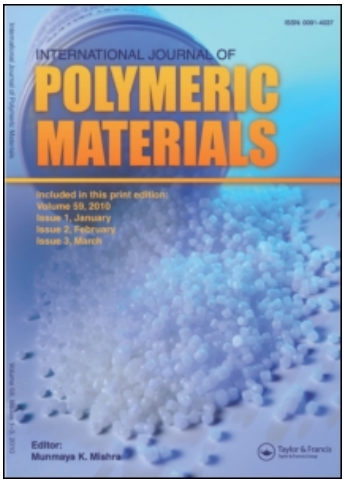

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Oil Palm Empty Fruit Bunch – Polypropylene Composites – The Effect of Modified Alcell Lignin as a Compatibilizer on Tensile Properties

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ALCELL lignin has been employed as compatibilizer in the empty fruit bunch (EFB)-polypropylene (PP) composites. The lignin has been chemically modified with toluene diisocyanate (TDI) to various weight loadings. The effect of lignin as compatibilizer on the tensile properties has been studied. The results show that the TDI-modified lignin is able to impart greater compatibility between EFB and PP. This is reflected in the greater tensile properties shown by the composites with TDI-modified lignin than the ones with the unmodified lignin.

Keywords: Polypropylene; composites; oil palm; tensile properties; compatibilizers; lignin

1. INTRODUCTION

It is well known that compatibility between lignocellulosic material and polymer plays a crucial role in determining the properties of a composite. The compatibility and interfacial bond strength between lignocellulosic material and polymer matrix are expected to be poor in composites of lignocellulosics-thermoplastics. This is due to the hydrophillic and hydrophobic nature of lignocellulosics (contributed by

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hydroxyl groups in cellulose, lignin and hemicellulose) and thermoplastics, respectively. Various chemical reagents have been employed to enhance the compatibility between the constituent materials. These include Epolene, E-43 (maleic anhydride modified-polypropylene) [1], poly[methylene (polyphenyl isocyanate)] (PMPPIC) [2], γ-methacryloxy-propyltrimethoxysilane (A-174) [3], vinyltri (2-methoxy ethoxy) silane (A-172) [4], γ-aminopropyltriethoxysilane [5], poly(propylene-acrylic acid) [6] and poly(propylene-ethylene acrylic acid) [6]. In this study, lignin is used as a compatibilizer between empty fruit bunch fibres of oil palm (EFB) and polypropylene (PP) matrix. Since, lignin contains polar hydroxyl groups and non-polar hydrocarbon and benzene rings, it is believed that it can play a role in improving the compatibility between both components. The compatibility is expected to be further enhanced by chemically modifying the lignin with appropriate chemical reagent.

2. EXPERIMENTAL

2.1. Materials

EFB in fibre form was obtained from Sabutek (M) Sdn. Bhd., Teluk Intan, Perak, Malaysia. PP used was purchased from Polypropylene Malaysia Sdn. Bhd., Malaysia with melt flow index and density of 12.0 g/10 min and 0.903 g/cm^3, respectively. ALCELL® lignin was obtained from Repap Technologies Inc., USA. Toluene diisocyanate (TDI) was purchased from Chembumi (M) Sdn. Bhd., Penang, Malaysia.

2.2. Methods

2.2.1. Reaction of TDI and Lignin

Lignin was diluted with CCl_4, and added with dibutyl tin dilaurate as a catalyst, and put in a dropping funnel. TDI was placed in a round bottomed flask of capacity 500 ml. The lignin solution was added dropwise into TDI under constant stirring until the addition of lignin solution was complete (Fig. 1). The TDI-modified lignin produced was then washed with acetone under reflux for 3 hours, to remove

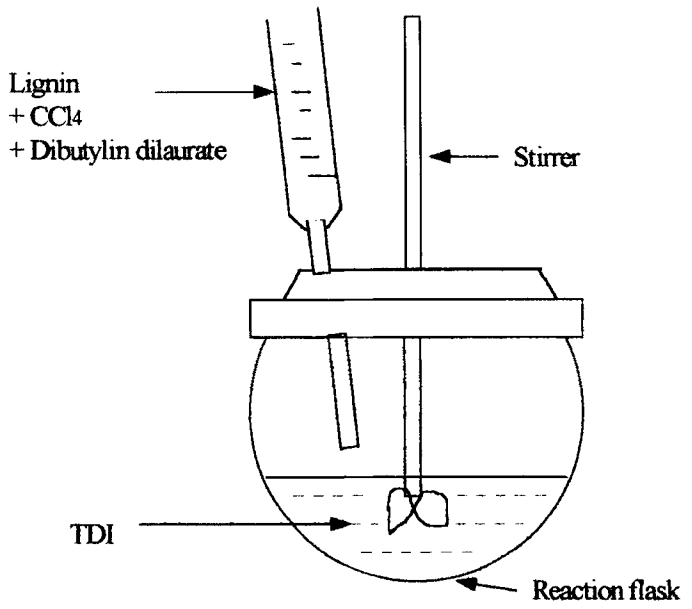

FIGURE 1 Experimental set-up for TDI-lignin reaction.

unreacted chemical reagents. The lignin was oven-dried at 80°C before being used in composite making.

2.2.2. Filler Preparation

EFB filler was obtained in the form of long strands of fibres. The fibres were ground into small particles. Endecotts sieve was used to separate the particles into different sizes. The filler size used in this study was of mesh 35–60, *i.e.*, 270–500 μm.

2.2.3. Filler Treatment

Three levels of lignin were employed: 1, 5 and 10% (based on the weight of the EFB filler). Lignin powder (unmodified or TDI-modified) was dissolved in ethanol (15% solution) before being mixed with the filler. The mixture was dried in an oven at 80°C for 3 hours to allow for complete evaporation of the ethanol.

2.2.4. Compounding and Processing

The compounding of EFB filler and PP was carried out using a Haake Rheocord System consisting of a Haake Rheodrive 5000 (drive unit) and Haake Rheomix 600 with roller blade (mixer). The mixing was carried out at 170°C for 20 minutes with a rotor speed of 25 rpm. The weight ratio of EFB to PP is 40 : 60. The compound was then transferred to a mould with dimensions of 160 × 160 × 3 mm. The compound was preheated for 10 minutes at 160°C followed by hot-pressing at the same temperature for another 10 minutes. Cooling was carried out for 5 minutes under pressure.

2.2.5. Testing

Tensile tests were conducted according to ASTM D618. The sheet produced was cut into test samples with the dimensions of 15 × 1.9 × 0.3 cm (length × width × thickness). The test was carried out using Universal Testing Machine at a cross-head speed of 5 mm/min. Tensile toughness was determined by measuring the area under stress-strain curve.

3. RESULTS AND DISCUSSION

3.1. Chemical Modification of Lignin with TDI

Table I shows that the chemical loading of TDI in the lignin increases with the increase in TDI/lignin ratio.

Evidence that chemical reaction between lignin and TDI has taken place is seen in Figures 2, 3 and 4 (unmodified lignin, 2% TDI-modified lignin and 18% TDI-modified lignin, respectively). TDI-modified lignin was thoroughly washed in acetone and dried prior to the FTIR analysis. Thus, any unreacted reagent and urethane residues

TABLE I Chemical loading of TDI in the lignin *vs.* TDI/lignin ratio (weight/weight)

TDI/lignin	*Chemical loading* (%) (*based on the oven-dried weight before reaction*)
1/2	2
2/3	13
1/1	18

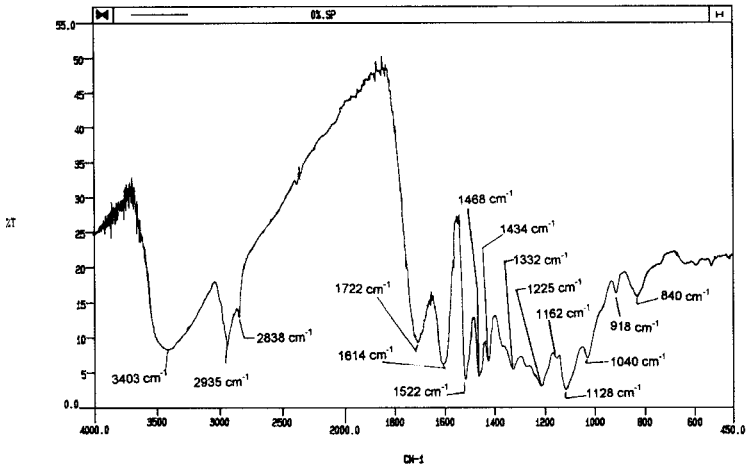

FIGURE 2 FTIR spectrum of unmodified lignin.

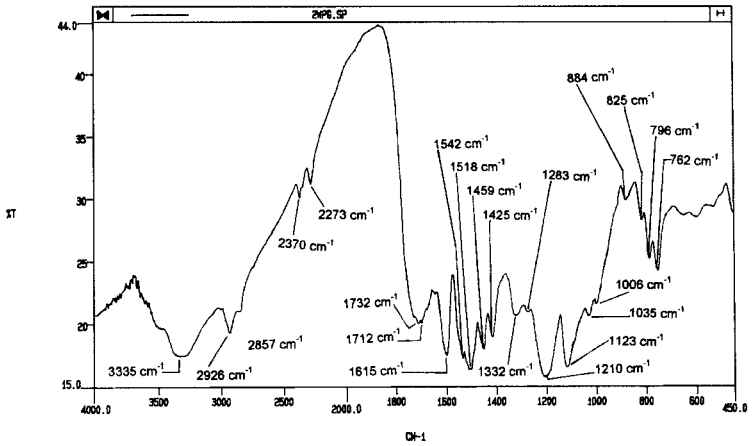

FIGURE 3 FTIR spectrum of 2% TDI-modified lignin.

formed during the reaction would be removed by this procedure. The spectra for TDI-modified lignin both at 2% and 18% loading show a small peak at about 1542 cm^{-1}, which is due to the N-H deformation frequencies of secondary amines [7]. However, there are two strong peaks at about 762 and 796 cm^{-1}, which are due to N-H deformation of bonded secondary amines [7]. Although it is not conclusive, peaks at

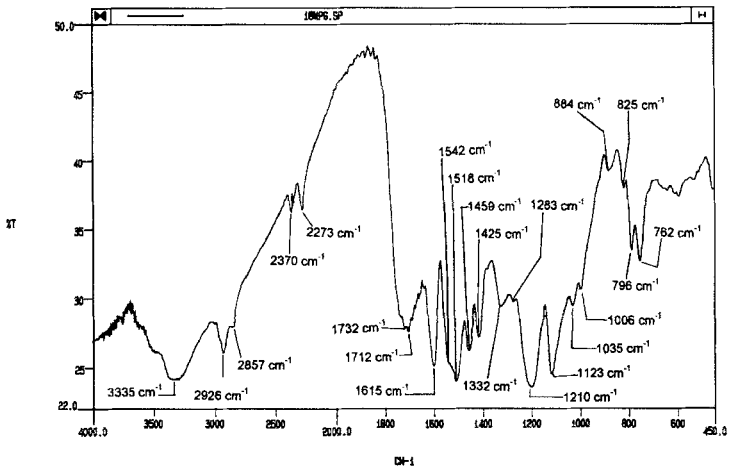

FIGURE 4 FTIR spectrum of 18% TDI-modified lignin.

825, 883 and 1006 cm^{-1} may show evidence of substituted benzene ring of lignin [8]. Peaks at about 2273–2370 cm^{-1} show evidence of unreacted —N=C=O groups of TDI. The strong absorption at about 3335 and 2926 cm^{-1} in all of the FTIR spectra is due to hydroxyl absorption. Since the substitution of TDI is not high enough to eliminate all hydroxyl groups, these bands are always present.

3.2. The Effect of TDI-modified Lignin as Compatibilizer on the Tensile Properties of EFB-PP Composites

Figure 5 shows the effect of the degree of TDI loading in the TDI-modified lignin on the tensile strength of the EFB-PP composites. The results demonstrate that all composites with TDI-modified lignin display higher tensile strength than the ones with unmodified lignin. It is clearly seen that the strength increases with the increase in the level of modification. It is also obvious that the strength of composites with the modified lignin show an increasing trend as the percentage of lignin is increased. However, composites with the unmodified lignin display a considerable reduction in the strength as the percentage of lignin is increased. Tensile modulus of the composites as shown in Figure 6 display the same trend as the tensile strength results.

The toughness of the composites is enhanced with the addition of TDI-modified lignin as opposed to the composites with unmodified

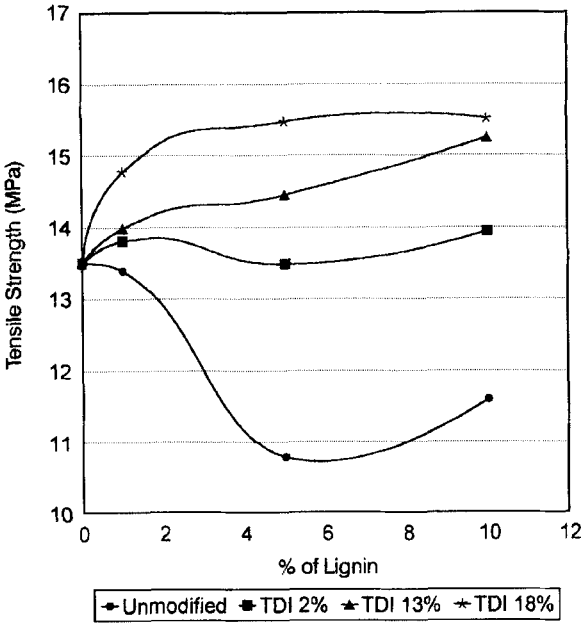

FIGURE 5 The effect of TDI loading in the TDI-modified lignin on the tensile strength.

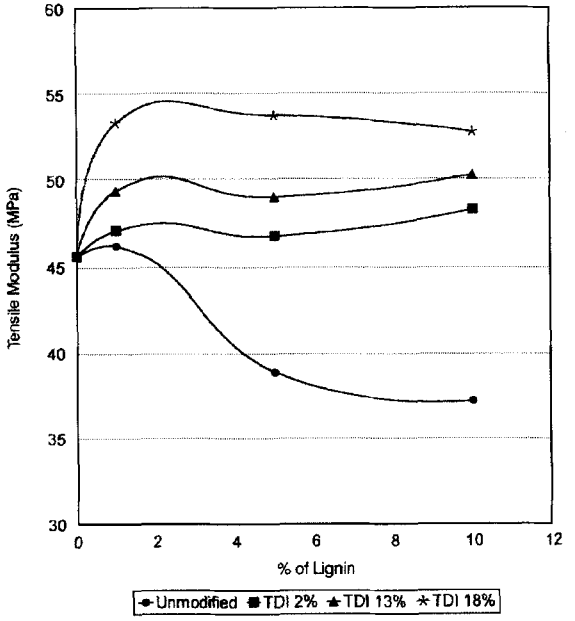

FIGURE 6 The effect of TDI loading in the TDI-modified lignin on the tensile modulus.

lignin (Fig. 7). The results show that the toughness increases as the degree of modification is increased. Thus, it can be inferred that more energy is needed to break the composite with the modified lignin than the ones with the unmodified lignin. This indicates that modified lignin is able to impart greater compatibility between EFB and PP, which requires more energy to break them apart.

Figure 8 shows the results of elongation at break (EB). It seems that the addition of lignin has no significant effect on the composites with modified lignin. However, the EB of the composites with unmodified lignin is significantly higher than the rest of the composites at 10 percent lignin loading. This phenomena is consistent with the tensile modulus results, where, the composites with unmodified lignin display significantly lower EB than the ones with modified lignin.

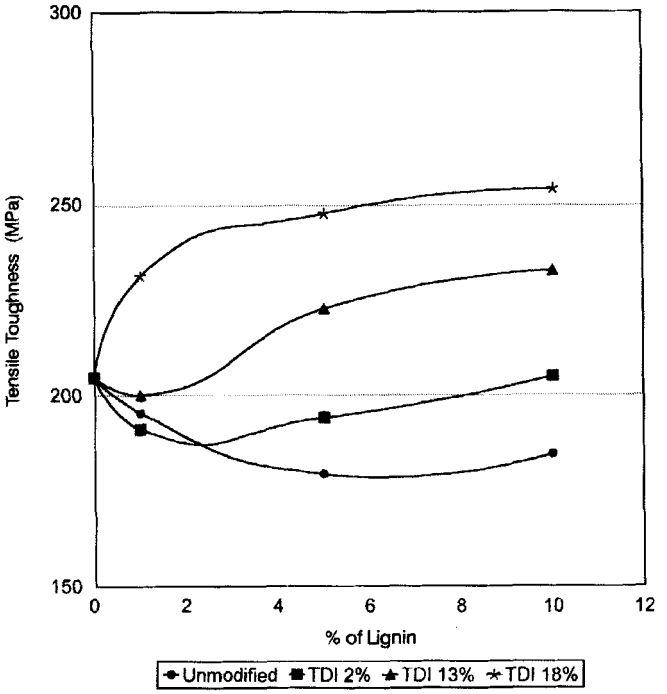

FIGURE 7 The effect of TDI loading in the TDI-modified lignin on the tensile toughness.

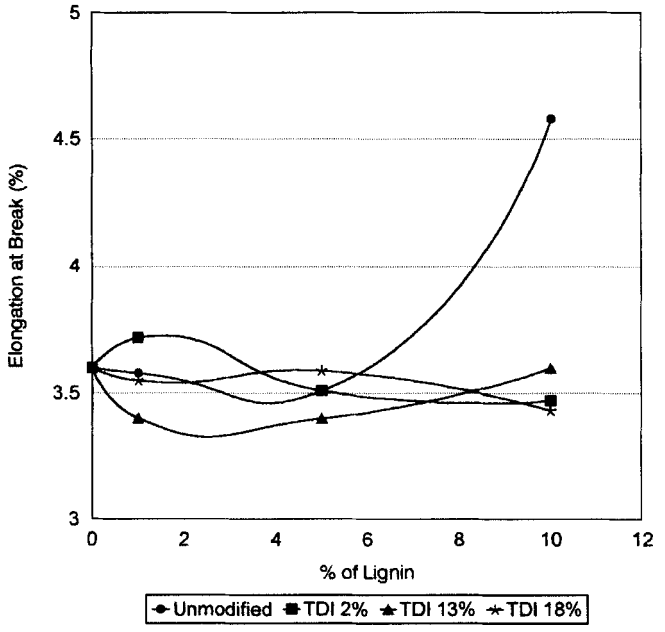

FIGURE 8 The effect of TDI loading in the TDI-modified lignin on the elongation at break.

4. CONCLUSIONS

1. From the results of TDI loading and the evidence from FTIR spectra, TDI is shown to chemically react with lignin. The degree of modification can be regulated by the TDI/lignin ratio.
2. Modification of lignin with TDI is able to impart greater compatibility between EFB and PP. This is reflected in the improvements shown in the tensile properties.

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