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Paper title: Effect of soybean oil on dynamic mechanical properties of Epoxy/Polyurethane IPNs

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Effect of soybean oil on dynamic mechanical properties of Epoxy/Polyurethane IPNs

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Abstract
A series of semi-interpenetrating polymer networks (SIPN) based on bisphenol A epoxy resin and thermoplastic polyurethane was prepared by solution blending. Epoxidized soybean oil (ESO) was used to modify the IPNs in this study. Polymer/ESO ratio of the system was changed from 100/0 to 70/30. The Effect of soybean oil content on morphology and dynamic mechanical properties of the system was studied systematically. Results revealed that the Epoxy/Polyurethane system with soibean oil showed lower glass transition temperature, lower crosslink density and higher toughness.
Keywords: Soybean oil, Epoxy/Polyurethane, Interpenetrating Polymer Network

Introduction
Epoxy (EP) resin is one of the most important thermoset resins for composite materials due to its high performance, cheap price, high mechanical strength and hardness. However, the highly crosslinked nature of cured epoxy produces some undesirable characteristics such as brittleness. This issue restricts the use of epoxy resin in many applications. In contrast, thermoplastic polyurethane (TPU) is a flexible and rubbery polymer with chemical resistance and good processability. Nevertheless, pure PU has some disadvantages, such as low mechanical strength and poor heat resistance. Making IPNs is one of the best ways to overcome these problems. IPNs are unique "blends" of crosslinked polymers in which at least one network is synthesized and/or crosslinked in the presence of the other [1]. EP/PU IPNs combine the advantages of polyurethane and epoxy resin. They show excellent mechanical properties and thermal stability due to the synergistic effects induced by the formation of IPNs [2]. The target of this study was to determine the effect of ESO on the morpholigy and dynamic mechanical properties of EP/PU IPNs.

Experimental
Materials: The following materials were used in this study. Diglycidyl ether of bisphenol A (DEGBA) epoxy resin, KER-828 was purchased from Kumho P&B Chemicals Inc. TPU (MDI-polyester/polyether polyurethane) with a glass transition temperature of -40°C was obtained from Sigma-Aldrich. A modified aliphatic amine (JOINTMINE 238) was used as the curing agent. ESO was obtained from Sajo Haepyo Co. and N,N-dimethylformamide (DMF) solvent was from Merck. **EP/PU/ESO IPN Preparation.** First, a certain amount of epoxy resin was dissolved in DMF by a magnetic stirring for 30 minutes. Then a predetermined amount of PU was added to the solution and stirred for 24 hours to obtain a homogenous solution. After that ESO (10 and 30%) was added and stirred for 4 houers. Then a stoichiometric amount of hardener was added to the system and stirred for half an hour. The solution was cast on a preheated polyethylene plate. Finally, samples were placed at room temperature for 24 hours and then at 60°C for 4 hours to complete the curing process. Table 1 shows the composition of EP/PU/ESO IPNs. Dynamic mechanical analysis (DMA) was carried out on a DMA 242 C analyzer (NETZSCH Instruments, Germany) with tension mode over a temperature range from −80°C to 180°C at a heating rate of 5°C/min at 1 Hz. Three rectangular specimens (0.5 cm×2.5 cm×0.6 mm) for each sample were used for the test. Scanning electron microscopy (SEM) images were obtained on a Leo VP-1450 (Germany) operated at 20 kV.

Table 1- The composition of the IPNs prepared in this study

Sample	SIPN1	SIPN2	SIPN3
TPU (wt%)	20	20	20
EP (wt%)	80	80	80
ESO based on total polymer (wt%)	0	10	30

Results and discussion

The cross-sectional morphologies of pure EP, EP/PU and EP/PU/ESO IPNs are shown in Figure 1. From Figure 1(A), it can be seen that for pure EP, the surface is very smooth which implies a brittle fracture and is attributed to the poor toughness of the neat EP. According to Figure 1(B), with the addition of TPU, the fractured surface of EP/PU IPNs becomes uneven and somehow rough due to the effect of soft segments of polyurethane. Figure 1(C,D) shows the morphologies of EP/PU/ESO IPNs at different magnification. Presence of dimples and voids in cross-sectional surface of the modified samples shows that toughness has increased significantly by addition of soybean oil. The epoxy groups of ESO are less reactive than those of EP. As a result, when the homogeneous mixture of EP and ESO is cured, the epoxy groups of EP react quickly and form networks. As phase separation is stopped after gelation, the ESO-rich phase cannot undergo phase separation and thus simply imparts a plasticizing effect on EP. DMA was carried out to study the dynamic mechanical properties of the IPNs and the loss factor as a function of temperature is shown in Figure 2. As could be seen, the tan δ peaks of all the IPNs associated with the glass transition temperature (Tg) exhibits a monodisperse peak which indicates that no phase separation took place and confirms the results of SEM. The detailed DMA results of EP/PU/ESO IPNs with different component ratios are summarized in Table 2. The PU/EP IPN showed lower Tg than pure EP (89.7°C), which could be ascribed to the presence of PU with a low Tg and its contribution to the deppression of the IPNs' Tg. It is clearly observed that the Tg of EP/PU/ESO IPNs decreased significantly when a small amount of soybean oil was added, which is due to the long aliphatic chains of soybean oil and its plasticizing effect [3]. Moreover, this can be traced to the fact that ESO reduces IPNs crosslink density and consequently decreases Tg. It should be noted that although the addition of ESO to the EP/PU/ESO IPNs led to a decrease in the Tg, the increase of ESO from 10 to 30 wt% did not affect the Tg significantly.

Conclusions

EP/PU/ESO IPNs were prepared via solution blending by adding different amounts of ESO to EP/PU (80/20) blend. The results of SEM and DMA analysis showed that the prepared EP/PU/ESO IPNs had lower Tg and enhanced toughness compared to pure EP. Moreover, the increment of ESO from 10 to 30 wt% decreased the crosslink density of the IPN but did not change the Tg considerably.

References

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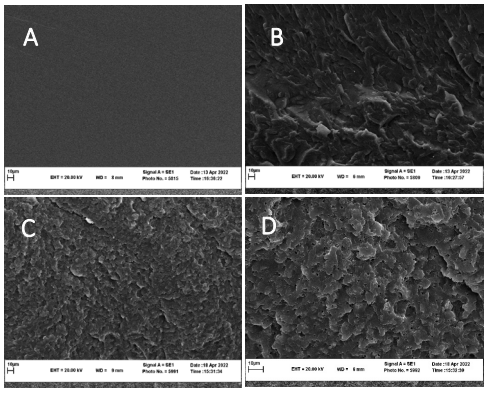

Figure 2- SEM micrographs of: (A) pure EP, (B) SIPN1 (C, D) SIPN2 at different magnification.

Table 2. Data obtained from DMA analysis of samples

Sample	Tg (℃)	E′ (Tg+30) (Mpa)	Crosslink density (kmol m^{-3})
SIPN1	83.3	11.70	1.21
SIPN2	71.3	8.12	0.87
SIPN3	70	6.03	0.65

cross-linking density (ν) , where E' is the storage modulus at rubbery plateau, R is the = E′/3RT universal gas constant (8.314 J K−1 mol−1) and T = Tg+30 (K)

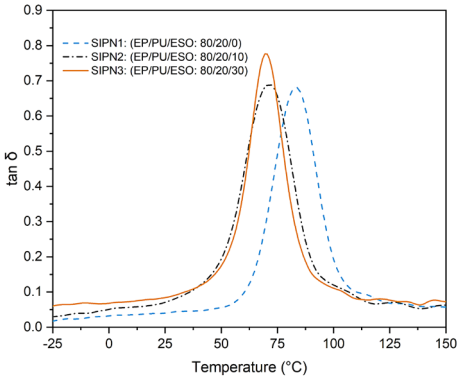

Figure 1- DMA plots of EP/PU/ESO IPNs at 1 Hz