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What is This?
Ultraviolet-induced free radical reactions in ENR50/alkyd blend leading to significant increase in cross-linking

Yoke Kum Khong, Desmond Teck Chye Ang and Seng Neon Gan

Abstract
In this work, the potential of palm stearin alkyd as a cross-linking agent for rubber compounding was investigated. Alkyd carrying pendent –COOH groups was blended with epoxidised natural rubber (ENR50) via solvent casting technique at ambient temperature. Spectroscopic characterisations show that ENR50 and alkyd have interacted with each other via chemical reaction involving the epoxide of the rubber and –COOH of alkyd. Consequently, notable increase in the glass transition temperature ($T_g$) and gel content of the blend was observed. In addition to the epoxide ring-opening reaction, the extent of cross-linking in the blend was further increased by means of ultraviolet (UV) curing. As a result, the UV-cured blends experienced lower percentage of swelling, produced higher percentage of gel, have higher cross-link density and higher $T_g$. The cross-link density in the rubber/alkyd blend has increased from $3.0 \times 10^{-6}$ mol cm$^{-3}$ to $2.0 \times 10^{-4}$ mol cm$^{-3}$ after UV curing. This is clearly evidenced from the swelling test results, where ENR and alkyd are completely soluble in the solvent, but upon blending and subsequent UV irradiation, the blend produced >90% of gel and swelled by 318%. Findings from this work show that sustainable material such as vegetable oil could be utilised in development of cross-linking agent for rubber. Significant increase in the cross-link density of the UV-cured blend suggests that environment friendly compound such as alkyd has great potential to serve as an alternative to conventional compounds in rubber vulcanisation.

Keywords
Epoxidised natural rubber, cross-linking, alkyd, UV curing, polymer blend

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Introduction

Polymer blending has become a very important contributor in the development of new polymer materials. It is one of the major areas of research and development in the field of polymer science and has captured interests of many researchers and polymer chemists in the past three decades.\(^1\) One of the factors that have led to rapid development in polymer blending is the opportunity to develop materials with new or improved properties at reduced material cost. Besides, higher rate of production of polymeric materials can be achieved via polymer blending to meet up with the market demands as the production of the materials do not need to undergo the polymerisation step.\(^2\)

Generally, polymer blends can be categorised as miscible, partially miscible and immiscible blend depending on the ability of the components in the blend to interact with each other. A blend is considered immiscible if it exhibits two or multiple phases. Miscible blend on the other hand is composed of only one phase and it is of considerable scientific and practical interests.\(^3\) A factor that determines miscibility in a blend is the interactions between the constituents. Significant interactions between the blend components such as hydrogen bonding, dipole–dipole interactions, ion–ion interactions and ion–dipole interactions could contribute to the miscibility of the blend.\(^4,5\)

Presence of chemical reactions between the reactive functional groups in the individual polymers in the blend could render them miscible as well.\(^6\)

In the current work, epoxidised natural rubber 50 (ENR50) was blended with alkyd via solvent casting technique. ENR is a semi-synthetic rubber obtained from epoxidation of natural rubber latex. It is known to have good abrasion resistance, high wet grip and good damping properties, but the modulus, hardness, tensile strength and elongation at break of the epoxidised rubber are relatively low and comparable with those of natural rubber (NR).\(^7\) Rubbers are commonly vulcanised using cross-linking agent such as sulphur to increase their mechanical strength and at the same time, making it more durable. In vulcanised rubber, there are several types of cross-links that could hold the rubber chains together, and amongst the common ones include the sulphide linkage, C–C bond formed by polyvalent organic radical and interactions involving polyvalent metal ion.\(^8\) Generally, rubbers such as ENR or NR are often cured via cross-linking reaction involving their C=C. In the case of ENR, the epoxide group provides an additional site for cross-linking with other functional chemicals. The highly strained three-membered ring of epoxide group makes it susceptible to nucleophilic addition reaction.

Alkyd that was used to blend with ENR in the current work was synthesised from palm stearin. Alkyd is a polyester produced from polycondensation of polyhydric alcohol, polyacid and fatty acid. A distinct feature of alkyd that differentiates it from the common polyester is the presence of fatty acids of vegetable oils as a major part of its composition.\(^9\) Palm stearin was chosen as the source of fatty acids in the production of alkyd due to economic reason. It is the solid fraction of palm oil, and it can be viewed as a by-product from the production of palm olein. Hence, it is often traded at discounted price.\(^10\) The selected alkyd was formulated to carry pendent –COOH groups along its main chain. Upon blending the alkyd and ENR, the two could interact...
via reaction involving the –COOH of alkyd with the epoxide ring of ENR. In this blend, the alkyd serves to cross-link the rubber chains.

In order to increase the extent of cross-linking in the ENR50-alkyd blend, benzophenone and trimethylolpropane triacrylate (TMPTA) were introduced into the system as ultraviolet (UV) photoinitiator and cross-linker, respectively, and the blends were then cured using UV light. Upon UV irradiation, benzophenone generates radicals that are responsible to initiate, and subsequently propagate cross-linking in the system. Cross-linking during UV irradiation could take place at the unsaturation sites, such as C=C from the isoprene units of ENR50, –OOC–CH=CH–COOH from the grafted maleic anhydride (MA) along the alkyd chain, and C=C from TMPTA. Earlier works on development of UV-curable coatings from palm stearin alkyd revealed that TMPTA, a trifunctional acrylate could serve as excellent cross-linker to increase the extent of cross-linking in a system. The novelty of this work lies in the usage of sustainable material to produce alkyd that could serve as a cross-linking agent in rubber blend. In addition to that increasing the extent of cross-linking via UV irradiation in ENR50/Alkyd blend has not been reported before. In this work, both functional groups of ENR, epoxide and –C=C– groups, have been utilised to increase the cross-link density in the rubber blend.

**Experimental**

**Materials**

Refined, bleached and deodorised palm stearin and glycerol 99.5% were kind gifts from Emery Oleochemicals Malaysia Sdn Bhd (Malaysia), and they were used as received. ENR50 was supplied by Rubber Research Institute of Malaysia (RRIM). The number 50 indicates mole percentage of the isoprene units of NR that have been converted to epoxide groups. TMPTA was obtained from Sigma Aldrich (St Louis, Missouri, USA), and it was treated with 1.5% w/v of sodium hydroxide aqueous solution and subsequently dried over activated molecular sieves prior to usage. All other chemicals were used without further treatment. Phthalic anhydride (PA) was purchased from DC Chemical Korea Corporate Limited (Seoul, South Korea), MA and benzophenone (99%) were obtained from Sigma Aldrich (Steinheim, Germany), and calcium hydroxide (Ca(OH)₂) were purchased from HmbG Chemicals (Hamburg, Germany).

**Alkyd synthesis**

Alkyd synthesis started with alcoholysis process using 185 g of palm stearin, 75 g of glycerol, and 0.15 g of Ca(OH)₂. The reaction was carried out at 230°C under constant stirring at 220 r min⁻¹ for approximately 2 h. The completion of the process was confirmed by performing solubility test on the reaction mixture in ethanol. The alcoholysis was deemed complete for most practical purposes when the reaction mixture gave a clear solution in ethanol. The reaction mixture was then allowed to cool down to below 120°C before adding 114 g of PA to start the polyestierification process. The temperature of the mixture was gradually raised back and maintained at 230°C during the polyestierification.
The progress of the reaction was monitored by measuring the acid value of the reaction mixture at every 30 min intervals from the beginning of the esterification. In the acid value determination, small amount of the reaction mixture was dissolved in neutral solvent (volume isopropanol:toluene = 1:1), followed by titration using standardised potassium hydroxide (KOH) solution, with phenolphthalein as indicator. Once the acid value of the reaction mixture has reached below 20 mg KOH g⁻¹ resin, the temperature was reduced to 120°C. The final step in the synthesis was to incorporate 19 g of MA into the existing alkyd chains in the reaction mixture. MA was added into the system whilst maintaining the temperature at 120°C and stirred at 220 r min⁻¹. The extent of the incorporation was monitored by measuring the acid value of the reaction mixture periodically. The reaction was stopped when the acid value of the mixture has dropped to approximately half from the value after all MA has been loaded into the system.

Characterisation of alkyd

FTIR analysis was conducted using FTIR spectrometer (Perkin Elmer Spectrum 400). The alkyd was coated on a potassium bromide cell and scanned from 4000–400 cm⁻¹ at a resolution 4 cm⁻¹. Hydroxyl number of the alkyd was determined according to the standard procedure adopted from ASTM D4274-94-B standard. PA reagent (composition 0.16 g PA mL⁻¹ pyridine) was used to dissolve the alkyd, and the solution was then heated at 98–100°C for 2 h in water bath. The alkyd solution was allowed to cool to room temperature before titrating with standardised KOH solution, using phenolphthalein as indicator.

Preparation of ENR50-alkyd blend

To prepare the blend, 5.0 g of ENR50 was blended with 0.5 g of alkyd for 12 h at ambient temperature via solvent casting technique, using toluene as the mutual solvent. The composition of the blend is equivalent to 10 parts of alkyd for every 100 parts of ENR50, and the ratio of epoxide of ENR50 to –COOH of alkyd is 83.9. After blending for 12 h, benzophenone and TMPTA were added into the mixture and stirred for another 1 h to form homogenous solution. Several blends with

<table>
<thead>
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<th>Blends</th>
<th>ENR50 (g)</th>
<th>Alkyd (g)</th>
<th>Benzophenone (g)</th>
<th>TMPTA (g)</th>
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</thead>
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<tr>
<td>EA-B0T0</td>
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<td>0.5</td>
<td>0</td>
<td>0</td>
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<tr>
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<td>0.28</td>
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<tr>
<td>EA-B5T3</td>
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<td>0.5</td>
<td>0.28</td>
<td>0.17</td>
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<tr>
<td>EA-B5T5</td>
<td>5.0</td>
<td>0.5</td>
<td>0.28</td>
<td>0.28</td>
</tr>
</tbody>
</table>

ENR50: epoxidised natural rubber 50; TMPTA: trimethylolpropane triacrylate.
different proportions of benzophenone and TMPTA were prepared, and the compositions of all the blends are shown in Table 1. Each of the blend solutions was then cast as thin film on glass panel and left to dry in fume hood at room temperature for 24 h, followed by drying in vacuum oven at 40°C for another 24 h. Subsequently, the dried film, which was still tacky were irradiated with UV light for (i) 60 s, (ii) 120 s, (iii) 180 s and (iv) 240 s. The source of UV light from this work was from a 400 W metal halide lamp that radiates UV light (λ = 365 nm) at intensity of 225 mW cm$^{-2}$. All the films were observed to have cured to non-sticky state after the UV irradiation.

**Thermal analysis**

The UV-cured blends were conditioned at room temperature for 24 h prior to analysis. The glass transition temperature ($T_g$) of the blends were measured using Mettler Toledo 822e, whilst thermogravimetric analysis (TGA) thermograms were obtained using Perkin Elmer thermogravimetric analyser 4000 (TGA4000). Differential scanning calorimetric (DSC) scans were conducted from −60 to 60°C at 20°C min$^{-1}$ under nitrogen (N$_2$) atmosphere, and $T_g$ of the blend was obtained from the heating cycle. TGA scans were conducted from 50 to 900°C at heating rate 20°C min$^{-1}$ under N$_2$ atmosphere.

**Swelling test and cross-link density measurement**

A known weight of UV-cured blend, $w_0$ was immersed in toluene for 48 h at room temperature in a covered glass bottle. After the soaking period, the swollen gel was removed and sandwiched between two pieces of filter papers, and the total weight of the gel and filter papers was recorded as $w_1$. Immediately after that, the swollen gel was removed from the filter papers and the weight of the wet filter papers was measured as $w_2$. The purpose of having the filter papers in the procedure was to remove the excess solvent on the surface of the samples. The swollen gel was left to dry in fume hood at room temperature for 24 h before further dried in air-circulating oven at 100°C until a constant dried gel weight, $w_3$, was obtained. The percentage of swelling of the blends was obtained using equation (1). Using the same set of data, the gel content of the blends was calculated using equation (2).

\[
\text{% Swelling} = \frac{(w_1 - w_2) - w_3}{w_3} \times 100, \quad (1)
\]

\[
\text{Gel content(\%)} = \frac{w_3}{w_0} \times 100 \quad (2)
\]

Flory–Rehner theory$^{12}$ was employed to calculate the cross-link density, $\nu$ of the blend, defined in equation (3):
\[ \nu = \frac{-\log_e(1 - \phi) + \phi + \chi \phi^2}{V_1 \left( \phi^{1/3} - \frac{\phi}{2} \right)} \]  

(3)

where \( \phi \) is the volume fraction of polymer in the swollen gel, \( \chi \) is the solvent–polymer interaction parameter, and \( V_1 \) is the molar volume of toluene. \( \phi \) was calculated using equation (4), where \( \rho \) and \( \rho_1 \) represent density of the blend and toluene, respectively.

\[ \phi = \frac{1}{1 + \left[ (w_1 - w_2)\rho \right]^{1/3} - \left( \frac{\rho}{\rho_1} \right)} \]  

(4)

In determination of cross-link density, \( \chi \) was obtained using Hildebrand solubility parameter theory, expressed in equation (5). The solubility parameters of toluene and the blend (\( \delta_1 \) and \( \delta_2 \) respectively) were obtained using turbidimetric titration method, as described by Ng and Chee.\(^{13}\) The \( \delta \) of ENR and alkyd were obtained via turbidimetric titration at 30°C using toluene as the solvent, and heptane and acetonitrile as the two non-solvents.

\[ \chi = 0.34 + \left( \frac{\delta_1 - \delta_2}{2} \right)^2 \frac{V_1}{RT} \]  

(5)

where \( R \) is the universal gas constant and \( T \) is the absolute temperature.

**Results and discussions**

**Alkyd synthesis**

A plausible alkyd synthesis route is shown in Figure 1. The synthesis began with alcoholysis process, where mixture of palm stearin and glycerol was converted into predominant mixture of monoglycerides. Subsequently, polyesterification took place between the mixture of glycerides and PA to form alkyd chains. MA was then introduced at carefully controlled reaction temperature to allow it to be grafted on the alkyd chains as pendent \(-\text{OOC–CH} = \text{CH–COOH}\). During the introduction of MA, the temperature of the mixture was maintained around 120°C to avoid the \(-\text{COOH}\) of the grafted MA from participating in undesired esterification. Besides temperature control, stopping the reaction when the acid value has dropped to approximately half of the value after all MA has been loaded into the system (from 74.9 mg KOH g\(^{-1}\) to 44.0 mg KOH g\(^{-1}\)) could prevent the newly generated \(-\text{COOH}\) from undergoing esterification as well. It is important to preserve the \(-\text{COOH}\) as it is the reactive group that ring-open the epoxide group of ENR50.

Experimental observations such as reduction in the acid value and hydroxyl value of the mixture without formation of water agrees with the proposed reaction where the anhydride ring of MA has opened to form \(-\text{OOC–CH} = \text{CH–COOH}\). The hydroxyl value has dropped from 156 mg KOH g\(^{-1}\) to 118 mg KOH g\(^{-1}\) after MA was introduced into the mixture due to the reaction between the \(-\text{OH}\) groups of alkyd with MA to form half...
Figure 1. Plausible synthesis route of palm stearin alkyd.
ester. Similar approach was reported by Ataei et al. when they used PA that ring-opened to form half ester.14

### Blending of ENR50 and alkyd

FTIR spectra of alkyd, ENR50 and EA-B0T0 blend are shown in Figure 2. The FTIR spectrum of alkyd agrees with the structure proposed in Figure 1. Some of the peaks present include peaks at 2854–2925 cm⁻¹ that corresponds to methyl and methylene groups, peak at 1732 cm⁻¹ from C=O stretching of ester, peak at 1599 cm⁻¹ from the aromatic rings and also peak at 1657 cm⁻¹ from the CH=CH of the grafted MA. After alkyd and ENR50 were blended for 12 h, the amount of epoxide groups in the blend has decreased, which is shown from the reduction in the epoxide peak absorbance at 870 cm⁻¹ and normalised to an internal standard peak at 835 cm⁻¹ (A₈₇₀/A₈₃₅). Note that the value A₈₇₀/A₈₃₅ only indicates the relative amount of epoxide groups and not the absolute quantity. The peak absorbance A₈₇₀/A₈₃₅ has decreased from 1.9 to 1.5, indicating some of the epoxide group was consumed during the blending. The –COOH group from alkyd has ring-opened the epoxide group of ENR50 to form ester linkage, as proposed in Figure 3.

Figure 2. FTIR spectra of (a) alkyd, (b) ENR50, and (c) EA-B0T0 blend. FTIR: Fourier transform infrared; ENR50: epoxidised natural rubber 50.
Reactions involving –COOH group with epoxide group have been reported by many earlier researchers.\textsuperscript{15–17} Other evidence that shows alkyd and ENR50 has interacted with each other is from the gel content of the blend. Individually, alkyd and ENR50 are easily soluble in toluene, with the former recorded 100\% solubility and the latter 98\% solubility. However, after the two were blended together for 12 h, the solubility of the blend in toluene is much lower compared with the individual components, indicated by the formation of 55\% of gel. A recent article has reported the cross-linking reactions between –COOH groups of alkyd and epoxide groups of ENR could occur readily in toluene solution even at ambient temperatures. The cross-link density is proportional to the amount of –COOH groups in the alkyd. As the –COOH content is increased, the blend produces higher percentage of gel insoluble in toluene.\textsuperscript{18}

**UV irradiation on ENR50-alkyd blend**

FTIR analyses were carried out on EA-B5T5 blend after it was irradiated with UV light, and the spectra of the blend before and after the UV irradiation are shown in Figure 4. The peak absorbance at 1657 cm\(^{-1}\) normalised to internal standard peak at 1380 cm\(^{-1}\) has decreased with UV irradiation time, as shown in Figure 5. The purpose of normalising the peak was to overcome the effect of uneven sample thickness during FTIR analysis. The reduction in \(A_{1657}/A_{1380}\) is attributed to cross-linking reaction involving the C=C during UV irradiation. Sources of C=C in the system are from the grafted MA in the alkyd, isoprene unit in the ENR50, and also from TMPTA. Figure 6 shows the proposed structure of UV-cured EA-B5T5 blend.
Figure 4. FTIR spectra of EA-BST5 blend at (a) 0 s of UV irradiation and (b) 120 s of UV irradiation. FTIR: Fourier transform infrared; UV: ultraviolet.

Figure 5. Normalised FTIR absorbance peak intensity ($A_{1657}/A_{1380}$) of EA-BST5 blend after different durations of UV irradiation. FTIR: Fourier transform infrared; UV: ultraviolet.
Figure 6. Proposed structure of UV-cured ENR50-alkyd blend comprising of benzophenone and TMPTA. ENR50: epoxidised natural rubber 50; UV: ultraviolet; TMPTA: trimethylolpropane triacrylate.

Figure 7. $T_g$ of ENR50-alkyd blends after different durations of UV irradiation. $T_g$: glass transition temperature; ENR50: epoxidised natural rubber 50; UV: ultraviolet.
UV-cured ENR50-alkyd blend

Thermal analysis. $T_g$ of the blends after they were irradiated with UV light for different duration of time is shown in Figure 7. EA-B0T0 blend acts as the control and no obvious increase in $T_g$ was observed even after 240 s of UV irradiation. It exhibits very small change in its $T_g$, that is, $<1^\circ C$. However, in EA-B5T0 blend, it showed a significant increase in $T_g$, from $-23.5^\circ C$ to $-17.2^\circ C$. The benzophenone in the blend was responsible to generate radicals to propagate cross-linking in the blend. Consequently, $T_g$ of the UV-cured EA-B5T0 blend is higher than the control.

Interestingly, the $T_g$ of all the blends were lower than the control before they were irradiated with UV. Presumably presence of small molecules such as benzophenone in the system could have disturbed the close proximity of the polymer chains in the blend, and consequently lowers the $T_g$. However, upon UV irradiation, $T_g$ of the blends have increased drastically, attributed to cross-linking reaction that has taken place. Introducing TMPTA as cross-linker into the blend has helped to further elevate the $T_g$. A molecule of TMPTA contains three reactive $C=\overset{\cd}{C}$ that are able to participate in radical

![Figure 8. TGA thermograms of (a) EA-B5T0 blend, (b) EA-B5T1 blend, (c) EA-B5T3 blend, and (d) EA-B5T5 blend. TGA: thermogravimetric analysis.](image-url)
cross-linking reaction during the UV irradiation. As shown in Figure 7 (EA-B5T1 blend), introducing 0.01 wt% of TMPTA was sufficient to increase the $T_g$ of the blend to $-16.5^\circ$C after 240 s of UV irradiation. Loading higher amount of TMPTA however did not have significant effect on the $T_g$ of the blends.

TGA thermograms of UV-irradiated blends are shown in Figure 8. As a result of UV curing, the onsets of degradation of the blends have increased. With reference to the figure, $T_{\text{onset}}$ is taken to be the temperature at which 1% of weight loss has taken place. The significant increase in the onset of degradation after 240 s of UV irradiation is another clear evidence to support the occurrence of cross-linking during UV irradiations.

**Gel content measurement and swelling test results.** Percentage of gel produced after ENR50-alkyd blends were immersed in toluene for 48 h are shown in Figure 9. UV-cured ENR50-alkyd blends showed significant increase in their gel content. It should be pointed out that individual components in the blend are easily soluble in toluene. However, upon blending and subsequent UV irradiation, the products are able to resist total dissolution in toluene. In fact, some of the UV-cured blends, especially those with TMPTA, produced more than 90% of gel (not shown). Introduction of TMPTA as cross-linker could have increased the cross-link density in the UV-cured blend and enable them to produce higher percentage of gel.
Figure 10 summarises the swelling test results, together with the cross-link density, $\nu$, and molecular weight between cross-links, $M_c$, of the UV-cured blends. The percentage of swelling of the blends has decreased with UV irradiation time. As shown in Figure 10(a), EA-B5T5 blend experienced the smallest percentage of swelling after it was UV cured, whereas EA-B0T0 blend experienced relatively high percentage of swelling despite the same exposure to UV light. The trend observed in the figure suggests that greater extent of cross-linking has taken place in the systems containing benzophenone and TMPTA. As a result of UV curing, the cross-link density in the blends has increased significantly as shown in Figure 10(b). EA-B0T0 blend however does not exhibit noticeable increase in its cross-link density due to the absence of benzophenone to generate radicals during UV irradiation. The increase in cross-link density in the UV-cured blend could resist the swelling of the polymer networks during the immersion, thus preventing it from accommodating excessive solvent. The molecular weight between cross-links has also reduced due to the increase in the cross-link density.
Conclusions

Palm stearin alkyd carrying pendent –COOH group was blended with ENR50 at ambient temperature via solvent casting technique. Results from FTIR analysis suggest that ENR50 and alkyd have interacted with each other via reactions involving the epoxide ring of the rubber and –COOH of alkyd during the mixing in toluene solution. As a result of the cross-linking reactions, up to 55% of gel insoluble in toluene was formed. In a separate set of ENR50-alkyd blend, benzophenone and TMPTA were introduced into the system and subsequently irradiated with UV light. Characterisations conducted on the UV-cured blends showed further increase in cross-link density, higher \( T_g \), higher gel content and with corresponding lower percentage of swelling compared with those that did not undergo UV curing. The cross-linking reaction during UV irradiation was plausible due to the presence of significant amount of unsaturation in the system. In conclusion, palm stearin alkyd carrying pendent –COOH groups has ring-opened the epoxide groups of ENR50 during solution blending. UV irradiation has incorporated free radical reaction involving the \(-\text{C}==\text{C}-\) in the blend, which increased the extent of cross-linking significantly.

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