

Research Article

Dependence of Adhesion Property of Epoxidized Natural Rubber (ENR 25)/Ethylene-Propylene-Diene Rubber Blend Adhesives Crosslinked by Benzoyl Peroxide

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The loop tack, peel strength, and shear strength of crosslinked epoxidized natural rubber (ENR 25)/ethylene-propylene-diene rubber (EPDM) blend adhesives were investigated. Coumarone-indene resin, toluene, and benzoyl peroxide were used as the tackifier, solvent, and crosslinking agent, respectively, throughout the experiment. The adhesive was coated on a polyethylene terephthalate (PET) substrate using a SHEEN hand coater at 60 μm and 120 μm coating thickness. It was cured at 80°C for 30 minutes before testing on a Lloyd adhesion tester operating at testing rates from 10 to 60 cm min^{-1} . Results show that loop tack and peel strength of the ENR 25/EPDM adhesive pass through a maximum value at 2 parts per hundred parts of rubber (phr) of benzoyl peroxide content. This observation is attributed to the increase in crosslinking which enhances the cohesive strength of the adhesive. Further addition of the crosslinking agent decreases the tack and peel strength due to the decrease in wettability of the over-crosslinked adhesive. Shear strength, however, increases steadily with benzoyl peroxide content, an observation which is associated with the steady increase in the cohesive strength. The adhesion properties increase with increasing coating thickness and testing rate.

1. Introduction

Natural rubber has been widely used to prepare pressure-sensitive adhesives [1]. One of the main reasons is that natural rubber is a renewal bioresource material. Natural rubber is known to exhibit several outstanding properties due to its ability to crystallize under stretching, that is, strain-induced crystallization [2, 3]. In formulating a rubber-based pressure-sensitive adhesive, an elastomer provides the elastic component while a low molecular weight tackifying resin imparts the viscous component. However, natural rubber alone has low tack and adhesion to surfaces. Hence, it is necessary to add tackifying resins to the rubber to increase its adhesion properties. It is generally accepted that adhesion properties of pressure-sensitive adhesives depend strongly on the viscoelastic properties of the adhesives. Many studies on the uncrosslinked natural rubber have been carried out. However, the adhesion properties of crosslinked rubber-based adhesives are scarcely reported in the literature. Hamed and Preechatiwong [4] found that during peeling action,

strain-induced crystallization occurs for uncrosslinked and lightly crosslinked natural rubber. In the case of highly crosslinked natural rubber, no strain-induced crystallization was observed. Neoh et al. [5] studied the adhesive properties of cured styrene-natural rubber adhesives. Meanwhile, Basak et al. [6] discovered that the peel strength of vulcanized EPDM cocured with tackifier containing unvulcanized EPDM depends on compatibility and viscoelastic properties of the adhesives. Kajtna and Krajnc [7] found that adhesive properties are strongly influenced by the degree of crosslinking of the microsphere. The decrease in adhesion may be correlated with higher crosslinking density. Roseley et al. [8] investigated the creep response of thixotropic ambient temperature cure adhesives measured by DMTA in static tension and shear. It is observed that at high stress and temperatures, the adhesives eventually fail by rupture of the adhesive bonds. Meanwhile, Thitithammawong et al. [9] observed that shear strength and peel strength of adhesives depend on the vulcanization systems. Nakamura et al. [10] studied the influence of crosslinking and peeling rate on tack properties of polyacrylic

pressure-sensitive adhesives. It is revealed that the interfacial adhesion increased while the cohesive strength decreased as crosslinking and probe rate are decreased. On the other hand, Czech et al. [11] discovered that the selection of suitable photoinitiator plays an important role in obtaining the optimum properties of acrylic pressure-sensitive adhesive including tack, peel adhesion, and shear strength. Recently, we have also reported on the adhesion properties of crosslinked natural rubber-based adhesives [12–14]. It is observed that loop tack and peel strength pass through a maximum value before decreasing with further addition of benzoyl peroxide. This finding is associated with the optimum crosslinking of rubber chains which enhances the cohesive and adhesive strength of the adhesive. Shear strength, however, increases steadily with benzoyl peroxide loading due to the steady increase of the cohesive strength as the rubber chains are crosslinked. Our study reported so far involves the effect of crosslinking on the adhesion properties of adhesive prepared from a single component of rubber. There is no research published on the adhesion behaviour of rubber blends adhesives crosslinked by benzoyl peroxide. In view of the lack of research data in this field of interest, it is thus the objective of this paper to report and discuss our investigation on the adhesion properties of crosslinked ENR 25/EPDM blend adhesives. Based on our previous study on the uncrosslinked ENR 25/EPDM system [15], the blend ratio was chosen at 60% ENR 25 where maximum tack and peel strength occur.

2. Experimental

2.1. Materials. Epoxidized natural rubber (ENR 25 grade) with 25 mol% epoxidation and ethylene-propylene-diene rubber (EPDM) were selected as the elastomers for the preparation of the pressure-sensitive adhesives. ENR 25 was supplied by the Rubber Research Institute of Malaysia whereas EPDM was obtained from Bayer Company. The EPDM used consists of 67% ethylene and 4.3% ethylidene norbornene (ENB) contents. The glass transition temperatures of ENR 25 and EPDM are -45°C and -60°C , respectively. The respective Mooney viscosity of the two rubbers is 110 and 63. Coumarone-indene resin, toluene, and benzoyl peroxide were used as the tackifier, solvent, and crosslinking agent, respectively. Polyethylene terephthalate (PET) film was chosen as the coating substrate throughout the experiment. This is because PET film can withstand heating in an oven at 80°C for 30 minutes during crosslinking of the rubber blend adhesives. Also, coating on PET film resembles the production of a rubber adhesive tape. All the materials and chemicals used were freshly supplied and no purification was carried out prior to use.

2.2. Preparation of Adhesive. A two-roll mill was used to masticate the rubber for 10 minutes. The total weight for each rubber blend sample was 5 g, that is, 3 g of masticated ENR 25 and 2 g of masticated EPDM rubber. This means that 60% (3/5) ENR 25 in the ENR 25/EPDM rubber blend was used to prepare the adhesive. The rubbers were cut into small pieces before dissolving in 30 mL of toluene. The tightly closed rubber-toluene mixture was kept for 24 hours at 30°C

to ensure complete dissolution of the rubbers. A fixed amount of 2 g of pulverized coumarone indene resin (40 phr) was added slowly to the rubber solution with constant stirring. The mixture was then left for 2 hours before the addition of benzoyl peroxide. Five different dosages of benzoyl peroxide, that is, 0.05, 0.10, 0.15, 0.20, and 0.25 g corresponding to 1, 2, 3, 4, and 5 phr of benzoyl peroxide, were added to the adhesive solution. One control sample without benzoyl peroxide was also prepared for comparison purposes.

2.3. Measurement

2.3.1. Tack. Loop tack is essentially a peel test involving low contact pressure and short application time [16]. A SHEEN hand coater was used to coat the ENR 25/EPDM blend adhesive on a PET film (with dimensions of $4\text{ cm} \times 25\text{ cm}$) at the centre of coating area ($4\text{ cm} \times 4\text{ cm}$) at $60\text{ }\mu\text{m}$ and $120\text{ }\mu\text{m}$ coating thicknesses. The coated sample was conditioned at 30°C for 24 hours before heating in an oven at 80°C for 30 minutes in order to crosslink the rubber blend. Then, a loop was formed and the adhesive-coated portion of the substrate was gently brought into contact with a clean glass plate. The debonding force from the glass plate was determined by a Lloyd adhesion tester operating at testing rates from 10 to 60 cm min^{-1} . The three highest peaks from the load-propagation plot recorded were used to calculate the average debonding force. The loop tack is expressed as the debonding force per area of contact with the glass plate (N m^{-2}).

2.3.2. Peel Strength. Three modes of peel test, namely, T-peel, 90° -peel, and 180° -peel tests were used to determine the peel strength of the ENR 25/EPDM blend adhesives. The dimensions of the test samples were shown in Table 1.

The adhesive was coated from the end of the PET substrate using a SHEEN hand coater at a coating area of $10\text{ cm} \times 4\text{ cm}$ at $60\text{ }\mu\text{m}$ and $120\text{ }\mu\text{m}$ coating thicknesses. Another uncoated PET film was then carefully laid on the coated PET film. The coated testing specimen was conditioned at 30°C for 24 hours before heating in an oven at 80°C for 30 minutes to crosslink the rubber blend adhesive. The peel force was determined by a Lloyd adhesion tester operating at testing rates from 10 to 60 cm min^{-1} . The average peeling force was computed from the three highest peaks recorded from the load-propagation graph. Peel strength is expressed as the average load per width of the bond line required to separate progressively a flexible member from a rigid member or another flexible member (ASTM D 907).

2.3.3. Shear Strength. A PET shear test sample with dimensions of $20\text{ cm} \times 4\text{ cm}$ was coated from the end of the substrate to form the base stock at a coating area of $10\text{ cm} \times 4\text{ cm}$ using a SHEEN hand coater. One end of another PET film (face stock) was then slowly placed on the coated area of the base stock to form a shear test specimen. The shear sample was conditioned at 30°C for 24 hours before heating in an oven at 80°C for 30 minutes to crosslink the rubber blend adhesive. The shear force was determined by a Lloyd adhesion tester operating at $10\text{--}60\text{ cm min}^{-1}$. The testing distance was 10 cm which corresponded to the coated length of the shear sample.

TABLE I: Dimensions of peel test samples.

Mode of peel test	Base stock	Face stock
T -peel	20 cm \times 4 cm	20 cm \times 4 cm
90°-peel	20 cm \times 4 cm	15 cm \times 7 cm
180°-peel	25 cm \times 4 cm	10 cm \times 10 cm

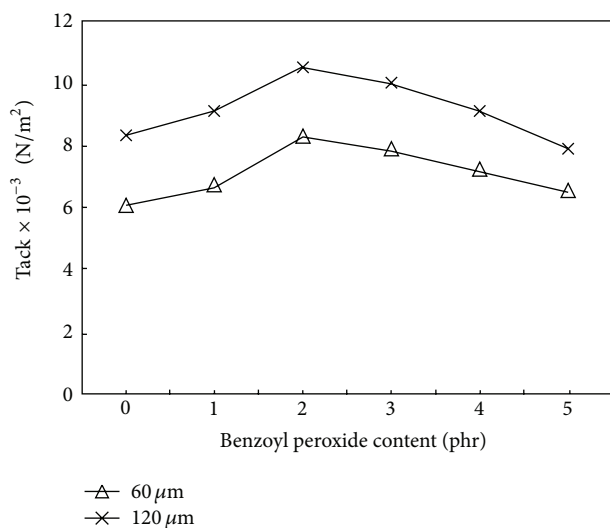


FIGURE 1: Variation of loop tack of ENR 25/EPDM blend adhesive with benzoyl peroxide content at 60 μm and 120 μm coating thickness.

The peak force of a plot of force against time was taken as the shear force. Shear strength is expressed as the shear force per unit area of testing (N m^{-2}).

3. Results and Discussion

The dependence of adhesion strength on benzoyl peroxide content and testing rate of the ENR 25/EPDM adhesives is discussed below.

3.1. Tack. The effect of benzoyl peroxide content on loop tack at 60 μm and 120 μm coating thicknesses is shown in Figure 1. The loop tack value for the neat ENR 25/EPDM (i.e., at zero content of benzoyl peroxide) is $6.101 \times 10^3 \text{ N/m}^2$ and $8.339 \times 10^3 \text{ N/m}^2$ at 60 μm and 120 μm coating thicknesses, respectively. The corresponding loop tack is $4.448 \times 10^3 \text{ N/m}^2$ and $6.517 \times 10^3 \text{ N/m}^2$ for neat ENR 25 whereas the respective values for neat EPDM are $4.713 \times 10^3 \text{ N/m}^2$ and $6.049 \times 10^3 \text{ N/m}^2$. From the plot, it is obvious that loop tack increases with benzoyl peroxide loading up to 2 phr and decreases with further addition of the crosslinking agent. The initial increase in tack is associated with the increase in cohesive strength of the adhesive due to the crosslinking of the rubber blend adhesive. However, after the optimum benzoyl peroxide loading at 2 phr, over-crosslinking of the rubber blend results in the decrease of wettability of adhesive as exhibited by the drop in tack value as shown in Figure 1. Over-crosslinked rubber hinders the chain mobility and increases the elastic

component of the adhesive. The failure mode is predominantly adhesive in nature which occurs at the interface between adhesive and substrate. From our recent study on the effect of benzoyl peroxide loading on adhesion property of single component rubber adhesives [12–14], maximum tack is also observed at an optimum benzoyl peroxide loading. This observation suggests that both single component and blend rubber-based adhesives exhibit maximum tack at an optimum benzoyl peroxide loading. Hence, it can be inferred that the dependence of tack on benzoyl peroxide content is similar for both rubber systems. In other words, the effect of crosslinking eclipses the rubber composition in the adhesive system. The effect of coating thickness is also indicated in Figure 1. Two coating thicknesses were used in order to compare the effect of coating thickness on the tack and other adhesion properties of the ENR 25/EPDM blend adhesives. For a fixed benzoyl peroxide concentration, the 120 μm coated sample consistently shows higher tack values than those of the 60 μm coated sample. This observation is attributed to the increase in the amount of adhesive in the thicker sample which enhances the viscoelastic response of the adhesive [17]. When coating thickness is increased, failure mode shifts from cohesive to adhesive failure [18]. Owing to the instrument constraint, the highest coating thickness that is carried out in this study was 120 μm . Based on our previous investigation on the adhesion properties of acrylonitrile-butadiene rubber/Standard Malaysian Rubber blend based pressure-sensitive adhesive [19], the peel strength and shear strength increase with coating thickness. However, the rate of increase of the adhesion properties with coating thickness decreases at higher thicknesses. Hence, it is suggested that the optimum coating thickness is about 150 μm thickness. Figure 2 illustrates the variation of tack with testing rate at 2 phr benzoyl peroxide content for 60 μm and 120 μm coating thicknesses. From the graph, it shows that tack increases steadily with testing rate for both coating thicknesses, an observation which is attributed to the change in viscoelastic response at various testing rates. At low testing rate, the response is predominantly viscous and cohesive failure occurs whilst at high testing rate, the response is predominantly elastic resulting in the adhesion failure [1]. The elastic component of rubber blend adhesive becomes significant at higher testing rate and adhesion failure occurs as shown by the higher tack value in Figure 2. The average rate of increase of tack with testing speed is $53.34 \text{ Nm}^{-2}/\text{cm min}^{-1}$ and $58.88 \text{ Nm}^{-2}/\text{cm min}^{-1}$ for 60 μm and 120 μm coating thickness, respectively. The result shows that there is no linear correlation between coating thickness and the rate of increase of tack. This observation suggests that the increment of tack per unit testing rate is marginally dependent on coating thickness.

3.2. Peel Strength. The dependence of peel strength on benzoyl peroxide content is shown in Figure 3 for a T -peel test at 60 μm and 120 μm coating thicknesses. The peel strength for the neat ENR 25/EPDM is 82.75 N/m and 116.38 N/m at 60 μm and 120 μm coating thicknesses, respectively. The corresponding values are 41.15 N/m and 63.55 N/m for neat

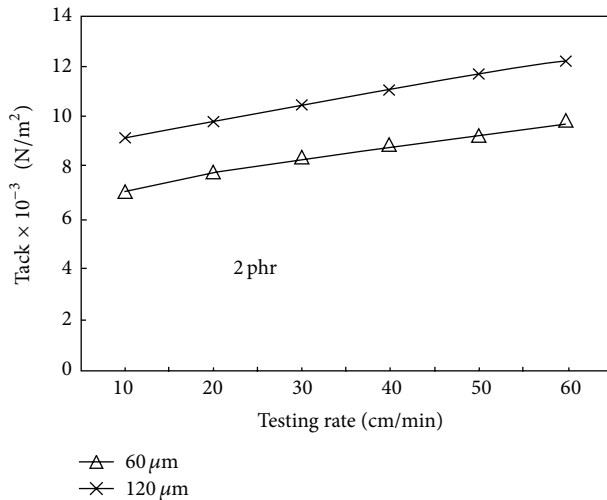


FIGURE 2: Variation of loop tack of ENR 25/EPDM blend adhesive with testing rate at 2 phr benzoyl peroxide content at 60 μm and 120 μm coating thickness.

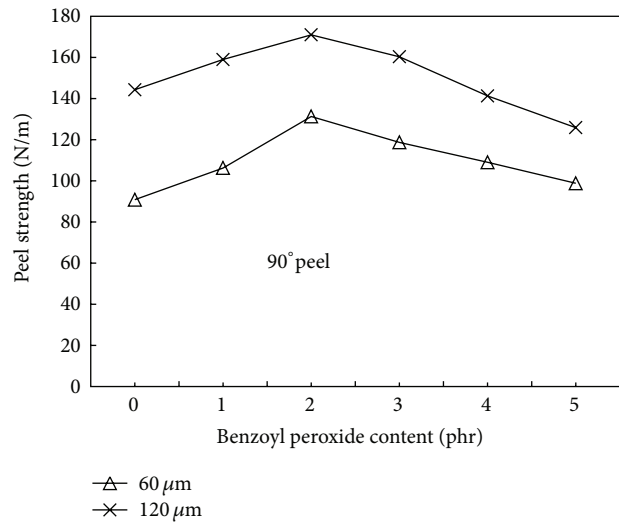


FIGURE 4: Variation of peel strength (90°-test) of ENR 25/EPDM blend adhesive with benzoyl peroxide content at 60 μm and 120 μm coating thickness.

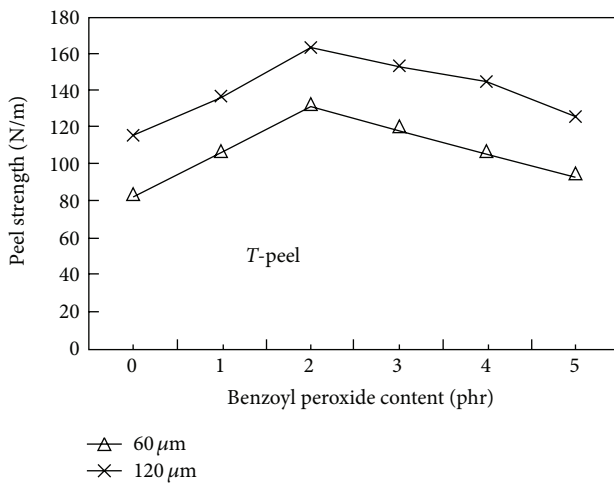


FIGURE 3: Variation of peel strength (T-test) of ENR 25/EPDM blend adhesive with benzoyl peroxide content at 60 μm and 120 μm coating thickness.

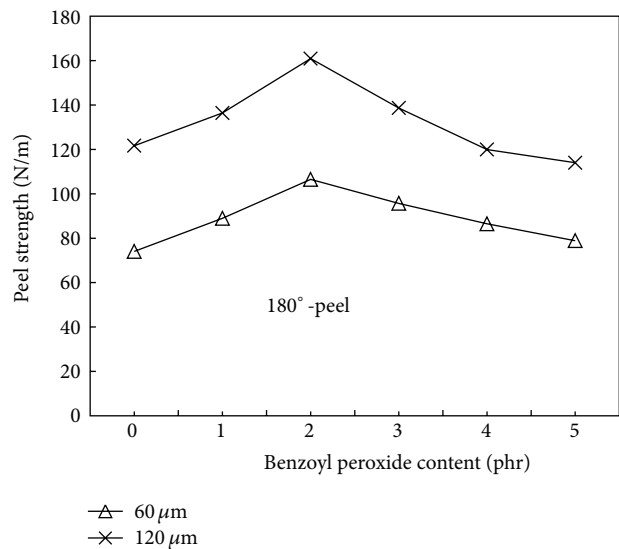


FIGURE 5: Variation of peel strength (180°-test) of ENR 25/EPDM blend adhesive with benzoyl peroxide content at 60 μm and 120 μm coating thickness.

ENR 25 whereas the respective values for neat EPDM are 29.90 N/m and 67.53 N/m . As in the case of tack, peel strength also increases with benzoyl peroxide content up to 2 phr before decreasing with further addition of the crosslinking agent. This observation can be explained by the steady increase in cohesive strength resulting from the increase in crosslinking of the rubber blend adhesive. The combined effect of cohesive and adhesive strength culminates at the optimum benzoyl peroxide loading of 2 phr. Further loading beyond the optimum loading decreases the wettability of adhesive due to over-crosslinking rubber blend which increases the elastic component of the adhesive. At this stage, the elastic component of adhesive plays a dominant role in the debonding process between the adhesive and the adherend [1] in the adhesion failure. Similar adhesion behavior is also observed for the 90° - and 180°-peel tests as shown in Figures 4

and 5, respectively. For the 90°-peel test, the peel strength for the neat ENR 25/EPDM is 90.83 N/m and 144.28 N/m at 60 μm and 120 μm coating thicknesses, respectively. The corresponding values are 38.35 N/m and 93.30 N/m for neat ENR 25 whereas for neat EPDM, the respective values are 20.80 N/m and 70.80 N/m . In the case of 180°-peel test, the peel strength for the neat ENR 25/EPDM is 74.08 N/m and 121.68 N/m at 60 μm and 120 μm coating thicknesses, respectively. The corresponding values are 41.00 N/m and 85.60 N/m for neat ENR 25 whereas the respective values for neat EPDM are 18.33 N/m and 64.95 N/m . Both plots also indicate that the optimum benzoyl peroxide content occurs at 2 phr loading, a value which is consistent with

the result observed for the *T*-peel test. At 2 phr benzoyl peroxide content, maximum peel strength is observed for the two modes of peel tests due the culmination of adhesive and cohesive strength as discussed earlier. The decrease in peel strength after the optimum benzoyl peroxide loading is again attributed to the decrease in wettability arising from the over-crosslinking of the rubber blend adhesive where the viscoelastic response is predominantly elastic in nature. Figures 3–5 also show that 120 μm coated sample indicates higher peel strength compared to 60 μm coated sample, an observation which is ascribed to the higher amount of adhesive in the former sample that enhances the viscoelastic property of the adhesive. The dependence of peel strength on testing rate for the 90°-peel test at 2 phr benzoyl peroxide loading is shown in Figure 6. The 90°-peel test was selected for the testing rate study because the 90° testing mode gives the highest peel strength among the three modes of peel tests. As in the case of tack study, peel strength also increases steadily with testing rate for the 60 μm and 120 μm coating thicknesses. This behavior is attributed to the different viscoelastic response with the testing rate. At low peeling rate, the viscoelastic response is predominantly viscous and the adhesive fails to resist internal separation; that is, cohesive failure occurs. On the other hand, as the testing rate is increased, the viscoelastic response is predominantly elastic response [20, 21]. The increase in elastic response is accompanied by the enhancement of the adhesive strength as shown by the increase in peel strength with testing rate. Based on Figure 6, the average rate of increase of peel strength with testing speed is $1.52 \text{ Nm}^{-1}/\text{cm min}^{-1}$ and $1.59 \text{ Nm}^{-1}/\text{cm min}^{-1}$ for 60 μm and 120 μm coating thickness, respectively. The result indicates that the increment of peel strength per unit testing rate is virtually independent on the coating thickness of adhesive.

3.3. Shear Strength. Figure 7 shows the variation of shear strength with benzoyl peroxide content at 60 μm and 120 μm coating thickness. The shear strength for the neat ENR 25/EPDM is $16.221 \times 10^4 \text{ N/m}^2$ and $23.943 \times 10^4 \text{ N/m}^2$ at 60 μm and 120 μm coating thicknesses, respectively. The corresponding values are $19.309 \times 10^4 \text{ N/m}^2$ and $29.864 \times 10^4 \text{ N/m}^2$ for neat ENR 25 whereas the respective values for neat EPDM are $11.631 \times 10^4 \text{ N/m}^2$ and $18.120 \times 10^4 \text{ N/m}^2$. The plot clearly indicates that shear strength increases steadily with benzoyl peroxide loading for both coating thicknesses. This observation is associated with the increase in crosslinking of rubber blend which enhances the cohesive strength of adhesive as benzoyl peroxide loading is increased. The enhancement of cohesive strength means the increase in resistance towards shear action of the adhesive—an indication of holding power—as shown by the increase in the shear strength in Figure 7. For a fixed benzoyl peroxide loading, the 120 μm coated sample consistently indicates higher shear strength compared to that of the 60 μm coated sample. This observation is attributed to the higher amount of adhesive in the 120 μm coated sample which provides higher resistance to shearing action, and hence higher shear strength is observed as shown in Figure 7. Figure 8 illustrates the effect of testing

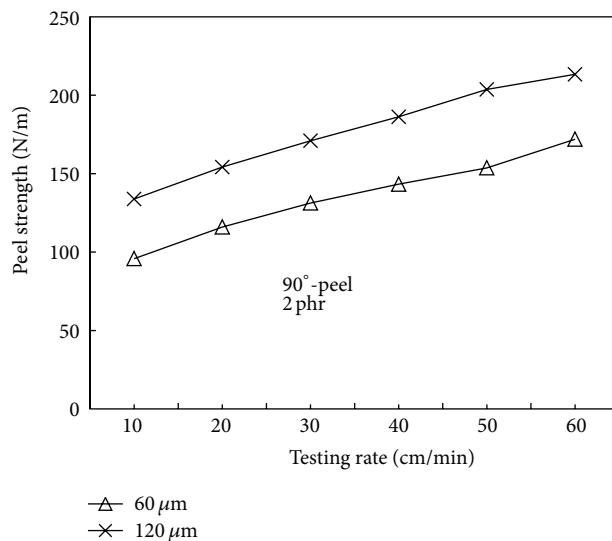


FIGURE 6: Variation of peel strength (90°-test) of ENR 25/EPDM blend adhesive with testing rate at 2 phr benzoyl peroxide content at 60 m and 120 m coating thickness.

rate on the shear strength at 2 phr benzoyl peroxide content for both coating thicknesses. As in the case of tack and peel strength, shear strength also increases with testing rate for both coating thicknesses. This observation is attributed to the different viscoelastic response as the testing rate is increased. As mentioned earlier, the viscoelastic response is predominantly viscous at low rate of testing and the failure mode is cohesive in nature whereas at higher testing rate, the viscoelastic response is predominantly elastic and adhesive failure mode occurs [1]. The increase in adhesive failure with testing rate accounts for the increase in shear strength as shown in Figure 8. The average rate of increase of peel strength with testing speed is $2.46 \times 10^3 \text{ Nm}^{-2}/\text{cm min}^{-1}$ and $2.61 \times 10^3 \text{ Nm}^{-2}/\text{cm min}^{-1}$ for 60 μm and 120 μm coating thickness, respectively. This result suggests that the increment of shear strength per unit testing rate is not significantly affected by the coating thickness of adhesive, an observation similar to that reported in the tack and peel strength section.

4. Conclusions

Loop tack and peel strength of ENR 25/EPDM blend adhesive increase with benzoyl peroxide content up to 2 phr loading before decreasing with further addition of the crosslinking agent, an observation which is associated with the increase in the cohesive strength arising from the crosslinking of the rubber blend chains. The drop of tack and peel strength after the optimum benzoyl peroxide content is due to over-crosslinking of rubber blend chains which results in the decrease of wettability of the adhesive. For the shear strength, it increases steadily with benzoyl peroxide loading for both coating thicknesses. This observation is ascribed to the steady enhancement of cohesive strength of adhesive due to the steady increase of crosslinking of rubber blend chains. In all cases, the adhesion properties of the blend adhesive increase

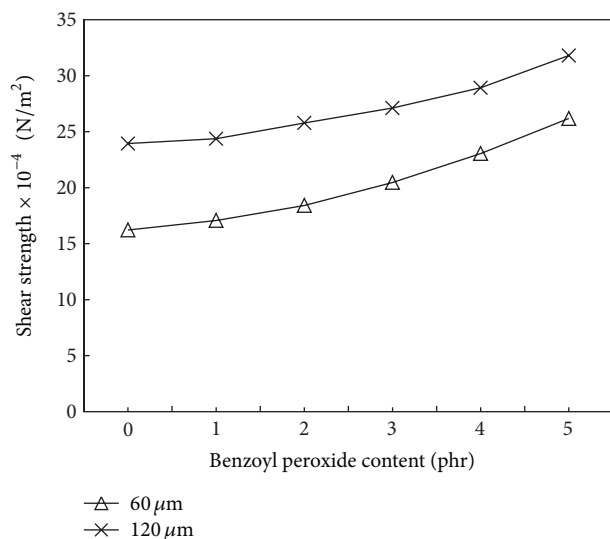


FIGURE 7: Variation of shear strength of ENR 25/EPDM blend adhesive with benzoyl peroxide content at 60 μm and 120 μm coating thickness.

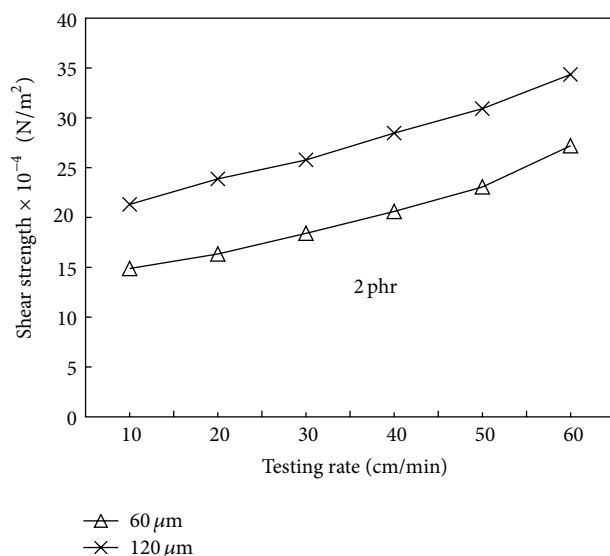


FIGURE 8: Variation of shear strength of ENR 25/EPDM blend adhesive with testing rate at 2 phr benzoyl peroxide content at 60 m and 120 m coating thickness.

with coating thickness. This phenomenon is associated with the increase in adhesive amount in thicker sample which enhances the viscoelastic response of the adhesive. Tack, peel, and shear strength of the blend adhesive increase steadily with testing rate. This rate-dependent adhesion property is attributed to the different viscoelastic response with testing rate; that is, viscous response occurs at lower testing rate whereas at higher testing rate, elastic response is predominant. The corresponding failure mode changes from cohesive failure at low testing rate to adhesive failure at higher testing rate. The increment of adhesion properties per unit testing rate, however, is not significantly affected by the coating thickness of the adhesive.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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