

Replacement of styrene with acrylated epoxidized soybean oil in an unsaturated polyester resin from propylene glycol, isophthalic acid, and maleic anhydride

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ABSTRACT: Commercial unsaturated polyester (UPE) resins typically contain a high amount of volatile toxic styrene. A non-volatile acrylated epoxidized soybean oil (AESO) was found to be an excellent replacement of styrene in a commercially available UPE resin [designated as Styrene-(PG-IPA-MA)] that is derived from propylene glycol (PG), isophthalic acid (IPA), and maleic anhydride (MA) in terms of the mechanical properties of the resulting kenaf fiber-reinforced composites. The AESO-(PG-IPA-MA) resins had low viscosity and long pot life below 70°C for a typical fiber-reinforced composite application. AESO and PG-IPA-MA were not able to form a strong polymer matrix individually for fiber-reinforced composites. However, a combination of AESO and PG-IPA-MA saw strong synergistic effects between them. The flexural, tensile, and water absorption properties of kenaf fiber-reinforced composites made from AESO-(PG-IPA-MA) resins were comparable with or even superior to those from the Styrene-(PG-IPA-MA) resin. The AESO/(PG-IPA-MA) weight ratio was investigated for maximizing the mechanical properties of the kenaf fiber-reinforced composites. The curing mechanism of the AESO-(PG-IPA-MA) resins is discussed in detail. © 2015 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43052.

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INTRODUCTION

Glass fiber-reinforced thermoset composites are widely used for productions of automobile parts, boat hulls, oil tanks and pipes, bathtubs, and many other products.¹ The production of glass fibers is energy-consuming and the human exposure to fine glass fiber or its dust can cause irritation to eyes, skin, and the respiratory system.² Replacements of glass fibers with renewable natural fibers in fiber-reinforced composites have drawn much attention in recent years because natural fibers are generally inexpensive, abundant, renewable, safe to handle, and nonabrasive to processing equipment. Kenaf fibers have been a popular choice for replacing the glass fibers because kenaf can grow fast and the specific modulus of kenaf fibers is even higher than that of E-glass.³

Unsaturated polyester (UPE) resins are currently the most commonly used thermoset resins for fiber-reinforced thermoset composites.¹ They are less expensive than vinyl ester resins and epoxy resins.¹ One of the commercially available UPE resins from Ashland Inc. (Dublin, Ohio) is a mixture of styrene and PG-IPA-MA plastic that is derived from propylene glycol (PG), isophthalic acid (IPA), and maleic anhydride (MA) [designated as Styrene-(PG-IPA-MA)].

The PG-IPA-MA plastic is solid at room temperature and cannot flow for wetting reinforcing fibers. Styrene is typically used to dissolve the PG-IPA-MA plastic so that the resulting solution has a low viscosity at room temperature. Styrene acts as a solvent (more commonly called a diluent) and a crosslinking agent so that styrene is incorporated into the thermoset polymer matrix after being cured. Styrene is currently the most commonly used reactive diluent because it is inexpensive and can significantly improve stiffness, strengths, water resistance, and thermal stability of the final fiber-reinforced composites.¹ However, styrene is volatile, toxic, and of strong unpleasant odor. It is classified as a hazardous air pollutant (HAP) and a reasonably anticipated human carcinogen by the National Toxicology Program (NTP) in 2011.⁴ It has been reported that unreacted styrene could continue to emit during the lifetime of a fiber-reinforced composite product.^{5,6} The emissive styrene causes air pollutions and poses health hazard for people who manufacture and use styrene, styrene-containing thermoset resins, and fiber-reinforced composites. The emission limits for styrene are regulated by government agencies and have gradually come down in recent years. The regulated emission limits are becoming more and more difficult to comply.¹ Therefore, there is a pressing

need for development of a safer reactive diluent for the replacement of styrene.

Acrylate monomers such as methyl methacrylate and butyl (meth)acrylate and styrene analogs such as α -methylstyrene, vinyl toluene, and 4-*tert*-butylstyrene are also used as reactive diluents for UPE resins in industry.^{1,7} However, they are not an ideal styrene replacement because they are still harmful volatile organic compounds.

Vinyl propionate (VP), vinyl versatate (VV), ethylene glycol dimethacrylate (GDMA), or their mixtures have been investigated as replacements of styrene in UPE resins.⁸ It was found that the GDMA-VP or GDMA-VV mixture was comparable with styrene in terms of the flexural modulus and impact strength of the cured resins.⁸ However, the flexural strength of the styrene-free resins was significantly lower than that of the styrenated counterparts (i.e., 20%–50% lower).⁸ Resin viscosity and volatility of VP, VV, and GDMA were not reported.

Vinyl levulinate from bio-based levulinic acid has been investigated as a styrene replacement in UPE resins.⁹ The impact strength of the styrene-free UPE resins was comparable with that of the styrenated counterparts; but the flexural strength and flexural modulus of the styrene-free resins were 35% lower than those of the styrenated resins.⁹ Viscosity and volatility of the styrene-free resins were also not reported.

Vegetable oils have low viscosity at room temperature and are not toxic or volatile. They are inexpensive, abundant, and renewable. Methacrylates from reactions of glycidyl methacrylate and various fatty acids were found to be not volatile.^{10,11} It has been demonstrated that the partial replacement of styrene in vinyl ester resins with the methacrylates did not compromise the resin viscosity and mechanical properties of the cured resins and composites.^{10,11} However, the complete replacement of styrene resulted in inferior mechanical properties and resin viscosity.^{10,11} It is not known whether the methacrylates can be used for the replacement of styrene in UPE resins.

Acrylated epoxidized soybean oil (AESO) is a reaction product of epoxidized soybean oil and acrylic acid. It is not volatile or hazardous. AESO has been extensively investigated for the preparation of bio-based coatings,¹² adhesives,¹³ coupling agents,¹⁴ resins,^{15,16} and foams.¹⁷ In this study we found that AESO was able to replace styrene in the commercially available PG-IPA-MA-based UPE resins for production of superior kenaf fiber-reinforced composites.

EXPERIMENTAL

Materials

AESO (containing 4000 ppm monomethyl ether hydroquinone) was purchased from Sigma-Aldrich (St. Louis, MO). *tert*-Butyl peroxybenzoate (TBPB) was obtained from Akzo Nobel (Chicago, IL). Kenaf fibers were purchased from Kenaf Industries Ltd (Raymondville, TX). The PG-IPA-MA plastic and the Styrene-(PG-IPA-MA) resin (a mixture of the PG-IPA-MA plastic and styrene) were obtained from Ashland Inc. (Dublin, OH). The PG-IPA-MA plastic was slowly ground into powder before use.

Preparation of Kenaf Fiber Mats

Kenaf fiber mats were prepared in accordance with a previously described method.^{18–20} Specifically, kenaf fiber bundles (130 g, 1 inch in length) were fed into a LOUET drum carder for separating the large fiber bundles into small bundles or individual fibers. Through the carding, layering, and needle-punching process, a large piece of fine, unidirectionally oriented fiber mat was formed. The large mat was cut into five small square mats (200 mm \times 200 mm) by a paper cutter. The fiber mats were dried in an air circulating oven at 103°C for at least 20 h before use. The total weight of five oven-dried fiber mats was 78 g.

Preparation of AESO-(PG-IPA-MA) Resins

AESO (71.6 g) was heated to 90°C in a 250-mL beaker equipped with a mechanical stirrer and an oil bath. The PG-IPA-MA plastic (47.7 g) was slowly added into AESO over 5 min. The mixture was stirred for 10 min to generate a homogenous and pourable resin. The resin was purged with nitrogen for 3 min and cooled to 70°C. Subsequently, TBPB initiator (1.83 g, 1.5 wt % based on the total weight of AESO and the PG-IPA-MA plastic) was added into the resin and then stirred for 3 min. The resulting AESO-(PG-IPA-MA) resin had an AESO/(PG-IPA-MA) weight ratio of 60/40 and was designated as AESO60. AESO50 and AESO70 had the AESO/(PG-IPA-MA) weight ratio of 50/50 and 70/30, respectively, and were prepared with the same procedure as described for AESO60. Styrene-(PG-IPA-MA) was mixed with TBPB (1.5 wt % based on Styrene-(PG-IPA-MA) and used as a control. A mixture of AESO and TBPB (1.5 wt % based on AESO) was also used as a control.

Preparation of Kenaf Fiber-Reinforced AESO-(PG-IPA-MA) Composites

AESO60 (7.8 g) was slowly poured onto the upper surface of a kenaf fiber mat that was placed in the chamber of a stainless steel mold with a dimension of 200 mm \times 200 mm \times 3 mm. The mat was flipped and subsequently coated with the same amount of resin on the opposite surface. Afterward, a second fiber mat was stacked above the first mat with the fiber direction parallel to each other, and subsequently coated with AESO60 on both surfaces with the same procedures as described previously. The same procedure was repeated until five fiber mats were saturated with the resin. The beaker containing the AESO60 resin was stored in a hot water bath at 70°C during the application of the resin onto the fiber mats for maintaining the low viscosity of the resin. A stack of the five resin-saturated fiber mats in the mold was covered by a stainless steel plate on top and was pressed at 3.5 MPa with an automatic Benchtop Carver press (Carver Inc., Wabash, IN) for 10 min at room temperature, and subsequently pressed at 4.5 MPa for 40 min with the temperature being raised to 160°C at about 10°C/min. The mold was then placed between two plywood panels, pressed under a pressure of 4.5 MPa, and allowed to slowly cool down for 100 min. The resulting kenaf fiber-reinforced AESO-UPE panel was removed from the mold, designated as kenaf-AESO60, and stored at room temperature overnight before being evaluated for its mechanical properties and water resistance. Two panels were made for each resin formulation. Kenaf-AESO50 and Kenaf-AESO70 composite panels were prepared with the same procedure as described previously. Kenaf-Styrene-

(PG-IPA-MA) composite panels were prepared with the same procedure as described previously, except that the Styrene-(PG-IPA-MA) resin was kept at 40°C during the resin application. Kenaf-AESO composite panels were prepared with the same procedure as described previously, except that the AESO resin was kept at 55°C during the resin application.

Determination of Flexural Properties of the Composites

The test specimens with the dimension of 65 mm × 12.7 mm × 3 mm and with the fibers aligning along the lengthwise direction of the specimen were evaluated for the flexural strength and flexural modulus through a three-point bending test that was performed according to ASTM D790, with a span of 50 mm and a rate of crosshead motion of 1.28 mm/min. At least six specimens were tested and averaged values were reported.

Determination of Tensile Properties of the Composites

The tensile strength and tensile modulus were obtained from a tensile test in accordance with ASTM D3039. For the preparation of dumbbell shape specimens for the tensile test, the composite panels were first cut into rectangular specimens with the dimension of 58 mm × 14.5 mm × 3 mm and the fibers aligning along the lengthwise direction of the specimen. The rectangular specimens were further cut into dumbbell shape specimens that had a gripping length of 11 mm on each end, a width of the narrow section of 8.5 mm, and a length of the narrow section of 30 mm. The distance between grips was 36 mm, the rate of crosshead motion was 0.5 mm/min, and the time to failure was about 2 min. At least six specimens were tested and averaged values were reported.

Statistical Analysis

Data from the flexural and the tensile tests were statistically analyzed by one-way ANOVA with SAS software (SAS Institute, Cary, North Carolina). All comparisons were based on a 95% confidence interval.

Measurements of Water Absorption of the Composites

The water absorption tests were performed according to ASTM D570. Test specimens had the dimension of 76.2 mm × 25.4 mm × 3 mm. Prior to the tests, the specimens were dried in an oven for 24 h at 50°C, cooled in a desiccator, and weighed. Subsequently, the specimens were immersed in distilled water. At predetermined intervals, the specimens were removed from water, wiped with dry tissue papers, weighed, and replaced in the water. The water absorption was determined by the ratio of the weight gain to the dry weight of the specimen. Three specimens were tested and averaged values were reported.

Differential Scanning Calorimetry (DSC) Analysis

DSC experiments were performed on a TA Q2000 analyzer (TA Instruments, Inc., New Castle, DE). The DSC was calibrated in three steps: the first with nothing in the chamber to get a baseline correction, the second with sapphire to calibrate the heat capacity, and the third with indium for temperature calibration. The specimen chamber was purged with nitrogen during the experiments with a flow rate of 75 mL/min. For temperature sweep, test specimen in standard aluminum pan with lid was heated from room temperature to 210°C at a rate of 10°C/min. An empty aluminum pan with a lid was used as a reference.

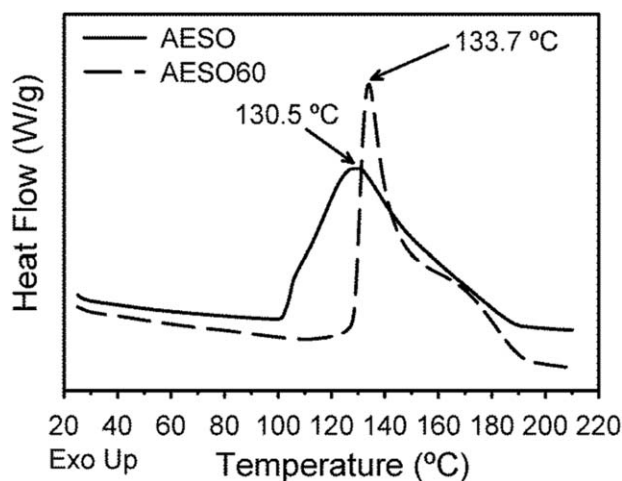


Figure 1. DSC curves for the course of curing of AESO and AESO60 (ramp rate: 10°C/min).

Measurements of Resin Viscosity

The resin viscosity was measured with an AR 2000ex rheometer (TA Instruments, Inc., New Castle, DE) with parallel plate geometry (25 mm in diameter). The gap between two plates was 400 μm. Test specimens (0.20 g) were steady-sheared at a shear rate of 5 Hz and a ramp rate of 5°C/min. Sampling delay time was 10 s.

Measurements of a Resin Pot Life

For the measurement of a resin pot life at use temperatures, an oscillatory time sweep was performed with the same AR 2000ex rheometer as described previously. Sampling delay time was set at 10 s. At t_0 , test specimens were started to be oscillatory-sheared at 5 Hz with a strain amplitude of 50% at a set temperature of 65°C, 70°C, 75°C, and 80°C, respectively. As the curing began, the storage modulus (G') increased rapidly. At t_1 , G' was equal to the loss modulus (G''). The time between t_0 and t_1 was defined as the pot life for the resin in this study. Three specimens were tested and averaged values were reported.

RESULTS AND DISCUSSION

Determination of Curing Conditions

As shown in Figure 1, the curing of AESO started at around 102°C and ended at 190°C, with an exothermal peak at 130.5°C (i.e., the temperature with the highest curing rate). The curing of AESO60 started at around 110°C and ended at 190°C with an exothermal peak at 133.7°C. The shifts in the onset curing temperature from 102°C to 110°C and in the exothermal peak from 130.5°C to 133.7°C indicated that copolymerization between AESO and the PG-IPA-MA plastic occurred during the curing of AESO60.

The hot-press condition of 160°C for 40 min was expected to be sufficient for the complete cure of the resin in the preparation of kenaf-AESO60 composites. DSC analysis indeed showed that there were no residual exothermal peaks for the kenaf-AESO60 composites (DSC graph not shown). The resulting kenaf-AESO60 composites had hard and smooth surfaces and had no visually detectable defects such as warpage, voids, blisters, cracks, and delamination, which also indicates the good curing of the resins.

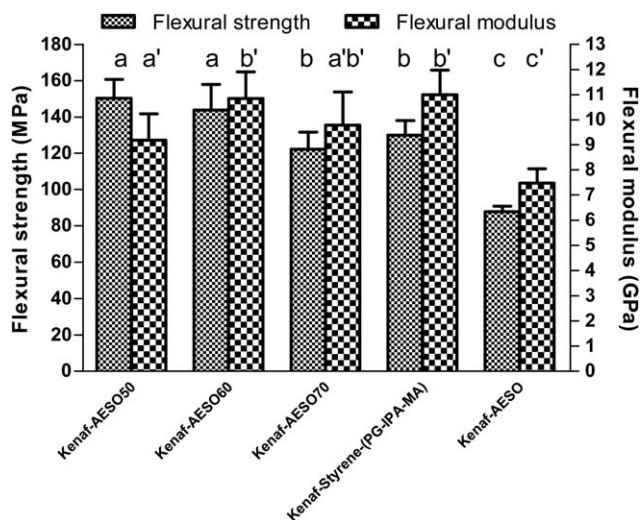


Figure 2. Effects of the AESO content on the flexural properties of the composites. (The means between two groups significantly differ if the letters on top of error bars are different.)

Effects of the AESO Content on the Mechanical Properties and Water Resistance of the Composites

As shown in Figure 2, increasing the AESO content in the AESO-(PG-IPA-MA) resin from 50 to 60 wt % did not significantly change the flexural strength, but significantly increased the flexural modulus of the composites. Further increasing the AESO content from 60 to 70 wt % decreased the flexural strength significantly, but did not significantly decrease the flexural modulus. Composites prepared from AESO60 had better flexural properties than those from AESO50 and AESO70. Kenaf-AESO60 composites had a significantly higher flexural strength than and a comparable flexural modulus to kenaf-Styrene-(PG-IPA-MA) composites.

As shown in Figure 3, increasing the AESO content from 50 to 60 wt % did not significantly change the tensile strength, but

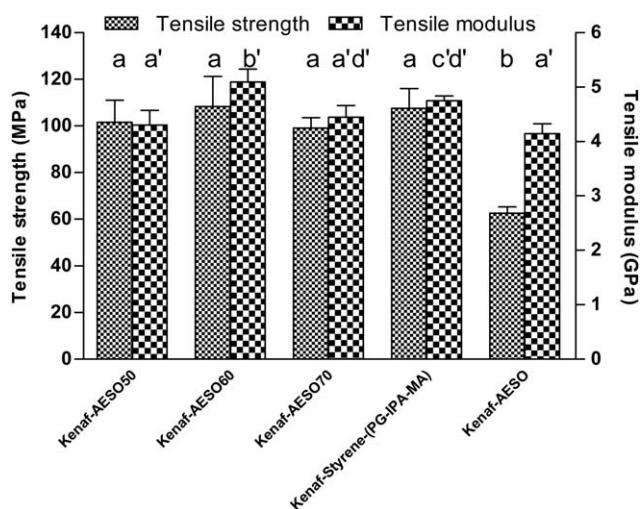


Figure 3. Effects of the AESO content on the tensile properties of the composites. (The means between two groups significantly differ if the letters on top of error bars are different.)

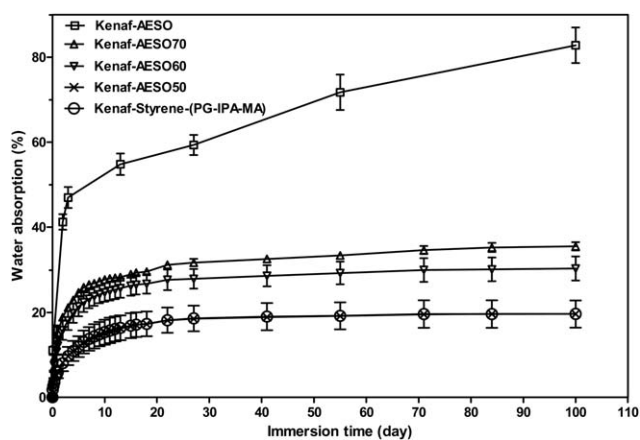
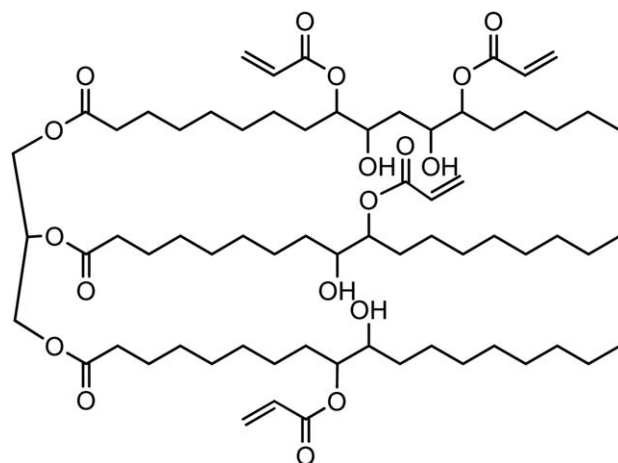


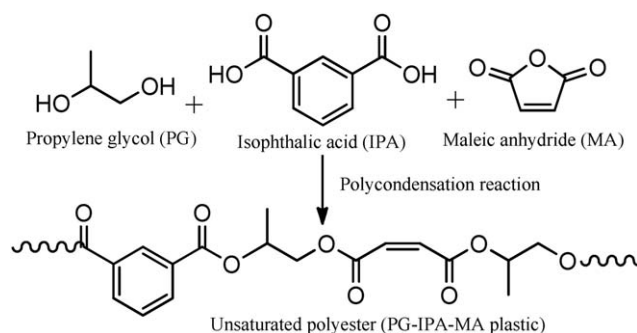
Figure 4. Effects of the AESO content on the water absorption of the composites.

significantly increased the tensile modulus of the composites. Further increasing the AESO content did not significantly change the tensile strength, but significantly decreased the tensile modulus. Kenaf-AESO60 had better tensile properties than kenaf-AESO50 and kenaf-AESO70. Compared with the kenaf-Styrene-(PG-IPA-MA) composites, the kenaf-AESO60 composites had a comparable tensile strength and a significantly higher tensile modulus. These results demonstrated that the optimum AESO content was about 60% and the AESO60 resin was superior to the Styrene-(PG-IPA-MA) resin in terms of the flexural and the tensile properties of the kenaf-reinforced composites.

As shown in Figure 4, the water absorption for the kenaf-AESO composites was much higher than any other composites at any given measurement time. For all other composites, the water absorption increased with increasing immersion time and then leveled out. Kenaf-AESO50 had approximately the same water absorption behavior as kenaf-Styrene-(PG-IPA-MA). As the AESO content increased from 50 to 70 wt %, the water absorption of the resulting composites increased. These results showed that AESO50 was comparable with Styrene-(PG-IPA-MA) and better than AESO60 and AESO70 in terms of the water absorption of the kenaf-reinforced composites.



Scheme 1. Representative structure of AESO.



Scheme 2. Representative structure of the PG-IPA-MA UPE plastic.

The representative structures of the AESO and the PG-IPA-MA plastic are shown in Schemes 1 and 2, respectively. AESO (~1260 g/mol) has a higher molecular weight than styrene (104 g/mol). The C=C bonds in AESO have greater steric hindrance than those of styrene and cannot move as freely as styrene for polymerization reactions. We speculated that the C=C bonds in AESO cannot efficiently polymerize to form strong polymeric matrixes, which is consistent with the fact that kenaf-AESO composites have significantly lower strength (Figures 2 and 3) and higher water absorption (Figure 4) than the kenaf-Styrene-(PG-IPA-MA) composites. The C=C bonds in the PG-IPA-MA plastic are distributed along the polymer chains and cannot freely move around without carrying the polymer chains. There is considerable steric hindrance surrounding the C=C bonds when two C=C bonds try to collide with each other for reactions because both ends of the C=C bonds are linked to the polymer chains. The inability of the C=C bonds to freely move around and the steric hindrance make the reactions among the C=C bonds in the UPE plastics very inefficient. As a result, the crosslinking/polymerization of the PG-IPA-MA plastic alone typically leads to weak and brittle polymeric matrixes containing substantial amounts of unreacted C=C bonds. It was unexpected that the mixture of AESO and the PG-IPA-MA plastic had strong synergistic effects between them and led to the generation of kenaf fiber-reinforced composites that had higher flexural and tensile

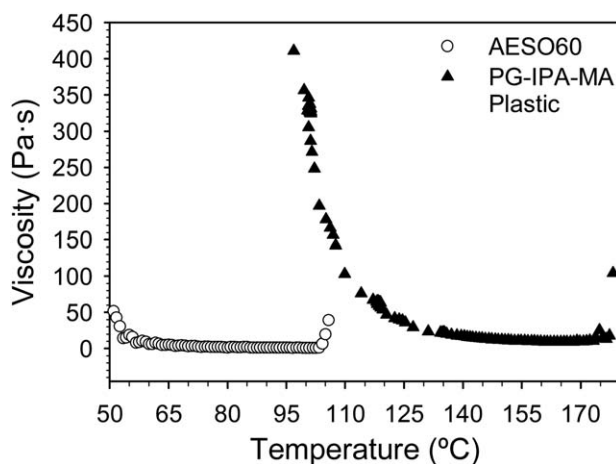


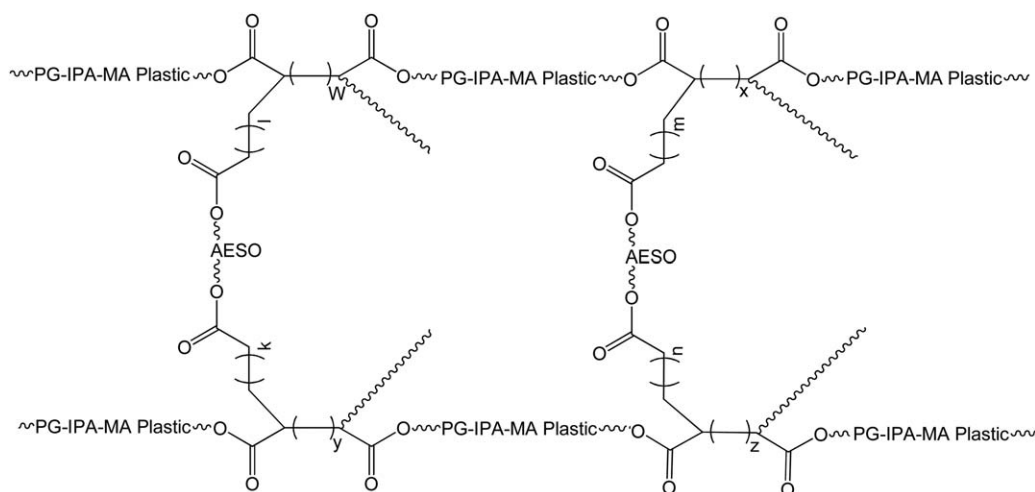
Figure 5. Effects of temperature on the resin viscosity (AESO60 contained 1.5 wt % of TBPB. The PG-IPA-MA UPE plastic did not contain TBPB).

properties than the kenaf-Styrene-(PG-IPA-MA) composites (Figures 2–4).

The proposed curing/crosslinking reactions between AESO and the PG-IPA-MA plastic are shown in Scheme 3. AESO has multiple C=C bonds that can homopolymerize by themselves or copolymerize with the C=C bonds in the PG-IPA-MA plastic. The C=C bonds on the PG-IPA-MA polymer chains may also react with each other. These reactions together generated rigid, crosslinked networks.

Resin Viscosity

A viscosity of 10 Pa s for a UPE resin was considered good for a hand lay-up resin application. The PG-IPA-MA plastic had to be heated to 160°C for reaching the viscosity of 10 Pa s, whereas AESO60 only needed to be heated to 56°C for reaching the same viscosity (Figure 5). The viscosity of AESO60 rapidly decreased from 10 to 3 Pa s when the temperature was raised from 56°C to 70°C. The viscosity continued to decrease as the temperature increased and was about 1 Pa s at 80°C. The viscosity began to increase rapidly when the temperature was raised to 105°C, which was in good agreement with the DSC results that the resin began to cure at 110°C (Figure 1).



Scheme 3. Proposed crosslinking reaction between AESO and the PG-IPA-MA UPE plastic.

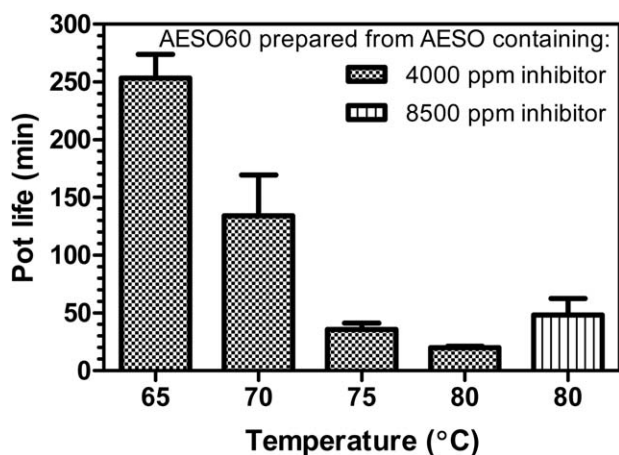


Figure 6. Effects of temperature on the pot life of AESO60.

Resin Pot Life

Compared with the commercial Styrene-(PG-IPA-MA) that has a low viscosity at room temperature, it is a drawback that AESO60 has to be heated and maintained at 56°C–80°C so that it has a low viscosity for its easy application. As shown in Figure 6, the pot life decreased along with the increase in temperature, which is consistent with the fact that TBPB decomposes faster at a higher temperature. At 70°C, AESO60 had a long pot life of 134 min and a low viscosity of 3 Pa s, which are very convenient conditions for a hand lay-up resin application process. For some composite fabrication processes that require an even lower viscosity, the temperature can be raised to about 80°C at which the viscosity of the AESO60 resin is about 1 Pa s. However, at this temperature, the resin has to be applied to fibers within 20 min.

Increase in the concentration of the inhibitor, monomethyl ether hydroquinone (MEHQ), is an effective way to increase the pot life of the resin. As shown in Figure 6, the pot life of AESO60 at 80°C increased from 20 to 50 min when the concentration of MEHQ was raised from 4000 to 8500 ppm based on AESO.

CONCLUSIONS

Styrene-free UPE resins with AESO as a styrene replacement and their kenaf fiber reinforced composites were successfully prepared and evaluated. DSC analysis showed that the composites were completely cured when the composites were pressed at 160°C for 40 min. Kenaf-AESO60 had higher flexural and tensile strengths and moduli than kenaf-AESO50 or kenaf-AESO70. Kenaf-AESO60 had a higher flexural strength and a higher tensile modulus than kenaf-Styrene-(PG-IPA-MA). Kenaf-AESO60 had comparable flexural modulus and comparable tensile strength to kenaf-Styrene-(PG-IPA-MA). The water absorption of kenaf-AESO50 was lower than that of kenaf-AESO60 or kenaf-AESO70, and was comparable with that of kenaf-Styrene-(PG-IPA-MA). Kenaf-AESO50 had a higher flexural strength than kenaf-Styrene-(PG-IPA-MA) and had a comparable tensile strength to kenaf-Styrene-(PG-IPA-MA). AESO60 had low viscosity of 3 Pa s and a sufficiently long pot life of 134 min at 70°C for a hand lay-up process. Increase in temperature reduced the resin viscosity, but shorten the pot life

of the resin. Increase in the concentration of the inhibitor, MEHQ, effectively increased the pot life of the resin.

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