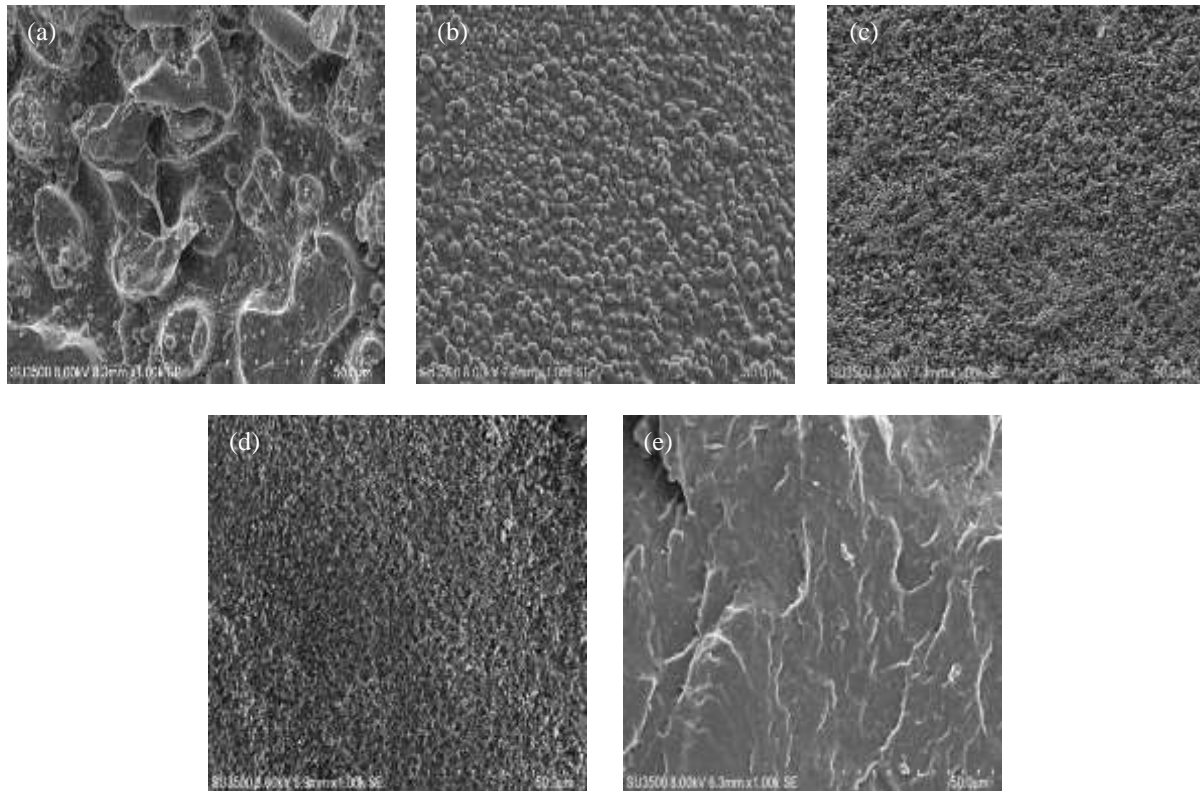


Figure 4. SEM micrographs at  $\times 1,000$  magnifications of (a) PBS40, (b) PBS30, (c) PBS20, (d) PBS10 and (e) PBS0.

### Rheological Observations

The change of logarithm of the complex viscosity ( $\eta^*$ ) on the logarithm of angular frequencies ( $\omega$ ) of the neat polymers (i.e. PBS and PBAT) and all PBS/PBAT blends is presented in Figure 5.

The frequency dependence of the complex viscosity for neat polymers and all PBS/PBAT blends at  $160^\circ\text{C}$  exhibits a declined complex viscosity with an increase in frequency, representing the pseudoplastic behavior (i.e. shear thinning behavior, that is the fluids whose viscosity decrease with increase shear rate<sup>(34)</sup>). This could be explained by the fact that as applying the force, there is an occurrence of

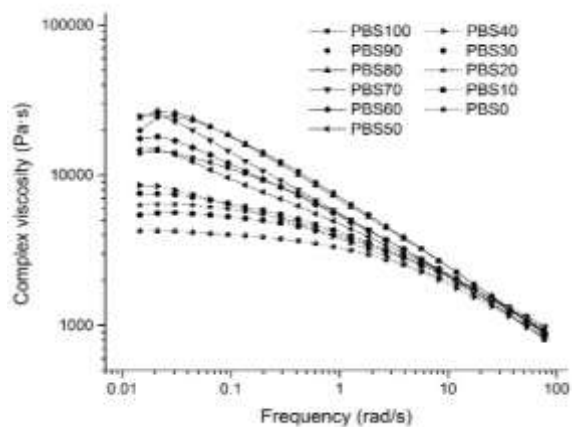


Figure 5. Complex viscosity of neat polymers and all PBS/PBAT blends.

chain deformation, which is generally caused by disentanglement and chain slippage over each other<sup>(35)</sup>. These mechanisms initiate a molecular alignment in the direction of applied force enhancing flow of polymer chain resulting in reduced complex viscosity. With energy transformation enhancement to the sample by increasing frequency, this amplifies the chain deformation mechanism providing lower complex viscosity values.

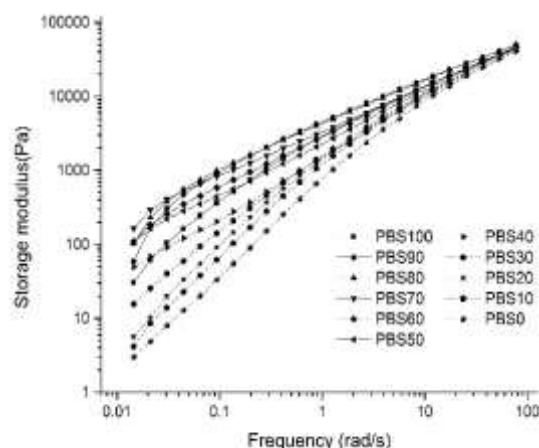
The complex viscosity of the neat PBAT (i.e. PBS0) is lower than the neat PBS (PBS100), which exhibits nearly Newtonian behavior (i.e. viscosity doesn't vary with shear rate<sup>(34)</sup>) at below 0.03 rad s<sup>-1</sup> frequency and follows by shear thinning behavior beyond that. It can be seen that the neat PBAT has longer Newtonian region at below 0.2 rad s<sup>-1</sup> frequency. It's noticeable that the Newtonian region of the PBS/PBAT blends intensifies with increasing PBAT content. Similar behavior was observed by Li et al.<sup>(24)</sup> for the PLA/PBAT blends, that is a distance of Newtonian region is subject to PBAT content. As anticipated, the complex viscosity values of PBS40-PBS0 blends falls between the values of neat PBS and neat PBAT where the complex viscosity value of PBS40-PBS0 blends increases with an increase in PBS content.

For the PBS major phase (i.e. PBS100-PBS60 blends), it is surprisingly observed that the PBS/PBAT blends exhibit higher complex viscosity than the neat PBS. This may be a result of a dispersed phase (i.e. PBAT in this case) has an interaction or entanglement with the matrix phase (i.e. PBS)<sup>(24)</sup> where this interaction appears to a transesterification reaction. This can be supported by a study of Muthuraj et al.<sup>(6,17)</sup> which discloses the transesterification product, that is pseudo structures, via PBS/PBAT blend. This transesterification product can withstand applied force providing less polymer chain deformation (i.e. higher complex viscosity value). They also revealed that the transesterification product gradually increases with decreasing PBAT content. It's observable from the previous studies that the transesterification reaction normally occurs in polyester blend, e.g. PHB/PLA,<sup>(36)</sup> PBS/PCL,<sup>(7)</sup> poly(ethylene-terephthalate)/poly(etherimide)<sup>(37)</sup> and PBS/PBAT<sup>(6,17)</sup>.

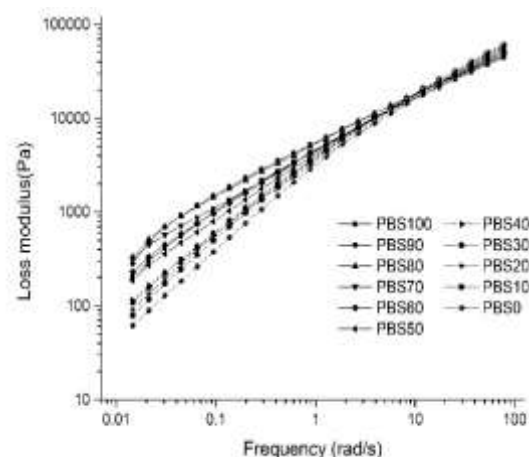
For PBS50 blend, it is noticeable that the complex viscosity at greater 0.03 rad s<sup>-1</sup> frequency is lower than the neat PBS. Li et al.<sup>(24)</sup> reported similar phenomenon for the PLA/PBAT blend at 50/50 ratio where lower

complex viscosity than the neat PLA exhibits at greater 0.06 rad s<sup>-1</sup> frequency. They revealed that this phenomenon may be attributed to a change of continuous evolution morphology.<sup>(24)</sup> In this study, the continuous evolution morphology is supported by the morphological observations earlier, which is a morphological change from one phase dispersed in another continuous phase to co-continuous phase for PBS40-PBS60 blends. This co-continuous phase reveals apparent phase separation providing weak interaction between PBS and PBAT phase resulting in lower complex viscosity.

Figures 6 and 7 show storage modulus ( $G'$ ) and loss modulus ( $G''$ ) on the logarithm of angular frequencies ( $\omega$ ) of the neat polymers and the PBS/PBAT blends.



**Figure 6.** Storage modulus of neat polymers and all PBS/PBAT blends.



**Figure 7.** Loss modulus of neat polymers and all PBS/PBAT blends

It appears that both  $G'$  and  $G''$  increase with greater frequency for all compounds, which is the typical behavior of polymers under the effect of frequency. In lower frequency, there is sufficient time allowing polymer chains to reorganize themselves displaying soft properties and lower modulus. Conversely, in higher frequency, the time to response to the outer change is very short, so polymer shows hard properties. Moreover, it is noticeable that similar behavior to the complex viscosity is illustrated, which the storage and loss modulus of PBS40-PBS0 blends falls between the values of neat PBS and neat PBAT, and the storage and loss modulus of PBS100-PBS60 blends show greater value than the neat PBS. As abovementioned, the greater storage and loss modulus of PBS100-PBS60 blends may be because a dispersed phase (i.e. PBAT in this case)

has an interaction or entanglement with the matrix phase (i.e. PBS). This generates more recoverable energy in the blends.<sup>(24)</sup>

It's generally known that the plot of imaginary viscosity ( $\eta''$ ) versus real viscosity ( $\eta'$ ) (where  $\eta'' = G''/\omega$  and  $\eta' = G'/\omega$ <sup>(24)</sup>), which is known as a Cole-Cole plot, is applied to explain the phase structure of polymer blends.<sup>(38-39)</sup> For homogenous blend, the Cole-Cole plot gives single arc curve. If the curve has any deviation from single arc curve, it indicates phase separation in the blends caused by a second relaxation mechanism in the samples.<sup>(40-42)</sup> Namely, the Cole-Cole plot clearly shows different relaxation mechanism of two different phases.<sup>(43)</sup> Figures 8 and 9 show the Cole-Cole plots of PBS100-PBS50 and PBS50-PBS0, respectively.

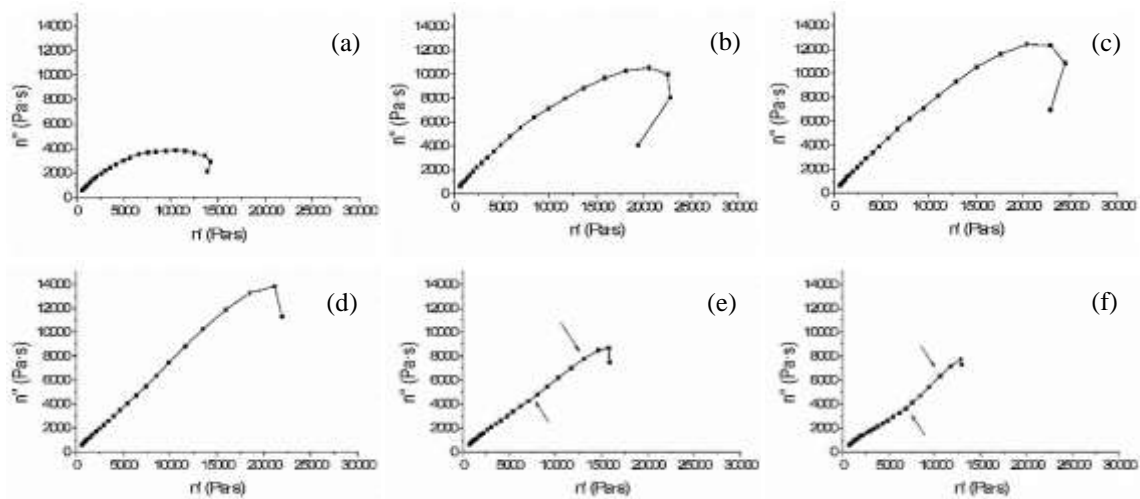


Figure 8. Cole–Cole plots of (a) PBS100, (b) PBS90, (c) PBS80, (d) PBS70, (e) PBS60 and (f) PBS50.

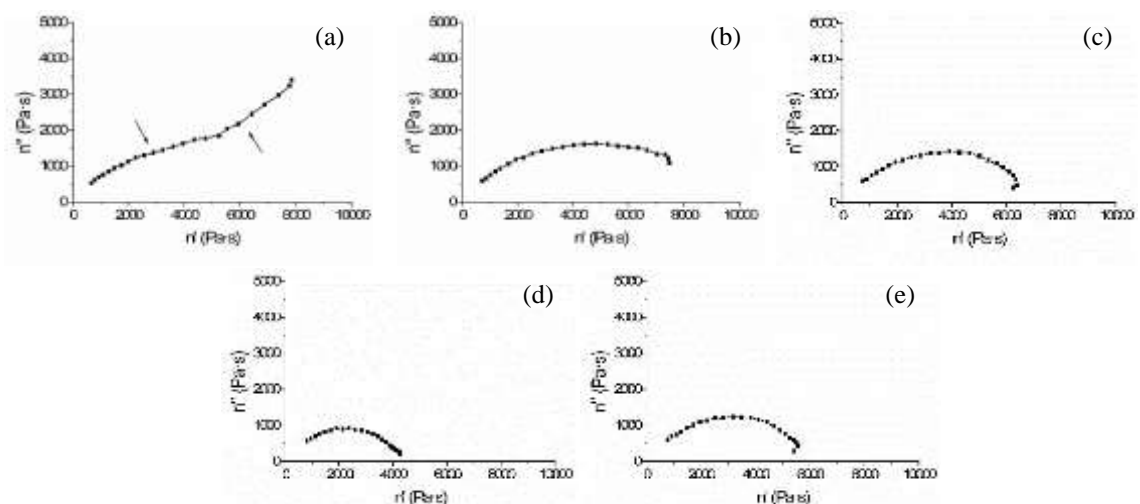


Figure 9. Cole–Cole plots of (a) PBS40, (b) PBS30, (c) PBS20, (d) PBS10 and (e) PBS0.



It's clearly seen that both neat PBS and PBAT (as seen in Figures 8(a) and 9(e)) show semicircle indicating homogenous blend. The Cole–Cole plots of the prepared blends deviated from the semicircular shape due to second relaxation mechanism which from the minor phase in the blends. The deviation from the semicircular shape was more obvious for PBS60-PBS40. The extent of this deviation is related to the co-continuous phase separation in the blends as illustrated earlier in morphological observation.

### *Tensile Properties*

Table 2 shows tensile properties of the neat polymers and the PBS/PBAT blends. It's noticeable that the neat PBS exhibits higher Young's modulus and tensile strength values than the neat PBAT. While the neat PBAT shows greatly higher elongation at break than the neat PBS. This may be caused by nature properties of PBAT, which has high flexibility and toughness. As anticipated, Young's modulus of the PBS/PBAT blends falls between the values of the neat polymers, and decreases with increasing PBAT content, suggesting heterogeneous blend in the system.

It can be observed that the tensile strength results may relate to the morphological observation. As spherical particles/cavities dispersed in another continuous phase (i.e. PBS70-PBS100 and PBS0-PBS30 blends), the results shows that the tensile strength decreases with increasing amount of particles dispersed phase. This may be attributed to phase separation causing weak interaction between the PBS and PBAT phase. For co-continuous phase (i.e. PBS60-PBS40 blends), both polymers tend to retain their properties, hence; the tensile strength remains decreased as increasing the PBAT content. It is further noticeable that the tensile strength value of PBS40 blend is greater than PBS30 blend although greater PBAT content in PBS40 blend. This may caused by the co-continuous of each phase.

It's apparent that the neat PBAT displays significantly higher elongation at break value than the neat PBS owing to PBAT having better flexibility. As expected, the elongation at break of the PBS/ PBAT blends tends to increase with increasing PBAT content. John *et al.*<sup>(7)</sup> also found the same phenomenon on PBS/ESTAR blend, that is the elongation at break of the blends increase with increasing ESTAR content.

**Table 2.** Tensile properties of neat polymers and PBS/PBAT blends

Sample	Young's Modulus (MPa)	Tensile strength at break (MPa)	Elongation at Break (%)
PBS100	918.26 ± 12.73	38.11 ± 1.58	6.54 ± 0.62
PBS90	773.81 ± 10.40	36.35 ± 0.75	8.77 ± 0.48
PBS80	629.22 ± 23.12	29.79 ± 1.15	10.41 ± 0.83
PBS70	544.07 ± 11.91	25.66 ± 1.09	9.00 ± 0.70
PBS60	461.83 ± 24.04	22.14 ± 1.94	10.00 ± 1.69
PBS50	365.36 ± 13.70	18.51 ± 1.00	11.00 ± 1.66
PBS40	263.03 ± 13.42	15.80 ± 0.84	19.59 ± 2.67
PBS30	163.78 ± 11.47	11.70 ± 0.58	59.69 ± 5.16
PBS20	134.75 ± 10.90	14.20 ± 1.76	225.88 ± 56.33
PBS10	94.04 ± 4.24	18.27 ± 1.43	374.75 ± 58.78
PBS0	78.13 ± 5.70	22.04 ± 0.67	532.50 ± 11.39

### **Conclusion**

Morphological, rheological and mechanical properties of biodegradable PBS/PBAT blends using the conventional melt blending technique are influenced by the PBAT content. The SEM micrographs reveal an apparent co-continuous structure for PBS40-PBS60 where both polymers having somewhat comparable amount.

This morphological observation has an impact on tensile properties, which, for the co-continuous structure, each polymer trend to keep their character and have further effect to change of tensile property. This can also be observed by the Cole–Cole plots where the intensity of deviated semicircular shape was detected for PBS60-PBS40. For rheological properties, it is surprisingly observed that the



PBS/PBAT blends exhibit higher complex viscosity than the neat PBS and PBAT. This assumes to be caused by a transesterification reaction generated pseudo structures, which can withstand applied force providing less polymer chain deformation, more energy being dissipated.

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