

## **Blending Poly (3-Hydroxybutyrate) with Polyethylene Glycol (PEG): Thermal behaviour and rheological study**

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### **ABSTRACT**

Poly (3-hydroxybutyrate) (PHB) is a biodegradable polymer which its application in industrial was limited due to sensitive to high processing temperature and poor mechanical properties. The thermal and rheological behavior for blends of PHB extracted from dry weight of *Jatropha* and Poly (ethylene glycol) (PEG) were investigated. Blends of PHB/PEG (PHB 100, PHB 80, PHB 60 and PHB 50) have been prepared through solution blending method. The thermal using Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) and rheological behavior using Oscillating Disk Rheometer have been investigated. The results indicate all PHB/PEG blends showed decreased at lowest melting temperature crystallinity ( $X_c$ ). The TGA result showed that PHB 80 less degraded and suggest better thermal stability compared to PHB 60 and PHB 50. Adding PEG to PHB also improved the rheological behavior of PHB. At higher frequency range, PHB 100 showed less dependency on frequency indicating the elastic structure it has compared to PHB 80, PHB 60 and PHB 50.

**Keywords:** Polyhydroxybutyrate; Polyethylene Glycol; Thermal Stability; Rheological Behaviour

### **INTRODUCTION**

Poly(3-hydroxybutyrate) (PHB) is naturally occurring polyester that produced from renewable sources by different kind of bacteria in nature. Biodegradability of PHB gained many interest mainly in food packaging, storage and agriculture. Biocompatibility of PHB also became key feature for use in biomedicine as drug delivery vehicles, scaffolds for tissue engineering and surgical sutures. Unfortunately, the marketability of PHB is limited and therefore makes PHB more expensive than other available bioplastic although PHB have good properties. However, high processability, high crystallinity [1]-[2] and reduced mechanical performance of PHB are the effects resulting from unstable PHB at higher temperature (higher than melting temperature) causing by thermal degradation [3]. High thermal instability cause PHB crystallizes from melt at high temperature and brittleness

makes PHB fragile materials [4]-[5]. Polymer blends have been of great interest to materials scientist because these blends possess improved and modified properties over the individual constituent polymers. These blends include completely biodegradable blends with poly(vinyl alcohol), polylactide (PLA) and partially biodegradable blends with poly(methyl methacrylate) and poly(vinyl acetate) [6]. Poly (ethylene glycol) (PEG) is known to have an efficient miscibility with PHB. PEG has outstanding properties including good solubility in organic solvents and water, hydrophilic thermoplastic and not biodegradable. In addition, PEG is a lack of toxicity, no antigenicity and no immunogenicity makes PEG suitable for biomedical applications [7]-[8], [9]-[10]. Since PHB is a semi-crystalline polymer, there is strongly dependent between properties and processing condition [3] and therefore it is highly desirable to study on how to improve PHB characteristics. Aim of this paper is to study the effect of thermal and rheological behaviour to PHB/PEG blend. In this work, PHB were extracted from *Jatropha* dry weight (CDW). Previous studies used *Jatropha* oil as carbon source for PHB production that extracted from *Jatropha curcas* seeds. *Jatropha curcas* is a non-edible plant that has capability to grow on saline and sandy soils. Since, it is non-edible plant, it does not compete with global food issues [11], [12], [13]. To our knowledge, there is no published report related to rheological properties of PHB extracted from *Jatropha curcas* seeds and being blended with PEG.

## METHODS AND MATERIALS

### Materials

*Jatropha* dry weight (DW) used in this study was kindly supplied by School of Biology, University of Science, Malaysia produced by Prof Kumar Sudesh. Low molecular weight of Polyethylene Glycol (PEG) with molecular weight,  $M_w = 1500$  commercially purchased from (R & M marketing, United Kingdom) were obtained from chemical laboratory, University Teknologi MARA (UITM). The extraction of PHB from DW followed the procedure conducted in study made by Prof Kumar Sudesh [14]. Approximately 10 gram of DW was dissolved in chloroform solution with ratio of 1:100 (1 gram of DW: 100 mL of chloroform) and keeps stirring for 5 days. The solution then was filtered using No 1 Whatman filter paper (42.5 mm diameter) filter paper to remove cell debris and then concentrate using rotary evaporator (Heidolph) with 60 rpm at 27°C. Approximately 0.3g of PHB obtained then was subjected to methanolysis with ratio 1:10 (1 g of PHB: 10 mL of methanol). The extracted PHB was finally dried in oven at 45°C. The solution blending method using chloroform (CHCl<sub>3</sub>) as co-solvent was carried out according to previous method prepared by [9] with some modification. The PHB and PEG were dissolved in chloroform with total polymer concentration was 2 wt%. The PEG solution was kept stirred for 1 hour at 27°C. Since PHB is insoluble in chloroform at room temperature, the solution was prepared at 60 °C followed on method prepared by [15]. After PHB and PEG completely dissolved, both polymer solutions were mixed with different blend ratio of 100/0, 80/20, 60/40 and 50/50 by % v/v which are indexed as PHB 100, PHB 80, PHB 60 and PHB 50 at 27°C. The solutions were mixed and stirred using magnetic stirrer for 8 hours at 50 rpm. The homogeneous solution then was cast on a horizontal glass surface followed by slow evaporation. The readily films then was placed in sealed bottle for further tests. Table 1 showed the detailed of PHB/PEG blends.

Table 1. The Details of PHB/PEG Blends

Code	Blend Composition PHB/PEG	Solutions
PHB 100	100/0	100 % v/v of PHB (Bare PHB)
PHB 80	80/20	80 % v/v of PHB, 20 % v/v of PEG
PHB 60	60/40	60 % v/v of PHB, 40 % v/v of PEG
PHB 50	50/50	50 % v/v of PHB, 50 % v/v of PEG

### Experimental Techniques

The thermal analyses of polymers were performed using Differential Scanning Calorimetry (DSC 910). The procedure was performed under protection of liquid nitrogen cooling system. The operating temperature range for polymers was between -50 °C to 220 °C with heating rate 10 °C/min. Thermogravimetric Analysis (TGA) test was performed on a Perkin Elmer instrument under nitrogen atmosphere. Measurement of all polymers was carried out with heating rate 10 °C/min at temperature range between 20 °C to 800 °C. Temperature of the beginning mass loss was denoted as ( $T_{\text{onset}}$ ) and temperature of maximum mass loss was denoted as ( $T_{\text{max}}$ ). To study the rheological behaviour of polymers, solid form of PHB 100, PHB 80, PHB 60 and PHB 50 were directly loaded into Oscillating Disk Rheometer, (Physica 600, Italy) at desired temperature. The rheometer was equipped with round plates of 2.5mm diameter and the gap size between plates was always kept at 1mm.

## RESULTS AND DISCUSSION

### Thermal Stability

The measurement The Figure 1 showed the DSC curves while Table 2 showed the data obtained for PHB 100, PHB 80, PHB 60 and PHB 50. Data in Table 2 were in double repeated and the average was reported.

Referring to Figure 1, the ( $T_m$ ) of PEG 100 are 54.97 °C which is a little lower than was obtained by [16] about 64.9 °C. In Figure 1 also showed the ( $T_m$ ) of PHB/PEG blends decreased with increased of PEG contents. It seen that PHB 100 has ( $T_m$ ) at 157.69 °C and adding PEG does change the ( $T_m$ ) of the PHB/PEG blends. Decreased of ( $T_m$ ) would improve thermal stability of polymer as stated by [17]. Reduction in ( $T_m$ ) offers in increases size of the (limited) processing window and decreases amount of degradation that can occur [18]. As displayed in Table 2, crystallinity ( $X_c$ ) of all PHB/PEG blends also were decrease than PHB 100. According to [19] this may attributed to reduction in crystal size, changes in surface free energy or PEG crystal structure changing.

As seen in Table 2, PHB 50, PHB 60 and PHB 80 have two endothermic melting peaks where; at ( $T_{m1}$ ) about 128.45 °C and 144.31 °C 150.59 °C belonged to melting of PHB imperfect PHB crystal and the highest one at ( $T_{m2}$ ) about 154.82 °C, 157.61 °C and 157.48 °C corresponds to melting of PHB crystal formed from recrystallization during heating cycle. Presence of multiple melting peaks was also known as melting-recrystallization-remelting process (MRR). A similar trend was also obtained in [20] where multiple melting peaks were

observed for each PHB incorporated with plasticizer (PEG). This may be resulted from lamellae rearrangement resulting from greater mobility provided by plasticizer. The tendency of PHB phase to crystallize is less intense for PHB 60 and PHB 50 which have less PHB contents resulting lower value of crystallinity ( $X_c$ ). The PHB 60 and PHB 50 showed decreased of ( $X_c$ ) about 23% to 26% in relation to PHB 100 in PHB phase may indicate faster crystallization process. The PHB 80 also showed reduction in crystallinity ( $X_c$ ) about 1.6% in relation to PHB 100. Higher PHB content in PHB 80 may cause this little reduction in crystallinity ( $X_c$ ) while lower PHB contents such as PHB 60 and PHB 50 have more reduction in crystallinity resulting from limited molecules organization in forming PHB crystal during crystallization and recrystallization process [19].

The main finding obtained from DSC result is addition of PEG decrease the ( $T_m$ ) and crystallinity ( $X_c$ ) of PHB/PEG blends. Although PHB 100 has high ( $T_m$ ) and crystallinity ( $X_c$ ) than PHB/PEG blends may indicate that PHB 100 has rigid structure but highly brittle.

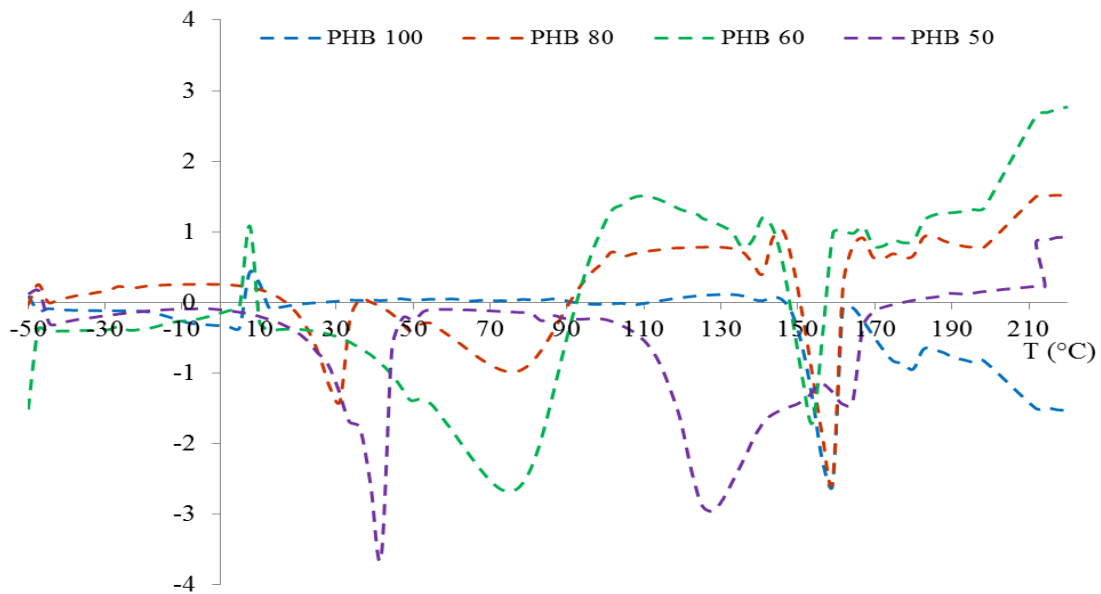


Figure 1. DSC Curves of PHB 100, PHB 80, PHB 60 and PHB 50

Table 2. Data Obtained from DSC for PHB 100, PHB 80, PHB 60 and PHB 50

PHB/PEG	T <sub>c</sub> (°C)	Cooling		Heating	
		ΔH <sub>c</sub> (Jg <sup>-1</sup> )	X <sub>c</sub> (%)	T <sub>m1</sub> (°C)	T <sub>m2</sub> (°C)
PHB 100	106.06	59.57	40.80	157.69	
PHB 80	110.83	57.26	39.21	150.59	157.48
PHB 60	103.10	26.27	17.99	144.31	157.61
PHB 50	94.98	21.18	14.50	128.45	154.82
PEG 100	25.78	97.75	37.31	54.97	

Figure 2 displayed the TGA curves for PHB 100, PHB 80, PHB 60 and PHB 50. The temperature of the start degradation (T<sub>onset</sub>) and the maximum peak (T<sub>max</sub>) for all PHB blended are summarized in Table 3.

As seen in Figure 2, there are three decomposition patterns were obtained. Overall, mass loss of all PHB blends is slightly higher than PHB 100. The first mass loss happened between temperature ranges from 30 °C to 100 °C. It is due to evaporation of chloroform and water during synthesis that physically adsorbed onto solid PHB/PEG blends sample interlayers. As reported in studies performed by [21], they believed that the mass loss below 100 °C was possibly due to water evaporation including loosely bound water during initial process of heating. Except PEG 100, all tested polymers including PHB/PEG blends presented first mass loss located in the temperature range comprised between 30 °C to 100 °C. The second decomposition corresponds to the degradation of PHB at temperature ranges from 262.62 °C and 273.75 °C and the third resembles to degradation of PEG at temperature ranges 368.59 °C and 377.85 °C.

Specifically, the T<sub>onset</sub> moves to lower values for all PHB/PEG blends that are quite similar to T<sub>onset</sub> of PHB 100. The T<sub>onset</sub> presented in Table 2 showed that an addition of PEG to the PHB blends leads to decreased T<sub>onset</sub>. According to [22], incorporation of PEG was found to compromise thermal stability of PHB through decreased the T<sub>onset</sub>. This may be due to hydroxyl group in PEG which may accelerate the PHB degradation. The decreased trend in the T<sub>max</sub> related to PHB and PEG phase were observed in mostly PHB blends. Quite similar results also was obtained by [23] for PLA - PEG blends. In their studies, PLA was blended with different ratio of PEG (10%, 15% and 20%). Their findings showed that the T<sub>max</sub> of neat PLA (366.9 °C) much higher than blends (358.3 °C, 358.6 °C and 351 °C) at different ratio of PEG (10%, 15% and 20%).

From Figure 2, a significance shift to higher temperature was observed for all PHB blends with addition of PEG although value is a bit lower than PEG 100. The PHB 80 which had low PEG content showed higher onset degradation temperature (Table 3). The mass loss of PHB 80 started around 273°C under nitrogen atmosphere and the remaining mass loss of the sample reached 2% at around 406°C. The TGA curves of PHB 80 shifted significantly toward the higher temperature regions compared to PHB 100. The mass loss for PHB 60 started at around 265°C under nitrogen and no carbonaceous residue were left at around

402°C while mass loss for PHB 50 occurred at 262°C and the remaining mass loss was a little higher than PHB 60 around 5.4% at 404°C.

It can be observed that residual weight for PHB 80 at similar temperature was higher compared to other PEG blending. However, the thermal decomposition patterns of all PHB blends followed considerably different pattern from PHB 100 and as the PEG content in blends increased up to 50%, their thermal stability as measured by T<sub>max</sub> slightly changed from 406.35°C to 404.06°C (Table 3). The thermal properties parameters such as T<sub>onset</sub> and T<sub>max</sub> of the blends suggest that PEG affects the PHB properties in blends in two aspects: a slight decreased in melting temperature and increase in the thermal stability. It could be concluded that addition of PEG able to delay the thermal degradation of PHB as shown by the shift to weight loss to higher temperature.

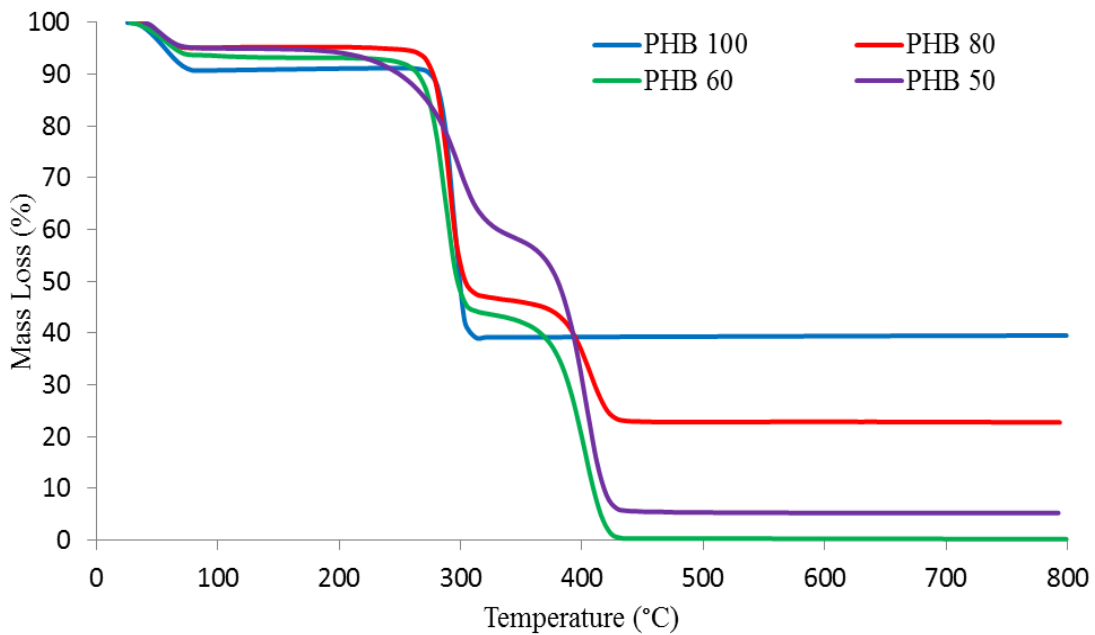


Figure 2. The TGA Analysis of PHB 100, PHB 80, PHB 60 and PHB 50

Table 3. Results of Thermal Analysis of PHB/PEG

	PHB		PEG	
	T <sub>onset</sub> (°C)	T <sub>max</sub> (°C)	T <sub>onset</sub> (°C)	T <sub>max</sub> (°C)
PHB 100	277.65	295.89		
PHB 80	273.75	289.29	375.89	406.35
PHB 60	265.99	288.96	368.59	402.60
PHB 50	262.62	299.26	369.70	404.36
PEG 100			395.98	411.56

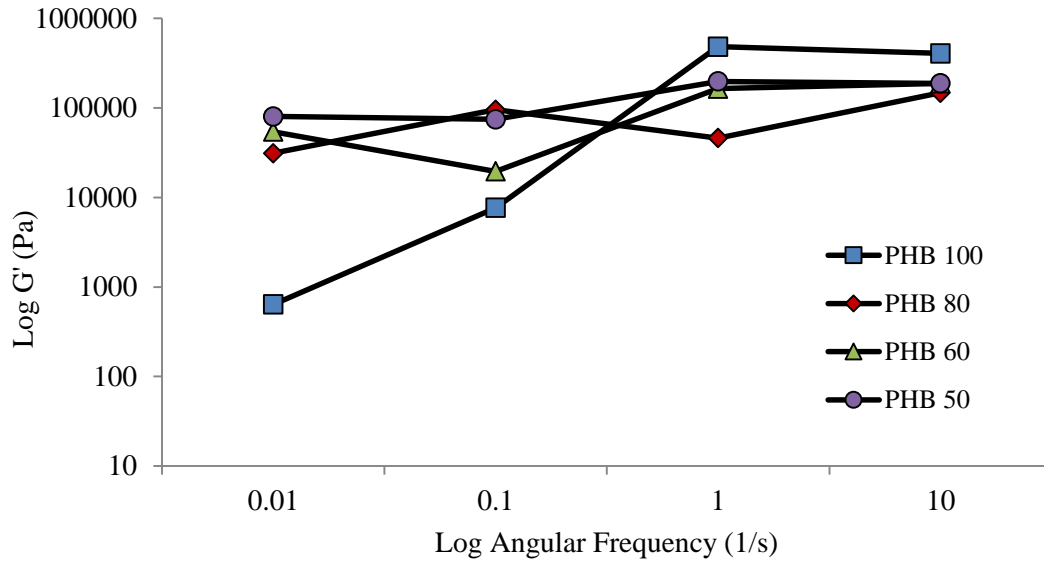
### **Rheological Analysis**

The effect of temperatures (150°C and 170°C) on storage modulus ( $G'$ ) and loss modulus ( $G''$ ) for PHB 100, PHB 80, PHB 60 and PHB 50 were illustrated in Figure 3 and Figure 4. As seen in Figure 3, the  $G'$  values increased along with frequencies. It is clear that  $G'$  values exhibited their highest magnitude at 150°C. At 170°C, values of  $G'$  decreased significantly. This happened attributed to increase restriction on PHB 100, PHB 80, PHB 60 and PHB 50.

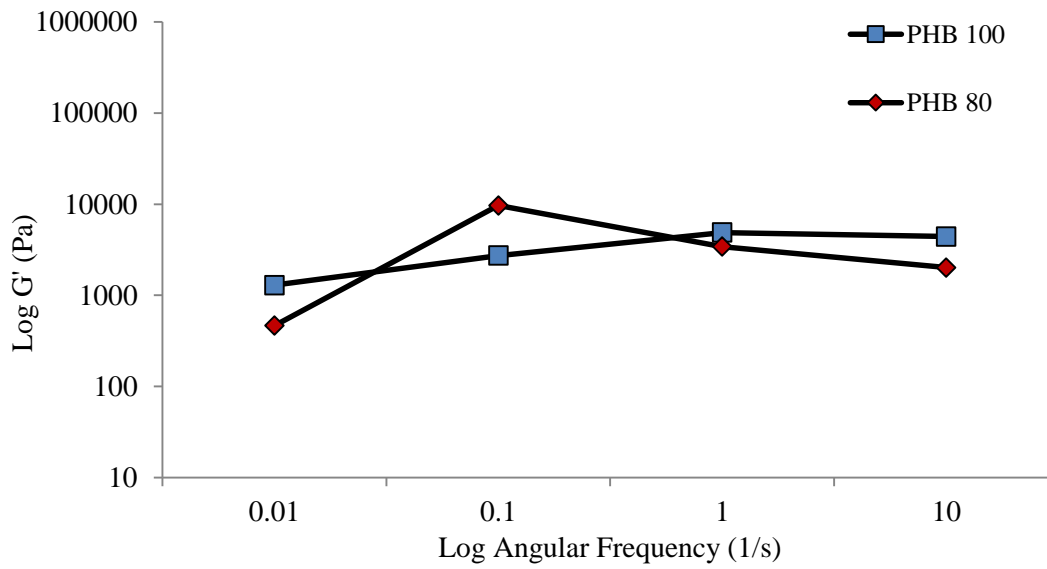
As illustrated in Figure 3 (a) higher PEG content (PHB 60 and PHB 50), the  $G'$  showed higher value than the  $G'$  of PHB 100 at lower range of frequency (0.01 to 0.1 s<sup>-1</sup>). Similar result was seen for Figure 4 (a). It seen that blends had strong dependency on frequency especially at higher range of frequency compared to PHB 100 indicating structure of blends less elastic than the PHB 100 structure. Referring to Figure 3 (b), the reason of PHB 100 showed higher value of  $G'$  especially at 170°C may be due to still have some capacity to store energy during the applied force. At this moment, PHB 100 might act like an elastic solid thus hinder the material to flow. At 170°C to 180°C, PHB 100 did not completely melted and this can be confirmed through visual inspection and little noisy during measurement.

And to PHB 80, both moduli for blend in Figure 3 and 4 continually drop due to applied force to blend sample is higher than inter particles forces and cause the blend structure collapses. As a result, the mechanical energy dissipated and cause blend to melt. This is confirmed through visual observation where at 170°C, PHB 80 is completely melted. There is liquid filled on the plate after a few second of sample loading. Similar observation was made with PHB 60 and PHB 50 where both blend melted at 160°C.

As seen in both Figure 3 and Figure 4, addition of PEG into PHB structure does effect both  $G'$  and  $G''$  of PHB/PEG blends. This phenomena had been previously discussed by [25]. Reduction in both  $G'$  and  $G''$  of PHB/PEG blends may be due two reasons. They made an assumption that addition of PEG content reduced the flow resistance between the PHB molecular chains. As a result, improved PHB mobility indicating through decreasing  $G'$  and  $G''$  values. Second, the concentration of long molecular chain of PHB in PLA/PEG blend decreased with increased PEG content. As a result, weaken the molecular chain of PHB/PEG blends. A similar result was obtained in [25] when study influence of  $G''$  on PLA/PEG blends. They found out that neat PLA achieved higher both  $G'$  and  $G''$  compared to PLA/PEG blend. Other reason that may contribute to reduction in  $G'$  and  $G''$  of PHB/PEG blends is crystallinity ( $X_c$ ).



(a)



(b)

Figure 3. Storage Modulus ( $G'$ ) vs Frequency for PHB 100, PHB 80, PHB 60 and PHB 50 at (a) 150°C and (b) 170°C



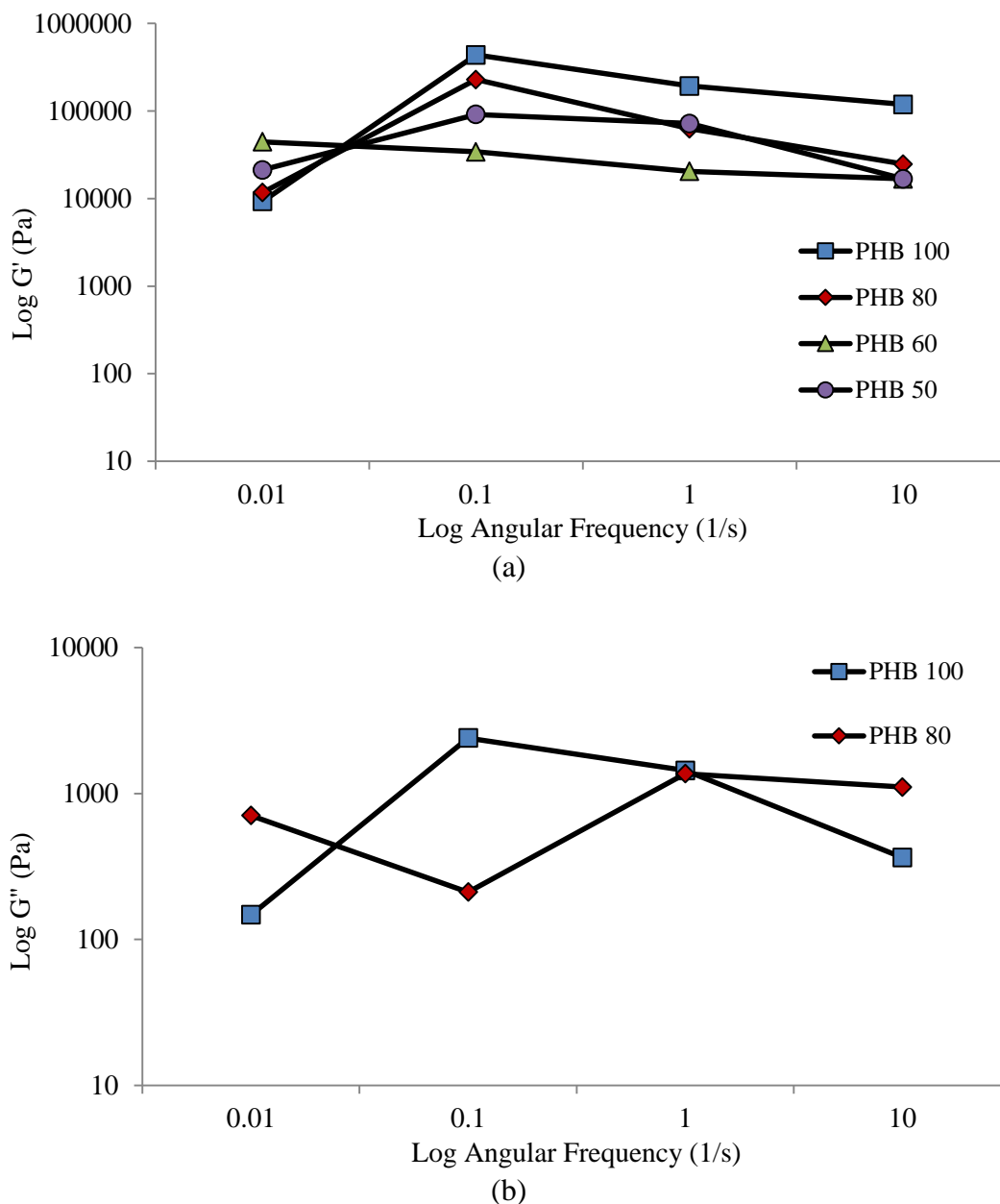


Figure 4. Loss Modulus ( $G''$ ) vs Frequency for PHB 100, PHB 80, PHB 60 and PHB 50 at (a) 150°C and (b) 170°

### Stability of Hybrid Nanofluids

Many methods have been developed to evaluate the stability of hybrid nanofluids. The simplest method is sedimentation method [28]. The investigation on stability is a key issue that influences the properties of hybrid nanofluids for application. A new method is used to estimate the suspension concentration with the increasing the sediment time. Figure 6 shows the peak absorbance of hybrid nanofluids appears at 364 nm. The absorbance of hybrid nanofluids decreases with increasing sediment time.

## CONCLUSIONS

The thermal and rheological behavior studies of blends PHB 100, PHB 80, PHB 60 and PHB 50 have been conducted. It was noticeable that addition of PEG in PHB does affect thermal and rheological behavior of polymers. Thermal stability study using DSC showed that both melting temperature and crystallinity of PHB/PEG blends decreased. For high PEG content; PHB 60 and PHB 50 have lower crystallinity than PHB 100 resulting from faster crystallization rate. PHB 100 has high crystallinity indicating rigid structure with brittle properties. The TGA result demonstrates that presence of PEG able to delay thermal degradation of PHB shown by the shifting of weight loss to higher temperature. The PHB 100 showed fast thermal degradation compared to others. As observed in the rheological test, PHB 100 has higher value of  $G'$  compared to blends at higher frequency and higher temperature (170°C) thus indicates the rigid structure of PHB 100 compared to blends. Increased adding PEG into PHB also showed reduction to  $G''$  with increased of frequency and temperature as flow resistance between PEG and PHB molecular chain reduced thus improved the mobility. It could be concluded that addition of PEG helps to improve PHB thermal behaviour thus broadened its application in industries.

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