Novel Materials from Unsaturated Polyester Resin/Styrene/Tung Oil Blends with High Impact Strengths and Enhanced Mechanical Properties

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ABSTRACT: A novel material was prepared through the blending of an unsaturated polyester resin/styrene mix with tung oil, which offered improved impact strength, creep resistance, modulus, and hardness. A nanoindentation technique was used to investigate the mechanical properties. With the incorporation of 1 wt % tung oil into the unsaturated polyester matrix, the impact strength, modulus, and hardness increased by 15, 20, and 41%, respectively. The impact-fractured surfaces were examined

INTRODUCTION

Petroleum-derived unsaturated polyester (UPE) resins are widely used because of their low cost, ease of handling, and good combination of mechanical, electrical, and chemical properties. A recent trend has been the incorporation of natural oils (specifically vegetable oils) into a polymer matrix to tailor its properties for the generation of green materials and minimize the dependence on nonrenewable petroleum resources. There have been several reports on the modification of UPE resins via blending with rubber.^{1–5} However, the use of vegetable oils as biomodifiers in a polymer matrix can lead to the development of new materials. Some researchers have worked with vegetable-oil-modified resins.^{6–12} Lu et al.⁶ prepared a novel waterborne polyurethane with scanning electron microscopy. The dynamic mechanical analysis was performed with a nanoindentation technique. The storage and loss modulus values were determined under cyclic loading as a function of indentation. The flexural properties also significantly increased with the incorporation of tung oil. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 2174–2182, 2011

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from a rapeseed-oil-based polyol and used it to modify glycerol-plasticized starch to overcome the intrinsic drawbacks of starch materials (e.g., poor mechanical properties and water sensitivity). Mehta et al.⁷ used a blend of a UPE resin and functionalized vegetable oils to prepare biocomposites with hemp fiber as a reinforcement. They reported improvements in the properties with the incorporation of a derivatized vegetable oil. Can et al.⁸ prepared maleic anhydride modified, soybean- and castor-oil-based monomers. They maleinated the alcoholysis products of the oils with various polyols such as pentaerythritol, glycerol, and bisphenol A, and they copolymerized them with styrene to produce hard, rigid plastics. The polymers prepared from castor oil exhibited significantly improved moduli, strength, and glass-transition temperatures in comparison with soybean-oil-based polymers. Haq et al.⁹ synthesized biobased resins through the partial substitution of UPEs with an epoxidized soybean oil reinforced with nanoclay. The incorporation of epoxidized maleinated soybean oil led to an increase in the toughness but lowered the stiffness, whereas the incorporation of a nanoclay improved the barrier and thermal properties. Donell et al.¹⁰ used vacuum-assisted resin-transfer molding to make composite panels from a plant-oil-based resin

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(acrylated epoxidized soybean oil) and natural fiber mats made of flax, cellulose, pulp, and hemp. Phuong et al.¹¹ prepared biocomposites from maleic anhydride and epoxide-functionalized soybean oils that were cured in the presence of various biofibers (e.g., kenaf, Kayocels, protein grits, and Solka-Floc) by an amine catalyst. Rigid thermosets, characterized by a high-crosslink-density network and a high gel fraction, were obtained. Miyagawa et al.¹² incorporated an epoxidized methyl soyate into a UPE matrix. The storage modulus and heat deflection temperature decreased with increases in the epoxidized methyl soyate content, but the impact strength of the biobased UPE resin remained constant. Li et al.¹³ reported on soybean oil/divinylbenzene thermosetting polymers and their synthesis, structure, and properties (as well as the relationships of the latter). La Scala et al.¹⁴ used fatty-acid-based monomers as styrene replacements for liquid-molding resins. They reported that fatty acids could be used as styrene replacements in thermosetting polymers because of their capacity for free-radical polymerization. However, unsaturation sites along the fatty acid backbone reduced the cure rate, monomer conversion, and polymer properties.

Thus, in most of these works, it is clearly evident that the vegetable oils were first functionalized and then incorporated into the UPE matrix. New tung oil/styrene/divinylbenzene copolymers were prepared by Li and Larock.¹⁵ They also reported in detail improvements in the coefficient of thermal expansion and the compressive modulus. Biobased UPE/layered silicate nanocomposites were prepared by Haq et al.¹⁶ They reported in detail a proper stiffness-toughness balance and enhancements in other thermophysical properties.

A nanoindentation technique has emerged as an important tool for the characterization of polymeric materials in recent times. Hu et al.17 carried out nanoindentation studies on nylon 11/clay nanocomposites and reported the hardness and modulus values with different clay loadings. They compared the modulus data obtained by the nanoindentation technique with those obtained by a tensile method and dynamic mechanical analysis. They showed that nanoindentation is a simple but effective method for the mechanical characterization of polymers and polymer composites. White et al.¹⁸ carried out the viscoelastic characterization of polymers such as epoxy, poly(methyl methacrylate), and polydimethylsiloxane by instrumented indentation. They observed excellent agreement between the bulk rheological data and dynamic indentation data.

This work provides an overview of a study examining the use of an unmodified vegetable oil (tung oil) as a toughening agent in a UPE resin without its mechanical properties being sacrificed. Tung oil was blended with the UPE and styrene in three proportions (1, 2, and 5 wt %), and these blends were designated UPE_TO 1, UPE_TO 2, and UPE_TO 5, respectively (UPE_TO 0 indicates the blank). The blends were characterized with impact testing, and the impact-fractured surfaces were investigated with scanning electron microscopy (SEM). The mechanical characterization was performed by the nanoindentation technique. The nanoindentation technique was also used to determine the storage and loss moduli of the blend samples by cyclic indentation on the samples.

EXPERIMENTAL

Materials

A UPE resin with a styrene content of 40% (grade G P Fibrebond 333, Ruia Chemicals) was used. Methyl ethyl ketone peroxide and cobalt naphthenate were used as the catalyst and accelerator, respectively. Tung oil was kindly supplied by DIC India, Ltd., India.

Sample preparation

A metal mold $(140 \times 120 \times 5 \text{ mm}^3)$ was used for casting the resin and resin/tung oil blend laminates. A release agent (Waxpol India Ltd., India) was applied to the inner surfaces of the mold for the quick and easy release of the composite sheets. A measured amount of tung oil was uniformly blended with the UPE resin by stirring. The mix was then degassed *in vacuo* and mixed with the accelerator and catalyst (2% each). It was then poured into the mold and allowed to cure at room temperature for 24 h; this was followed by 4 h of postcuring in an oven at 100°C.

Characterization

The impact properties of the different blend samples were investigated according to ASTM D 256 with the help of WinPEN (Ceast SpA, Italy), and the mean of at least six samples was reported for each set. The surface morphology of the different cured blends was investigated with SEM (S 3400N, Hitachi) at a voltage of 15 kV with a gold coating. Nanoindentation was carried with a CSM NHTX S/N:55-0019 nanohardness tester with a Berkovich triangular diamond indenting tip. For dynamic mechanical analysis, the sinus mode was used, for which a load of 200 mN (loading/unloading rate = 400 mN/min, holding time/pause = 5 s) with a sinus frequency of 5 Hz and an amplitude of 10 mN was selected. The load/penetration depth curve of a particular indentation was obtained, and this could be switched to the dynamic mechanical analysis mode of the instrument. It was purely based on a spring-mass mechanical model involving a damper with a damping coefficient of 239.700 Ns/mm, a mobile mass of 11.272 g, and a spring stiffness of 1.070 N/mm. The storage and loss moduli (GPa) along with the elasticity (GPa) and hardness (MPa) as output parameters were obtained. The viscosities of the blends were measured with the help of a Brookfield DV-II+ Pro viscometer with an S-21 spindle. The viscosity measurements were performed between 20 and 24°C.

RESULTS AND DISCUSSION

UPE resins produce brittle materials on curing, and they very often need to be impact-modified to meet end-use requirements. Apart from the incorporation of a rubber phase into the UPE matrix, the biomodification of UPE is nowadays becoming an interesting area of research for material scientists. In this study, tung oil, a well-known drying oil extensively used in the surface-coating industries, was used as the biomodifier. Tung oil is a triglyceride of eleostearic acid (82%) with a conjugated triene structure, linoleic acid (8.5%) with a conjugated diene structure, oleic acid (4%) with mono-unsaturation, and palmitic acid (5.5%) with no unsaturation.¹⁹ Because of its unsaturated, conjugated structure, it undergoes rapid polymerization by both free-radical and cationic polymerization. A modification of the UPE resin was performed through the addition of tung oil in three different proportions (1, 2, and 5 wt %). The variation of the viscosity as a function of the tung oil content is shown in Figure 1. The addition of tung oil clearly decreased the viscosity significantly, and this could be ascribed to the plasticizing effect of the oil. A small quantity of the blend (5 mL) was subjected to gelation testing by the addition of 2 wt % methyl ethyl ketone peroxide and



Figure 1 Viscosity of the UPE resin and tung oil blends observed with a Brookfield viscometer.



Figure 2 Variation of the gelation time of the UPE resin and tung oil blends as a function of the tung oil content.

cobalt naphthenate. The variation of the gelation time with the tung oil content is shown in Figure 2. Tung oil, being highly reactive, crosslinked with the UPE resin and styrene monomer and lowered the gelation time. A probable crosslinking reaction of all the components is shown in Figure 3. Free-radical curing took place through the unsaturations present in all three components.

SEM analysis

An attempt was made to correlate the observed SEM microstructures of the bioblends with schematic diagrams in Figures 4–7. The fracture surface of the



Figure 3 Probable intercomponent crosslinking reaction in the bioblends.



Figure 4 Schematic diagram and SEM microstructure of the impact-fractured surface of UPE.

UPE (Fig. 4) had a rough morphology with some spots at which microgels were formed during curing [Fig. 4(a,b)]. Similar microgels were reported by Mehta et al.⁷ For UPE_TO 1 [Fig. 5(a,b)], the impact-fractured surface revealed yielding of the matrix, which was not observed in the UPE. This clearly indicates that the incorporation of long-chain fatty acid molecules in a small amount (1%) within the UPE and styrene matrix induced a yielding process in the matrix, and this increased the energy required for the fracturing of the surface.

In UPE_TO 2, a second phase seemed to be present in craters that contained the crosslinked molecules of the tung oil, UPE, and polystyrene [Fig. 6(a,b)]. Similar SEM observations were reported by Mehta et al.⁷ for a soybean oil methyl ester modified UPE resin. The fractured surface of UPE_TO 5 was significantly different from the surfaces of the other blend samples [Fig. 5(a,b)]. Here, the oil molecules were fully embedded within the UPE/styrene

blend matrix [Fig. 7(a,b)]. Highly crosslinked polymers resulted that were brittle and incapable of yielding.

Impact strength

The impact strengths of the bioblend samples are shown in Figure 8. The impact strength of UPE_TO 1 increased by 15% versus that of the UPE. The higher impact strength of UPE_TO 1 could be ascribed to the shear yielding of the matrix in the presence of 1% tung oil, which was observed from SEM microstructures [Fig. 5(a,b)]. With the incorporation of 1% tung oil, there was homogeneous blending of all the components, and more energy was absorbed through shear yielding. However, in UPE_TO 2, as observed from SEM photographs [Fig. 6(a,b)], the oil molecules clustered together, existed within the craters, and remained crosslinked with the UPE and styrene molecules. Hence, they



Figure 5 Schematic diagram and SEM microstructure of the impact-fractured surface of UPE_TO 1. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 6 Schematic diagram and SEM microstructure of the impact-fractured surface of UPE_TO 2. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

could not effectively take part in instantaneous energy absorption during impact testing, and the impact strength was only 2.5% higher than that of the UPE. In UPE_TO 5, the oil molecules were fully embedded within the matrix [Fig. 7(a,b)] and could not take part in instantaneous energy absorption; it exhibited 31% less impact strength than the UPE.

Nanoindentation tests

Nanoindentation of these bioblend samples was carried out, and the penetration of the indenter in the resin samples is shown in Figure 9. The test results are summarized in Table I. Figure 10 shows the load–penetration curves of the samples (UPE, UPE_TO 1, UPE_TO 2, and UPE_TO 5). The penetration of the UPE during nanoindentation testing was due to the mobility of the chain segments within the crosslinks, whereas in the blend matrix, the mobility of the fatty acid chain molecules might have contributed significantly. The penetration was associated with both elastic and plastic deformation. In the UPE and UPE TO 2, the curves showed a similar nonlinear nature up to a depth of 3700 nm; after that, the UPE curve had a steeper slope. The UPE showed 35% recovery after unloading, and UPE_TO 2 showed penetration up to 7500 nm and 41% recovery after unloading. The higher penetration and higher recovery of UPE_TO 2 could be attributed to the intermittent presence of oil molecules in craters, which facilitated deformation through their long-chain fatty acid segments. UPE TO 1 had a steeper slope and a higher recovery percentage (43.9%) in comparison with the UPE and UPE_TO 2. This could be ascribed to the homogeneous blending of the three components. Here, the energy absorbed through viscous dissipation was less, and the recovery percentage (43.9%) was highest. This recovery fully conformed to the impact strength, which was also highest. UPE_TO 5 exhibited an almost linear load-penetration curve. The oil molecules (5%) were crosslinked with UPE and styrene molecules and remained embedded within the matrix [Fig. 7(a,b)]. This increased the overall



Figure 7 Schematic diagram and SEM microstructure of the impact-fractured surface of UPE_TO 5. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 8 Impact strength of the UPE and bioblends.

rigidity of the material and led to an almost linear nature of the curve during loading. A larger number of fatty acid chains took part in energy absorption, and this was counterbalanced by high intercomponent crosslinking; the recovery was lowest (32%).

The loading curves were followed by a holding time of 5 s before unloading. The creep results (Table I) were almost comparable. The creep decreased in UPE_TO 1 by 0.7% versus the UPE, and this could be due to uniform blending of the oil with the resin components. For UPE_TO 2, the creep was almost the same as that of UPE, and this fully supports our previous observation that the oil molecules were not dispersed uniformly throughout the matrix but instead were confined within craters. In UPE_TO 5, the creep increased by 2.8% versus that of the UPE resin, and this might have been due to the mobility of the fatty acid chain segments. Similar



Figure 9 Optical, three-dimensional nanoindentation image of UPE.

TABLE I Maximum Penetration Depth and Recovery of the UPE/Tung Oil Blends with Nanoindentation

Sample	Maximum	Recovery	Creep
	penetration (nm)	(%)	(%)
UPE_TO 0	6816 5971	35.09 43.9	3.6
UPE_TO 2	7366	41.23	3.8
UPE_TO 5	6493	32.2	6.4

creep behavior was reported by Hu et al.¹⁷ for nylon 11/clay nanocomposites.

The variations of the modulus and hardness of the matrices, measured by nanoindentation testing, are shown in Figure 11(a,b), respectively. The modulus of UPE_TO 1 was 20% higher versus the UPE modulus, whereas those of UPE_TO 2 and UPE_TO 5 were 27 and 5.8% lower. The hardness values of UPE_TO 1 and UPE_TO 5 showed increases of 41 and 9%, respectively, versus the UPE hardness, whereas that of UPE_TO 2 showed a reduction of 11%. The dispersion of the tung oil molecules within the UPE/styrene matrix, intercomponent crosslinking, and the mobility of long-chain fatty acid segments were the key factors, and their relative predominance over one another controlled the properties of the materials.

Dynamic mechanical analysis by nanoindentation

The blend samples were also subjected to dynamic mechanical analysis by the nanoindentation technique for the analysis of their viscoelastic behavior. The variations of the storage modulus and loss modulus of the samples with increases in the depth of penetration are shown in Figure 12(a,b),



Figure 10 Load-penetration curves of the UPE and bioblends (determined by nanoindentation).

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Figure 11 Modulus and (b) hardness of the UPE and bioblends (determined by nanoindentation)

respectively. The initial high values of the storage and loss moduli could be due to some artifacts of the test method.²⁰ The storage and loss moduli were highest for the UPE, which had no long-chain fatty acid molecules. Because of the absence of long-chain fatty acid molecules, a consistent storage modulus was maintained with the penetration increasing. It was followed by UPE_TO 5. Here also, strong bonding of the oil molecules might have taken place with the UPE resin components, and their complete insertion within the matrix resulted in overall uniformity in the material, which showed a consistent storage modulus with increases in the depth of penetration. However, in UPE_TO 1 and UPE_TO 2, there was instability in the storage modulus values with the depth of penetration rising. Similar fluctuations were observed in the loss modulus values of the bioblend samples, and this could be attributed to the mobility of the long-chain fatty acid molecules.

Similar observations were reported by Miyagawa et al.²¹ for a biobased UPE resin with functionalized

linseed oil. They observed that with an increase in the impact strength, the storage and loss moduli decreased. Abbate et al.²² conducted research on a network of a UPE resin and bismaleimide resins. They observed an increase in the yield stress and Young's modulus of the blend. Similar bonding of UPE to a styrene butadiene rubber latex was reported in a previous study.²³

Flexural strength

The flexural properties of the UPE/styrene/tung oil blends were also investigated and are shown in Figure 13(a–d). The flexural strength increased by 4, 18, and 54% in UPE_TO 1, UPE_TO 2, and UPE_TO 5, respectively, in comparison with the UPE. The flexural modulus increased marginally by 4% in UPE_TO 1. In UPE_TO 2, there was almost no change in the modulus value, and in UPE_TO 5, the modulus decreased by 13%. The breaking energy increased by 28 and 68% in UPE_TO 2 and UPE_TO 5, respectively, although there was no change in



Figure 12 (a) Storage modulus and (b) loss modulus of the UPE and bioblends (determined by nanoindentation).



Figure 13 (a) Flexural strength, (b) flexural modulus, (c) strain, and (d) breaking energy of the UPE and bioblends.

UPE_TO 1. The strain increased by 2, 13, and 37% in UPE_TO 1, UPE_TO 2, and UPE_TO 5, respectively.

These results indicate that strong intercomponent bonding resulted in a higher flexural strength; this was most predominant in UPE_TO 5 because it had the highest weight percentage of tung oil. This is well supported by Figure 7. A marginal increase in the modulus was observed in UPE_TO 1 (4% only). In UPE_TO 2, there was no change in the modulus, and in UPE_TO 5, there was a decrease in the modulus of 13%. A uniform dispersion of tung oil in the UPE/styrene matrix (as observed by SEM) and the optimum proportion of tung oil showed this improvement in the modulus. However, with further increases in the tung oil content, the long-chain fatty acid molecules lowered the modulus value. Although the modulus values obtained by flexural testing were lower than those obtained by the nanoindentation technique, the trend was almost the same. The breaking energy increased by 24 and 68% in UPE_TO 2 and UPE_TO 5, respectively; the oil molecules could take part in viscous dissipation

with time. This observation was well supported by the nanoindentation results: the recovery percentage decreased and the creep percentage increased in UPE_TO 2 and UPE_TO 5 because of increased viscous dissipation.

CONCLUSIONS

Novel materials were developed through the blending of a UPE resin with tung oil in three proportions (1, 2, and 5 wt %). The characterization was performed with impact testing, SEM investigations, and the nanoindentation technique. The dispersion of the tung oil within the matrix, the crosslinking of the tung oil molecules with UPE/styrene blends, and the mobility of long-chain fatty acid molecules predominantly controlled the different properties of the bioblends. The incorporation of 1 wt % tung oil produced a unique combination of properties. This included a 15% increase in the impact strength and 20 and 41.3% increases in the modulus and

Journal of Applied Polymer Science DOI 10.1002/app

hardness, respectively, versus those of the UPE. This demonstrated that the impact strength of the UPE resin could be improved significantly by the incorporation of tung oil in a very small amount (1 wt % in our study) with simultaneous improvements of the modulus and hardness of the blends. The flexural strength, breaking energy, and strain percentage significantly increased with increases in the tung oil content in the blends. This is quite different from rubber-toughened plastics, for which the impact strength can be improved at the cost of mechanical rigidity. These improved multifaceted features of such bioblends can increase their demand in the fiber-reinforced composite industry. However, more work needs to be done to optimize the amount of added tung oil producing the best combination of properties.

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