The adhesion properties of natural rubber pressure-sensitive adhesives using palm kernel oil-based alkyd resins as a tackifier

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The adhesion properties of natural rubber pressure-sensitive adhesives using palm kernel oil-based alkyd resins as a tackifier

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Alkyd resins, synthesized from palm kernel oil (PKO), were investigated as the tackifiers in the formulation of natural rubber (NR) based pressure-sensitive adhesives (PSA). PKO alkyd resins were prepared via a step-wise polymerization process where the esterification was completed with the presence of xylene (solvent cook). Two alkyds, namely Alk-26 and Alk-41, with respective short and medium oil lengths of 26 and 41, were synthesized in xylene. NR was isolated from latex, dried, milled and dissolved in xylene. The alkyd solutions were then blended with NR solution in various ratios. The blend solutions were coated onto strips of corona-treated polypropylene film and the solvent removed by evaporation. The peel and shear strengths of the PSA tapes were measured. Circular samples of the blends were cast onto release paper and tested for viscoelastic properties using dynamic mechanical analysis (DMA). It was found that Alk-26 increased the shear strength of NR while Alk-41 improved the peel strength of NR significantly. These adhesion properties were comparable to some of the commercial PSA tapes available on the market. DMA study revealed that both Alk-26 and Alk-41 were immiscible with NR and there was neither cross-linking nor entanglement present in the blends.

Keywords: viscoelastic properties; natural rubber; alkyd; pressure-sensitive adhesive; tackifier

1. Introduction

Pressure-sensitive adhesives (PSA) are the type of adhesives that provide relatively low bond strength upon the application of slight pressure and usually by hand are sufficient.\textsuperscript{[1–4]} PSAs function primarily by the property of adhesion where the attraction of two different substances resulting from intermolecular forces between two different substrates. Hence, an ideal PSA must be able to flow rapidly and to resist further deformational flow as well. Therefore, during the application process, they must be able to flow over, wet the surface and eventually develop intermolecular forces to form surface attachment with the adherents.\textsuperscript{[1,3]} Usually, PSAs are coated on a film-like backing as a carrier and once the solvent evaporates, the tapes are ready for use. PSAs are found in all types of packaging and masking tapes, labels as well as surgical bandaging tapes.

Natural rubber (NR) has been widely used in many PSA formulations. In general, NR alone has a very low level of adhesion to many types of surfaces. It is well known that PSA

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performances, especially adhesion (peel strength), tack (probe tack, ball tack and loop tack) and holding power (shear strength), depend strongly on the viscoelastic properties of the adhesives. Hence, it is necessary to add tackifying resins to NR in order to produce the required balance of peel adhesion and resistance to shear in the application as PSA. A number of studies relating NR-based PSA formulated with other tackifying agents such as rosin, petroleum resins or acrylic polymers with their influences on the PSA performance have been reported in details.[5–8] There was, however, no previous report on the blending of NR and palm oil alkyds in the application of PSA.

Alkyd resin or oil-modified polyester, traditionally, is the most extensively used synthetic resin in the surface-coating industry. Alkyd-based coating is well known for its fast dryness, good corrosion protection, high gloss and ease of application even over poorly treated surface. The chemistry of the structural units connecting the ester groups allows alkyds to have immense diversity and versatility, such as labile biomedical matrices, liquid crystals, fibres and temperature resistant performance materials.[9–12]

Since palm oil is one of the major primary commodities of Malaysia, the idea of combining the two major commodities, NR and palm oil, in the application of adhesive was mooted. It was the objective of this study to investigate the performance and potential of the palm kernel oil (PKO)-based alkyds in order to produce the required balance of peel adhesion and resistance to shear of NR in the application as PSA. This work investigates the application of PKO alkyds as tackifiers in NR-based PSA with emphasis in effects of the modification of NR adhesion properties.

2. Experimental

2.1. Materials

PKO was a kind gift from Maskimi Polyols Sdn. Bhd., while NR latex was supplied by Malaysian Rubber Board. The corona-treated polypropylene plastic backing was kindly provided by Hong Leong plastics and packaging. Glycerol, lithium hydroxide, maleic anhydride and xylene were analytical grades. Other chemicals were reagent grade and were used without further purification.

2.2. Preparation of rubber solution

NR was coagulated from the latex by acetic acid. It was then pressed into thin sheet and dried in a vacuum oven at 60 °C. The dried rubber was milled on a laboratory two-roll mill with gap setting of 1–2 mm. The milled sheet was folded thrice before being put into the mill again. Seventy passes were carried out throughout the milling process. The milled rubber was dissolved in xylene to produce a 10% w/w solution.

2.3. Synthesis of alkyd resins

The formulations of the two alkyds are given in Table 1. During alcoholysis, the triglycerides of PKO were converted to monoglycerides by reaction with glycerol, catalysed by lithium hydroxide at 180–200 °C. Phthalic anhydride, glycerol and xylene were then added, and polymerization was carried out at 220–250 °C for more than 6 h. The required maleic anhydride was then added to complete the reaction. The concentrations of alkyd solutions were determined and finally diluted to 70–75% by addition of xylene. The progress of reaction was followed by measuring the evolved water of reaction and periodically checking the acid number by titration in accordance to ASTM D1980-87 (1998) with some modifications.
2.4. Determination of acid number

The acid number of the two alkyds was determined by a procedure adapted from ASTM D1980-87(1998). This test was carried out in two steps. The first step was to prepare and standardize the ethanolic potassium hydroxide solution. The second step involved sample and blank titration, where phenolphthalein solution (1% w/v) was used as the indicator. Sample and blank titrations were carried out in triplicates and the acid number was reported by averaging the results.

2.5. Determination of hydroxyl number

The hydroxyl number of the two alkyds was determined by a procedure adapted from ASTM D4274-05, test method B. This test was carried out in two steps. The first step was to prepare and standardize the standard potassium hydroxide solution; whereas, the second step involved sample and blank titrations, where phenolphthalein solution (1% w/v) was used as the indicator. Sample and blank titrations were carried out in triplicates and the acid number was reported by averaging the results.

2.6. Number-average molecular weight

Vapour pressure osmometry was used to determine the number-average molecular weight ($M_n$) of the alkyd resins. The alkyd resins were dried in vacuum and redissolved in toluene with concentration of the resin in toluene varying from 90 to 150 g/kg. An Osmomat model 070-B osmometer was used for the measurement which was calibrated with reference to solution OSMOREF® from 100 to 2000 mOsmol/kg.

2.7. Preparation of PSA

Blends of the alkyd tackifier in the milled NR were prepared in ratios of 10, 30, 50 and 70% by weight of alkyd. These blended solutions were then adjusted to contain 45% dry solids. Each of the blends was coated onto corona-treated polypropylene plastic, using the Cheminstrument laboratory drawdown coater to give a wet adhesive thickness of 12.5 μm or 0.5 mil. The coated strips were then allowed to dry in a vacuum oven for 2 h at 60 °C.

2.8. Testing of the adhesive strips

The adhesive strips were tested for peel strength in accordance to ASTM D3330-05, test method A. Peel test measured the force required to affect the peeling of the PSA tape with a constant load applied to the tape at an angle of 180°. A stainless steel panel Type 302 was used with the size of 50 mm by 125 and 2 mm thick with a bright annealed finishing. A 2 kg
hard-steel roller – 8.26 cm in diameter and 4.45 cm wide covered with rubber 0.64 cm thick and weighing 2.04 kg – was used to apply the tapes onto the panel. The peel strength was measured immediately upon sticking the tapes on the panels, utilizing the Instron Universal Testing Machine model 4466 with series IX automated materials testing system with the speed rate of 300 mm/min by a 0.5 kN load cell. The tests were carried out at 25 °C. The plate was cleaned with acetone three times before the next sample was tested. The peel strength of each sample was measured five times and the average value was taken. The standard error was found to be within 2–3%.

The shear strength was tested according to ASTM D3654-05, procedure C. This test measured the duration of the PSA tape still remain adhered to the panel under constant load, which was applied at an angle of 178° in order to negate the peel force. The load used for shear test was 1 kg. After each test, the plate was cleaned with acetone three times before the next sample was tested. Shear strength of each sample was measured five times and the average calculated. The standard error was found to be around 0.1–0.2%.

2.9. Dynamic mechanical analysis test

The viscoelastic properties of the various blends were studied using a Perkin–Elmer dynamic mechanical analysis (DMA)-7e instrument according to ASTM D4440-08. Circular samples were cast onto release paper in layers of approximately 0.1 mm wet thickness. Each layer was allowed to dry in vacuum oven at 60 °C before casting the next layer. The thickness of each of the samples was built up to 1 mm. The DMA tests were performed under temperature scan mode at a heating rate of 5 °C min⁻¹ over a temperature range from −100 to 100 °C at the frequency of 1 Hz, with a parallel plate attachment of 10 mm in diameter was used. The storage modulus \( G' \) and tan \( \delta \) of the complex shear modulus were determined.

2.10. Fourier transform infrared analysis

The Fourier transform infrared (FTIR) spectra of the alkyd resins were recorded on a Perkin–Elmer FTIR 1600 instrument for 16 scans from 4000 to 600 cm⁻¹ with resolution of 2.0. The cast film method was employed, where films of the polymers were cast from their solution directly on sodium chloride cell by evaporating off the solvent using a hot air blower.

2.11. Thermogravimetric analysis

This analysis was performed to characterize the thermal decomposition behaviour of the alkyd resins. Samples were cast onto release paper and allowed to dry in vacuum oven at 60 °C to remove solvent. Thermogravimetric analysis (TGA) was performed using a Rheometric scientific thermal analyzer of 1000 + series over the temperature ranging from 40 to 950 °C at a heating rate of 20 °C/min under the nitrogen gas atmosphere.

2.12. Differential scanning calorimetry

Rheometric Scientific differential scanning calorimetry (DSC) was used to measure the glass transition temperature \( T_g \) of the alkyd resins and blends. Approximately 5–10 mg of the sample was weighed and placed into an aluminium pan. Together with a blank aluminium pan as a reference, the sampling aluminium pan was placed into the sample chamber, which was located inside the temperature controller. DSC measurement was conducted over the temperature range from 30 to 200 °C at the heating rate of 10 °C/ min.
3. Results and discussion

3.1. Formulation of alkyd resins

The preparation of alkyds involved two steps of reaction, alcoholysis and esterification as shown in Figure 1. In alcoholysis (Figure 1(a)), the triglyceride oil was converted to monoglyceride when glycerol was added to PKO in the presence of lithium hydroxide as a catalyst. During esterification (Figure 1(b)), the first stage of reaction involved reaction between monoglycerides and phthalic anhydride to produce an alkyd polyester resin; subsequently, MA moieties were built into the alkyd polyester chain as the block co-monomer.

![Figure 1. Schematic depiction of the mechanism of (a) alcoholysis and (b) esterification and polymerization.](image-url)
In this study, two solvent-cook alkyds, Alk-26 and Alk-41, with oil length 26 and 41 respectively, were formulated under similar mechanism and conditions so that the comparison of their properties could be attributed mainly to the difference in oil length. Table 2 shows the properties of Alk-26 and Alk-41. To a major extent, the performance of alkyd resin was characterised by the oil length and fatty acid unsaturation [13,14] where oil length referred to the oil portion of an alkyd expressed as a percentage of the finished alkyd weight. Since both Alk-26 and Alk-41 were formulated in short oil length and synthesized from PKO, which had lower iodine value compared to other oils, they exhibited resin-like properties. Hence, the drying mechanism for these alkyds was expected to depend on the evaporation of solvent and not the oxidation mechanism as occurred in unsaturated alkyds.[9,15] On the other hand, both alkyds were formulated with higher hydroxyl group content. As the FTIR spectra of Alk-26 and Alk-41 appeared similar, due to the similarity in structures, for illustration, only the FTIR spectrum of Alk-26 is shown in Figure 2. From this spectrum, intense peak absorption at 3493 cm$^{-1}$ was due to $\text{–OH}$ stretching. These hydroxyl groups were expected to enhance the adhesion of the blends via hydrogen bonding across the interface of adhesive.

TGA was used to study the thermal stability of alkyds, and both thermogrammes of Alk-26 and Alk-41 showed that there were two distinct stages of thermal decompositions with similar weight loss percentage. Figure 3 displays the thermogramme of Alk-26. From this figure, the first thermal decomposition started at 180 °C and it reached the maximum rate at 320 °C with the weight loss of 17.2%; while the second weight loss of Alk-26 started at 320 °C and reached the maximum rate at 420 °C, with the weight loss of 72.2%. This indicated that Alk-26 (as well as Alk-41) contained a small amount of low molecular weight fraction, while the major part was of much high molecular weight fraction. Therefore, both alkyds could function as plasticizer with the presence of its low molecular weight fraction while its high molecular weight fraction will enable rapid drying during PSA tape coating. [9,16] The rapid drying is essential to enhance the PSA performance as the percentage of the occluded solvent in the dried tape could significantly affect the PSA performance. From the study of Toyama et al., they found that the occluded solvent down to even at the level of 0.01% could affect the result of the probe tack significantly.[17]

3.2. Adhesive properties

Adhesive properties basically comprise of peel and shear strength measurements. Peel strength describes the ability of the tape to resist against peeling force and could be characterized by peel test whereas shear test measured the resistance of the tape towards creep force. In this study, both tests were carried out to characterize the adhesive properties of the NR-alkyd PSA tapes and six selected commercial tapes so that comparison could be made later.

<table>
<thead>
<tr>
<th>Property</th>
<th>Alkyd resin</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alk-26</td>
<td>Alk-41</td>
</tr>
<tr>
<td>Oil length</td>
<td>26</td>
<td>41</td>
</tr>
<tr>
<td>Acid number (mg KOH/g)</td>
<td>67.2</td>
<td>48.1</td>
</tr>
<tr>
<td>Glass transition temperature*, $T_g$ (°C)</td>
<td>66.7</td>
<td>37.3</td>
</tr>
<tr>
<td>Hydroxyl number (mg KOH/g)</td>
<td>96.4</td>
<td>38.3</td>
</tr>
<tr>
<td>Number average molecular weight, $M_n$</td>
<td>8288</td>
<td>4956</td>
</tr>
</tbody>
</table>

* $T_g$ measurement was made using DSC.
Table 3 shows the adhesive properties of the selected six commercial tapes. It was found that the peel strength of the six commercial tapes fell between 28.9 and 33.9 N/100 mm and the difference was very small. However, the shear strength (in minutes) of the selected commercial tapes covered a wide range of values, from the longest duration of 69.5 min to the shortest of only 10.2 min.

The adhesive properties of NR, Alk-26 and NR/Alk-26 blends were shown in Table 4. From this table, the peel strength of pure Alk-26 and pure NR was close to each other, 2.92 and 2.50 N/100 mm respectively. It was found that NR/Alk-26 blends performed well when NR was the dominant constituent in the blend. The peel strength for the NR/Alk-26 blends peaked at the 30 wt.% of alkyd and the value was nearly two times that of either pure Alk-26 or pure NR. This showed that Alk-26 managed to improve the peel strength of NR, although...
the values were lower compared to the peel strength of commercial tapes (Table 3). However, Alk-26 had overall successfully reinforced the shear strength of NR and achieved even better values than all the selected commercial tapes. The shear strength of the blends, as shown in Table 4, peaked at 50 wt.% of alkyd with a maximum value of 72.7 min.

Figure 5 shows the adhesive properties of NR, Alk-41 and NR/Alk-41 blends. In fact, pure Alk-41 had stronger peel strength (about 31 times) than pure NR and thus improved the peel strength of NR much better than Alk-26. From Table 5, the peel strength of NR/Alk-41 increased significantly with the weight per cent of alkyd. The values increased with the higher weight per cent of alkyd in the blends and the maximum peel strength was 48.4 N/100 mm at 70 wt.% of alkyd. However, the shear strength of the blends was much weaker than the pure NR. Overall, the values decreased with increasing weight per cent of Alk-41 in the blends. In fact, the shear strength of pure Alk-41 was even less than 1% than that of pure NR. Hence, this result revealed that Alk-41 had failed to improve the shear strength of NR.

Two main factors have been identified as the main causes that influenced the changes of peel and shear strengths.[3,18] Higher molecular weight and higher degree of cross-linking were expected to cause a decrease in peel strength but an increase in shear strength. Thus, it was inferred that Alk-26 with higher number-average molecular weight, $M_n$, (refer to Table 2) improved the shear strength of NR more effectively than Alk-41.

On the other hand, Alk-26 with higher oil length depicted that the presence of more fatty acid side chains would impart a more non-polar character to the polymer. By relative comparison, Alk-26 would be more polar due to its shorter oil length and higher hydroxyl number (Table 2). Thus, Alk-26 would also become more reactive towards material that could interact with its hydroxyl groups. This hydrogen bonding effect on shear strength was similar to the cross-linking [3] and resulting in higher shear strength for Alk-26.

Alk-26 also exhibited higher $T_g$ than Alk-41 (Table 2) and would therefore be less sticky at room temperature, thus resulting in lower peel strength. Besides, pure Alk-26 appeared to be stiff at room temperature, causing an uneven stress distribution over the bonding area. However, Alk-41 appeared in a form of viscous liquid, which enabled the distribution of peeling stress uniformly over a wider bonding area and, as a result, exhibited greater resistance towards the peeling force.

Table 3. Adhesive properties of the commercial PSA tapes.

<table>
<thead>
<tr>
<th>Commercial tape code</th>
<th>Peel strength in N/100 (mm)</th>
<th>Shear strength in min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>31.0</td>
<td>10.2</td>
</tr>
<tr>
<td>2</td>
<td>31.7</td>
<td>10.5</td>
</tr>
<tr>
<td>3</td>
<td>28.9</td>
<td>18.8</td>
</tr>
<tr>
<td>4</td>
<td>33.0</td>
<td>69.3</td>
</tr>
<tr>
<td>5</td>
<td>29.9</td>
<td>69.5</td>
</tr>
<tr>
<td>6</td>
<td>33.9</td>
<td>36.7</td>
</tr>
</tbody>
</table>

Table 4. Adhesive properties of NR/Alk-26 PSA tapes.

<table>
<thead>
<tr>
<th>Weight % of alkyd in NR/Alk-26 blends</th>
<th>Peel strength in N/100 (mm)</th>
<th>Shear strength in min</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.92</td>
<td>14.0</td>
</tr>
<tr>
<td>10</td>
<td>4.36</td>
<td>30.1</td>
</tr>
<tr>
<td>30</td>
<td>4.64</td>
<td>59.2</td>
</tr>
<tr>
<td>50</td>
<td>3.80</td>
<td>72.7</td>
</tr>
<tr>
<td>70</td>
<td>2.13</td>
<td>70.0</td>
</tr>
<tr>
<td>100</td>
<td>2.50</td>
<td>54.1</td>
</tr>
</tbody>
</table>
3.3. Viscoelastic properties

The adhesive properties of PSA were greatly influenced by two types of viscoelastic properties, namely the storage modulus ($G'$) and the glass transition temperature ($T_g$), and to some extent, by the compatibility or the miscibility of both NR and alkyds in the blends. The $T_g$ of the blend corresponds to the maximum tan $\delta$ ($\delta_{\max}$) in the DMA graph. The variations of tan $\delta$ of the NR/Alk-26 and NR/Alk-41 blends were plotted in Figures 4 and 5. From these figures, both NR/Alk-26 and NR/Alk-41 blends exhibited two $T_g$s, indicating that the blends were immiscible at all weight per cent of alkyd. Therefore, complete phase separation could happen over the entire range of weight per cent of alkyd. Since the $T_g$ of the NR in the blends was not shifted to a higher temperature with increase in percentage of alkyd, there was no cross-linking between NR and alkyds.

The immiscibility of the blending system could affect the adhesive properties of the blends.[5,7,19] From our preliminary observation, phase separation occurred faster when the alkyd resins were the dominant constituent in the blend solutions, and NR/Alk-41 blends experienced faster phase separation rate than NR/Alk-26 blends. At 90 wt.% of alkyd, Alk-41 separated and formed a top layer of the blend solution within one day but phase separation in NR/Alk-26 blend solution happened slowly, and it took approximately one month to observe visually. This observation suggested that a resin-rich area of Alk-41 could have formed at the surface of tape coating, especially during the tape drying process in a vacuum oven and thus

![Figure 4. Tangent delta of the NR/Alk-26 blend.](image)

Table 5. Adhesive properties of NR/Alk-41 PSA tapes.

<table>
<thead>
<tr>
<th>Weight % of alkyd in NR/Alk-41 blends</th>
<th>Peel strength in N/100 (mm)</th>
<th>Shear strength in min</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.92</td>
<td>14.0</td>
</tr>
<tr>
<td>10</td>
<td>6.43</td>
<td>4.18</td>
</tr>
<tr>
<td>30</td>
<td>7.05</td>
<td>2.94</td>
</tr>
<tr>
<td>50</td>
<td>10.5</td>
<td>1.78</td>
</tr>
<tr>
<td>70</td>
<td>48.4</td>
<td>1.07</td>
</tr>
<tr>
<td>100</td>
<td>89.6</td>
<td>0.08</td>
</tr>
</tbody>
</table>
eventually contributing to the overall high values of peel strength for the NR/Alk-41 PSA tapes. On the other hand, since phase separation in NR/Alk-26 occurred at a slower rate, the blends required larger deformation process, and this explained that Alk-26 had stronger holding power and thus improved the shear strength of NR more effectively than that of Alk-41.

The immiscibility of the blending system could also affect the storage modulus, $G'$ of PSA. From Figures 6 and 7, the addition of alkyds to NR elevated the $G'$ values more than 0.5 MPa. According to the Dahlquist criteria, the $G'$ values should be less than 0.5 MPa [20] to enable good contact between the adhesive and the substrate through the lightest finger or hand pressure. Generally, a PSA tape required a $G'$ at room temperature between 0.5 and $2 \times 10^5$ Pa.[21] Hence, the NR/Alkyd solution adhesives did not exhibit significantly balanced PSA properties.

The DMA study also showed that the logarithmic values of $G'$ were always greater than $G''$ in all NR/Alk-26 and NR/Alk-41 blends, and in the absence of entanglements, the $G'$

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Figure 5. Tangent delta of the NR/Alk-41 blend.

Figure 6. Logarithmic plot of the storage modulus of the NR/Alk-26 blend.
appeared to be parallel with $G''$. This designated that the elastic behaviour had generally dominated the viscous flow. One possible explanation was that the molecular weight of these alkyds was below the required critical entanglement molecular weight ($M_c$) [22,23] and thus was insufficient to create crossover between $G'$ and $G''$.

4. Conclusion
From the adhesive properties’ measurements, NR/Alk-41 blends showed significantly higher values of peel strength compared to those of commercial tapes, but failed to increase the shear strength of NR. Alk-26 increased the shear strength of NR more effectively than Alk-41 but did not increase the peel strength significantly. DMA study had shown that both Alk-26 and Alk-41 were immiscible with NR but had increased the storage modulus of NR significantly. Notwithstanding that, DMA tests revealed neither cross-linking nor entanglement in the blending systems of NR with Alk-26 and Alk-41, respectively. Overall, this study showed that PKO-based alkyd resins could be used as a potential tackifier to modify the PSA properties of NR. The peel and shear strengths of NR have improved by solution blending with Alk-41 and Alk-26 respectively, and these values were found to be compatible to some of the commercial PSA tapes available in the market. However, further study on the reaction between the PKO-based alkyds with NR is needed in order to improve the immiscibility of the blending system and to enhance the adhesion properties of NR PSA.

References


