# Effects of Polyethylene Grafted Maleic Anhydride on the Mechanical, Morphological, and Swelling Properties of Poly (Vinyl Chloride) / Epoxidized Natural Rubber / Kenaf Core Powder Composites

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The effects of polyethylene grafted maleic anhydride (PE-g-MA) on the properties of poly (vinyl chloride)/epoxidized natural rubber (PVC/ENR) kenaf core powder composites were studied, with four different loadings of kenaf core powder (5, 10, 15, and 20 phr). The tensile properties indicated that the strength and elongation at break of the composites exhibited an increase for samples with PE-g-MA. Morphological analysis using a scanning electron microscope (SEM) showed better dispersion of kenaf fiber with the addition of PE-g-MA and less kenaf powder agglomeration. Furthermore, the swelling index indicated that composites with PE-g-MA showed lower toluene absorption than composites without PE-g-MA.

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## INTRODUCTION

Blending of thermoplastic and elastomer has evoked technological interest for use as a thermoplastic elastomer (TPE). A TPE can have properties of thermoplastic material such as high mechanical properties, high ozone resistance, and high ageing resistance (Ismail *et al.* 2004). In addition, a TPE can maintain elastomeric properties such as elasticity, high oil resistance, and low gas permeability (Ismail *et al.* 2004). A TPE offers attractive alternatives in a wide range of applications due to its unique properties and processing methods.

One of the most popular thermoplastic elastomer blends that have been recently used is poly(vinyl chloride) (PVC)/epoxidized natural rubber (ENR). Epoxidized natural rubber has been found to be mutually miscible with PVC. The PVC/ENR blend showed synergism in mechanical and dynamic mechanical properties as well as positive deviation from the rules of mixtures (Mathai and Thomas 2005). The miscibility of the PVC/ENR blend is expected to impart high tensile strength and good chemical resistance, whereas ENR itself can act as a permanent plasticizer for PVC (Mathai and Thomas 2005). In addition, ENR induces good tear strength and enhances the resistance against hydrocarbon oil.

Recently, the use of natural fiber has attracted more attention due to its renewable availability, low cost, light weight, and the fact that it is an environmentally friendly product (Rozman *et al.* 2001; Taib *et al.* 2010). Among natural fibers, kenaf fiber offers a potential for specific applications because of its low density, high specific mechanical

properties, and the ability to absorb nitrogen and phosphorous from the soil (Zampaloni *et al.* 2007). In this research article, the use of kenaf core powder in the blending of PVC/ENR blend has been studied. The incorporation of natural fiber into the PVC/ENR may reduce the consumption of plastic and rubber material. Furthermore, it can maintain the mechanical properties of the composites.

The different polarities of the PVC/ENR matrix and kenaf powder are the primary concern in developing the composite. It is essential to enhance the compatibility between a polar matrix and a natural non-polar material such as kenaf powder. To improve the mechanical properties of the composites, it is important to prepare a proper morphology and good interfacial adhesion using a compatibilizing agent. A major compatibilizer that has been used widely is polyethylene grafted maleic anhydride (PE-g-MA), which can increase the dipolar interaction between kenaf and matrix. In addition, PE-g-MA is more suitable to be used compared with PP-g-MA due to the processing temperature. In this study, the mechanical, morphological, and swelling properties of TPE composites, with and without polyethylene-grafted-maleic anhydride, were studied.

## EXPERIMENTAL

### Materials

Epoxidized natural rubber (Epoxyrene 50) with a 50% epoxidization level and Poly(vinyl chloride) (PVC) with a K value of 66, $M_n$ = 66 000; grade MH66, 6519 was purchased from Industrial Resin (M) Ltd. Johor Bahru, Malaysia. Polyethylene-grafted-maleic anhydride was purchased from Sigma Aldrich, Steinheim Germany. Diocthyl-phthalate (DOP) and barium/cadmium stearate were supplied by Bayer (M) Sdn. Bhd. The kenaf core powder was supplied by the Forest Research Institute of Malaysia (FRIM), Kepong Malaysia.

### Methods

*Compounding process of the composites*

The PVC was initially pre-mixed with a stabilizer (Cd/Ba stearate) and plasticizer (DOP) for 10 min at room temperature before compounding. The amounts of DOP and Cd/Ba stearate use for mixing of the composites were 50 parts per hundred resin (phr) and 3 phr, respectively.

The composition of PVC and ENR was fixed to a ratio of 70/30, and the PE-g-MA was fixed at 4 phf (part per hundred filler). The PE-g-MA used was fixed at 4 phr as accordance to previous study (Ismail *et al*. 2004). The amount of kenaf fiber was varied from 0 phr to 20 phr.

Table 1 shows the complete formulation for the composites. The PVC and ENR blends were prepared by melt mixing in a Haake Rheomix Polydrive R 600/610 (Thermo Electron, Osterode am Harz, Germany), at 140 °C and a rotor speed of 50 rpm, based on the total plastic content of the total composition. The ENR was placed in the mixing chamber for 1 min, followed by PVC with stabilizer and plasticizer that was allowed to equilibrate for 3 min. The kenaf core powder was then added after 4 min, followed by PE-g-MA. Mixing was then continued until a constant torque was obtained.

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**Table 1.** Formulation of PVC/ENR/Kenaf Core Powder without PE-g-MA

| Composition | Composites Formulation | | | | |
|:---:|:---:|:---:|:---:|:---:|:---:|
| | 1 | 2 | 3 | 4 | 5 |
| PVC | 70 | 70 | 70 | 70 | 70 |
| ENR | 30 | 30 | 30 | 30 | 30 |
| Cd/Ba Stearat (stabilizer)[a] | 3 | 3 | 3 | 3 | 3 |
| DOP (Plasticizer)[b] | 50 | 50 | 50 | 50 | 50 |
| PE-g-MA[c] | 0 | 4 | 4 | 4 | 4 |
| Kenaf Powder | 0 | 5 | 10 | 15 | 20 |

*a and b at part per hundred of resin (PVC)
*c at part per hundred of filler (kenaf)

*Compression moulding*

The PVC/ENR blends, PVC/ENR/kenaf core powder composites without PE-g-MA, and PVC/ENR/kenaf core powder composites with PE-g-MA were moulded in an electrically heated hydraulic press. Compression moulding involved pre-heating the press to 150 °C for 5 min and compressing for 3 min.

*Tensile test*

Tensile tests were carried out with a testometer M 500 (USA), according to ISO 37 (2011). Dumbbell-shaped specimens with a thickness of 1 mm were cut from the moulded sheets with a die cutter, Wallace S6/1/4 (MS Instrument (SEA) Sdn. Bhd., Penang, Malaysia. Five specimens were used in each case, and the mean value was recorded. The standard deviation was calculated to show the variation of each particular sample using the error bar. Reading of tensile strength, Young's modulus, and elongation at break ($E_b$) were recorded directly from the digital displays at the end of each test.

*Scanning electron microscopy*

Studies on the tensile fractured morphology of PVC/ENR/kenaf core powder composites were carried out using a Zeiss SUPRA36VP field emission scanning electron microscope (Oberkochen, Germany) operating at an accelerating voltage of 3kV. The samples (with surface exposed) were mounted on aluminum stubs with double-sided tape and sputter-coated with a thin layer (~20 NM) of gold to avoid charging and poor resolution during examination.

*Dynamic mechanical analysis*

The dynamic mechanical properties were evaluated using a dynamic mechanical analyzer (Perkin Elmer DMA7, Waltham, USA). The samples were subjected to a cyclic tensile strain with a force amplitude of 0.1 N, at a frequency of 10 Hz. The storage modulus and mechanical loss factor (tan $\delta$) were determined in the temperature range from -100 °C to 60 °C at a heating rate of 2 °C/min.

*Swelling test*

Determination of the swelling percentage of the composites was carried out in general accordance with ISO 1817 (2011). The test samples with the dimensions 30 mm x 5 mm x 1.5 mm were oven-dried at 50 °C for 24 h, and weighed using an electrical balance; this was considered to be the actual weight. The test pieces were immersed in toluene at room temperature for 24 h. After removal from toluene, the samples were wiped with tissue

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paper to remove excess toluene from the surface, and immediately weighed (swollen weight). The swelling index ($W_t$) was calculated according to Eq. 1,

$$W_t = \frac{(W_2 - W_1)}{W_1}$$ (1)

where $W_1$ and $W_2$ were the sample dry weight and the weight of the samples after exposure time $t$, respectively.

## RESULTS AND DISCUSSION

### Compounding Torque

The effect of kenaf core powder on the compounding torque properties of PVC/ENR/kenaf core powder composites without PE-g-MA is shown in Fig. 1a. After the ENR had been charged in the chamber, the compounding torque for all compounds stabilized at 2 N-m to 4 N-m. After 1 min, the PVC was charged in the chamber, and the torque value started to rise. The trend was the same for all compounds until the kenaf core powder was added at 4 min. The torque values, then increased because of the additional energy required following the addition of the kenaf core powder due to the increase in viscosity of the compound. Figure 1b. shows the compounding torque for PVC/ENR/kenaf core powder composites with PE-g-MA which shows the same trend as Fig. 1a.

The stabilization torque indicated that the torque value, and thus the viscosity, increased with increasing kenaf core powder loading. Stabilization torque at the end of each compounding can be seen in Fig. 2. The composite with PE-g-MA shows a higher stabilization torque compared to the composite without PE-g-MA. The increase in torque with the addition of PE-g-MA is due to the reaction between the chemically reactive groups of compatibilizer with the matrix and kenaf powder that produce branched chains and cross linking (Majid *et al.* 2010). The reaction produces branched chains and cross-linked macromolecules that have a higher melt viscosity compared to linear macromolecules. Because of this reaction, the stabilization torque of the compatibilized composite is higher than that of uncompatibilized composites.

Figure 3 shows the esterification reaction between kenaf and PE-g-MA that may occur during compounding. The enhanced interfacial adhesion between kenaf core and PVC/ENR matrix with the addition of PE-g-MA can be explained by the nature of the chemical structure between the functional group of PE-g-MA and the hydroxyl group of kenaf core fiber, combined with the diffusion of the polyolefin backbone of the coupling agent into PVC/ENR phase during processing. Furthermore, it is believed that PVC and ENR will form a self- cross-linking during compounding at a sufficient temperature above 140 °C even in the absence of any curing agent (Ramesh and De 1991).

Evidence of a self-cross-linking reaction between PVC and ENR can be seen in Fig. 4. The miscibility between PVC and ENR has been  proven by a single $T_g$ curve plotted in the DMA analysis result. This possible interaction explained how the poly olefin backbone of PE-g-MA can react with the PVC/ENR matrix as shown in the proposed chemical interaction in Fig. 5.
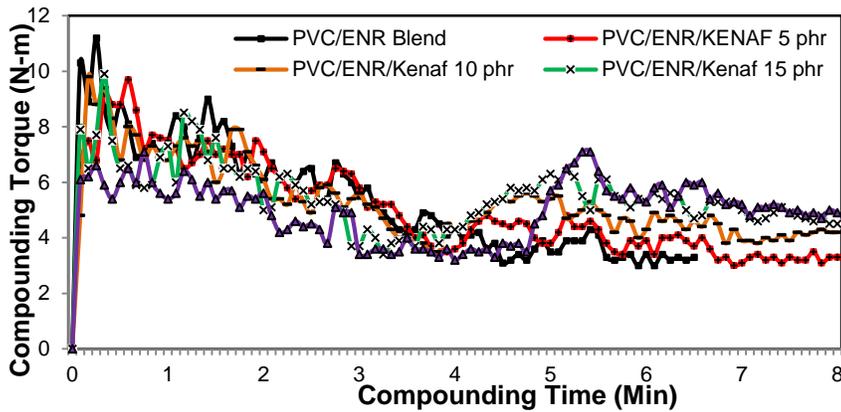
**Fig. 1a.** Processing torque for PVC/ENR/kenaf core powder composites without PE-g-MA for individual data
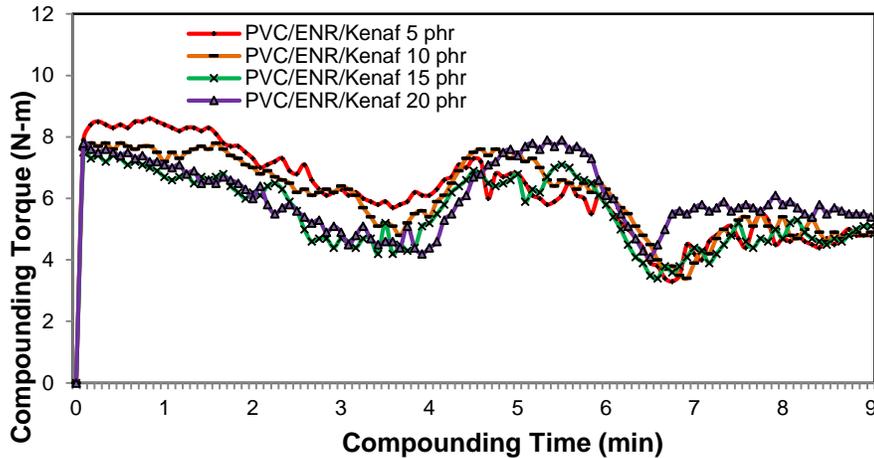


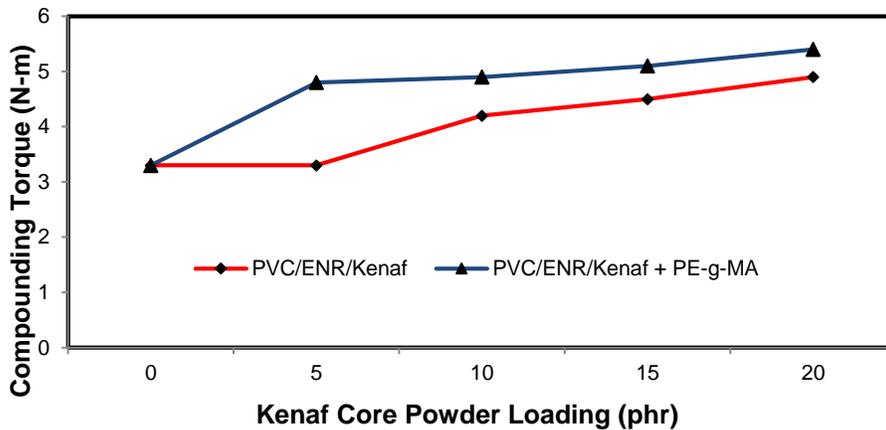**Fig. 1b.** Processing torque for PVC/ENR/kenaf core powder composites with PE-g-MA for individual data



**Fig. 2.** Stabilization torque for PVC/ENR/kenaf core powder composites with and without PE-g-MA for individual data
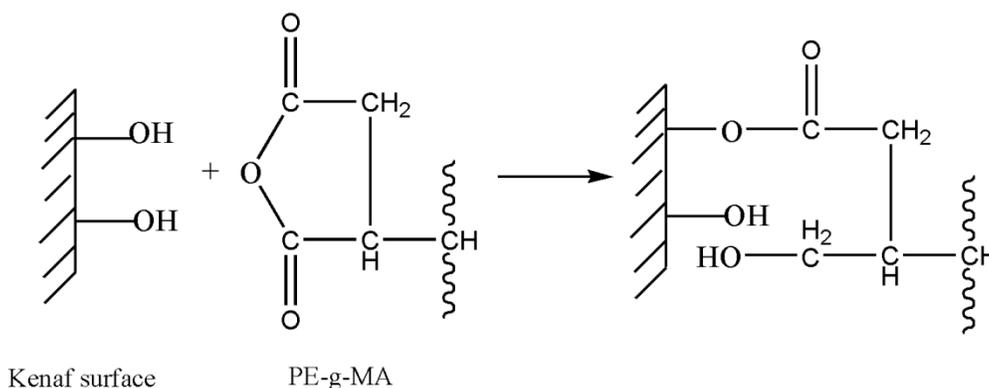
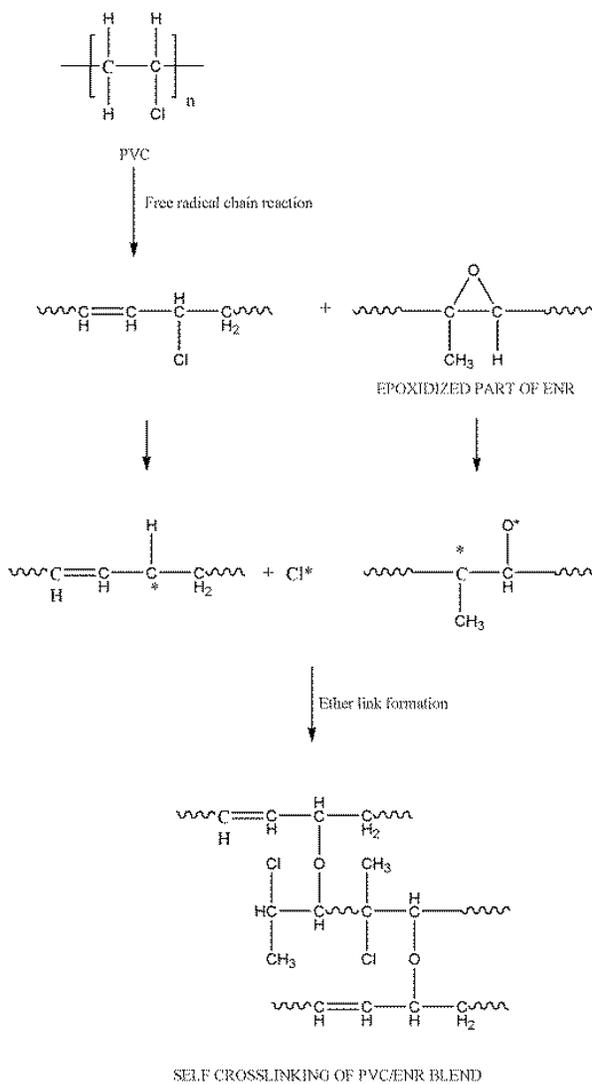**Fig. 3.** Esterification reaction between kenaf core powder and PE-g-MA



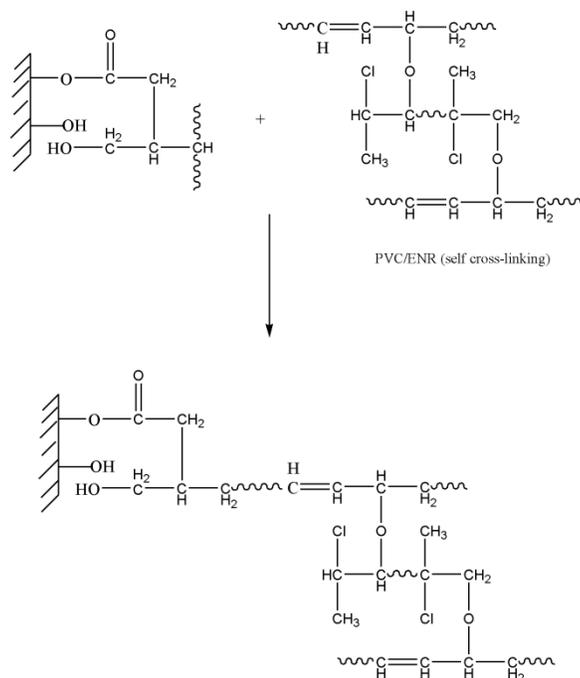**Fig. 4.** Self-cross-linking interaction between PVC and ENR

**Fig. 5.** Proposed chemical interaction between kenaf + PE-g-MA with PVC/ENR matrix (after self-cross-linking)

## Tensile Properties

The tensile properties of the PVC/ENR/kenaf core powder composites with and without the addition of PE-g-MA can be seen in Figs. 6 to 8. The incorporation of kenaf core powder reduced the tensile strength of the composites of the PVC/ENR/kenaf core powder composite without PE-g-MA, as shown in Fig. 6. The decrease in the tensile strength values with increasing kenaf core powder loading is attributable to the poor wetting of the fiber with the matrix of the composites. The PVC and ENR matrix is a hydrophobic material, whereas the kenaf core powder is a hydrophilic material. Because of the difference in polarity, the kenaf core powder has poor wettability. Poor wetting reduced the interfacial adhesion between the kenaf core powder and the matrix, which increased the stress concentration area (Ismail *et al.* 1999). The stress concentration area inside the composites initiated micro-cracks, resulting in low tensile strength. With increasing kenaf core powder loading, the kenaf core powder particles are no longer equally separated or wetted by the polymer matrix. Thus, the continuous decrease in tensile strength may be due to the agglomeration of the filler particles to create a domain that acts like a foreign body. However, for the PVC/ENR/kenaf composite with PE-g-MA, the tensile strength increases up to 10 phr of kenaf core, the optimum tensile strength value. The optimum tensile strength demonstrated that an improvement of interfacial adhesion occurred due to better interfacial adhesion, which can reduce the stress concentration area and kenaf powder agglomeration. As seen in Fig. 6, the results for kenaf powder loadings of 5 to 20 phr show that the incorporation of PE-g-MA increased the tensile strength of the PVC/ENR/kenaf composites compared to similar composites without PE-g-MA (Ismail *et al.* 1999; Pasbakhsh *et al.* 2009; Siriwardena *et al.* 2002).

Figure 7 shows the elongation at break of PVC/ENR/kenaf core powder composites. The elongation at break was reduced drastically with increasing kenaf core powder loading.

This effect is due to the decrease in deformability of the rigid interphase between the kenaf core powder and PVC/ENR matrix. With the addition of PE-g-MA, the elongation at break of the composites increased slightly. In composites with the addition of PE-g-MA, better filler dispersion and good adhesion between filler and matrix were obtained, which enhance the stress transfer from the matrix to the filler phase. Unfortunately, at higher filler loading, the stress propagation from the matrix to the filler was obstructed because of the irregular structure of the filler.
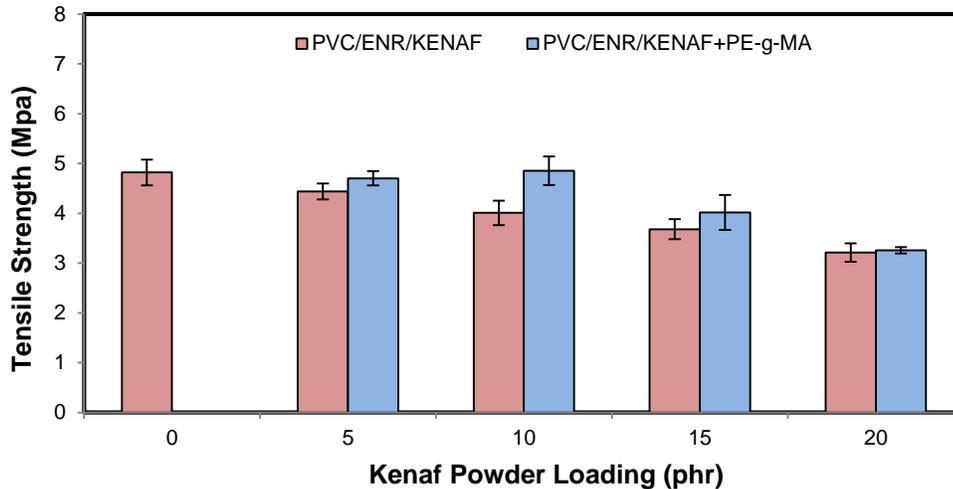


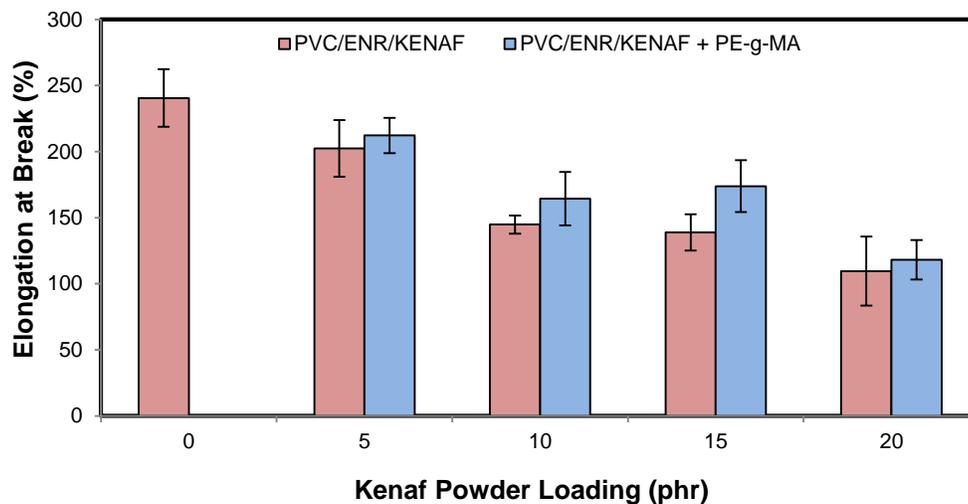**Fig. 6.** Tensile strength for PVC/ENR/kenaf core powder composites with and without PE-g-MA



**Fig. 7.** Elongation at break for PVC/ENR/kenaf core powder composites with and without PE-g-MA

The effect of kenaf core powder on the Young's modulus of PVC/ENR/kenaf core powder composites is seen in Fig. 8. Young's modulus showed a slight increase with increasing kenaf core powder loading. The incorporation of kenaf core powder into the matrix of PVC/ENR increased the stiffness of the composites resulting from the inclusion of rigid filler particles in the soft matrix. PVC/ENR/kenaf core powder composites with the addition of PE-g-MA showed lower Young's modulus values than composites without

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PE-g-MA. The PE-g-MA may act as a toughness modifier to absorb the energy in the composites, which lowers the Young's modulus results for PVC/ENR/kenaf core powder composites with the addition of PE-g-MA. Theoretically, a toughness modifier that is in rubbery phases facilitates the yielding of the matrix before failure when a sudden or continuous load is applied. This is because the dispersed rubbery phases maintain their particle morphology in the continuous PVC/ENR phase. The addition of PE-g-MA might improve the ductility of the composites with corresponding decrease in Young's modulus and the increase in elongation at break (EB). The tailoring of toughness modified composites has been always a trade-off between rigidity and ductility.
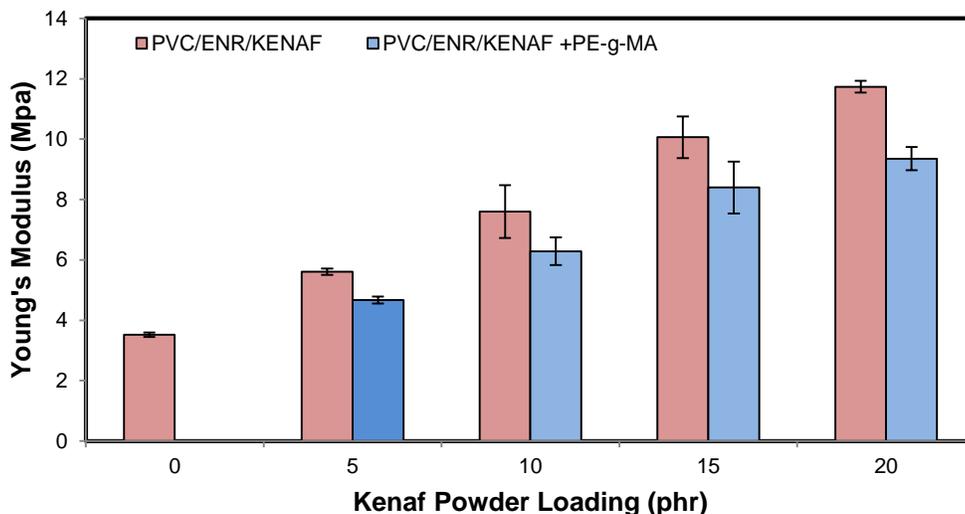


**Fig. 8.** Young's modulus for PVC/ENR/Kenaf core powder composites with and without PE-g-MA

## Morphological Properties

Figure 9 shows the fractured surface morphology of PVC/ENR/kenaf composites with a kenaf core powder loading of 5 phr with and without PE-g-MA. Three samples were analyzed for each PVC/ENR/kenaf composite with 5 phr kenaf core loading, with and without PE-g-MA. Figure 9a shows a fiber pulled from the matrix due to the low amount of kenaf core powder in the composite. Furthermore, the gap between fiber and matrix where no matrix covers the surface of the fiber is very clear. Compared to the PVC/ENR/kenaf composite with the addition of PE-g-MA, there were fewer fibers that were pulled out, with more fibers embedded in the matrix. In addition, the surface of the kenaf powder was covered with the matrix. This micrograph proves that there is interfacial adhesion between the kenaf core powder and matrix for PVC/ENR/kenaf composites with the addition of PE-g-MA.

The fractured surface morphology of the PVC/ENR/kenaf composite with a kenaf core powder loading of 20 phr is shown in Fig. 10. Pulled fibers are readily seen in the PVC/ENR/kenaf core powder composite without PE-g-MA (Fig. 10a). The dispersion of the kenaf core powder was poor, which led to higher agglomeration. Figure 10b is a micrograph of the PVC/ENR/kenaf core powder composites with PE-g-MA. Better kenaf dispersion can be seen, which reduces the size of agglomeration and causes fewer fibers to be pulled out. In addition, the kenaf surface is covered with the matrix. This result makes it clear that chemical interaction had occurred between the kenaf and matrix improved the adhesion. This morphology analysis also supports the tensile strength results, in which

greater strength was obtained by the composites with PE-g-MA. Furthermore, the higher tensile strength was obtained for the PVC/ENR/kenaf core powder composites with PE-g-MA due to higher stress transfer, which reduced the sudden failure of the composite (Nasir *et al.* 2003; Rozman *et al.* 2012).
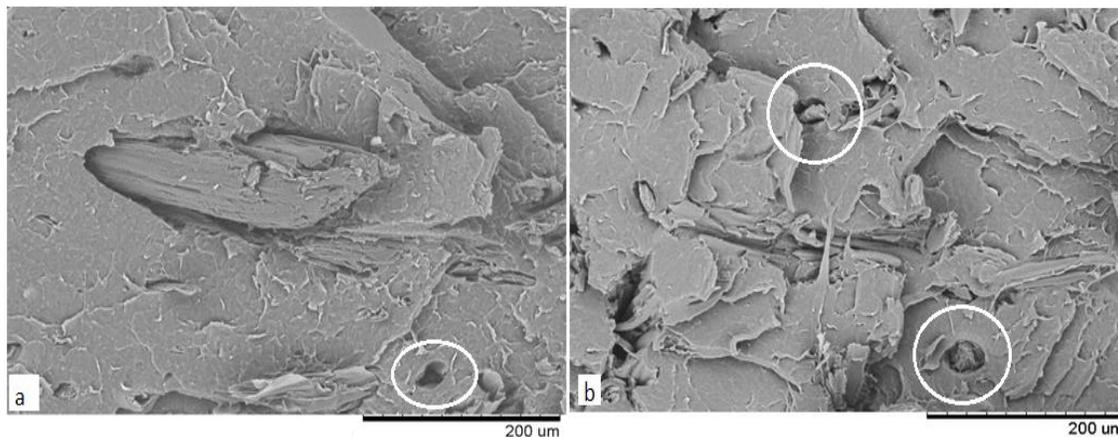


**Fig. 9.** Scanning electron micrographs of PVC/ENR/kenaf core powder composites at 5 phr: (a) without PE-g-MA (circle denotes a cavity left by a fiber), (b) with PE-g-MA (circles denote a cavity containing a broken fiber)
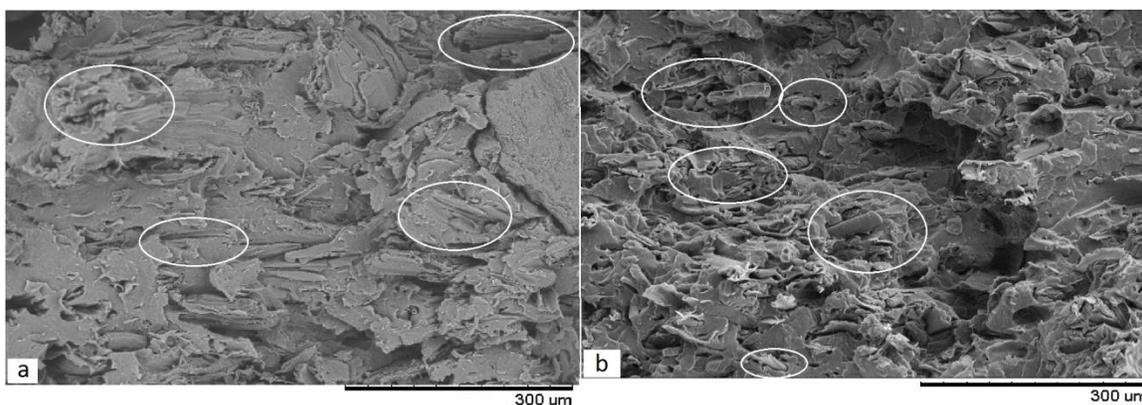


**Fig. 10.** Scanning electron micrographs of PVC/ENR/kenaf core powder composites at 20 phr: (a) without PE-g-MA (circle denotes an area of fiber agglomeration and fiber pulled out), (b) with PE-g-MA (circle denote areas of good fiber good adhesion with matrix)

## Dynamic Mechanical Analysis

Figure 11 shows the damping factor (tan $\delta$) as a function of temperature from DMA analysis to confirm the glass transition temperature, $T_g$ values for the PVC/ENR/kenaf core powder composites without the addition of PE-g-MA. The $T_g$ of the composites can be calculated from the tan $\delta_{max}$ of the graph. Figure 11 shows that the $T_g$ value increased with the increase in kenaf core powder loading up to 10 phr kenaf core powder loading and afterwards decreased. This dependence was similar in both PVC/ENR/kenaf core powder composites with the addition of PE-g-MA. The comparison of $T_g$ values for the composites with and without the addition of PE-g-MA can be seen in Table 2. These results can be explained by the co-existence of the two mechanisms; interfacial constraints and free volume increase (Bershtein *et al.* 2002). According to interfacial constraints mechanism, a short range, highly immorbilized layer is developed near the surface of the fillers. In this

interaction region of the polymer layer surrounding the particles, the conformation entropy and the chains kinetics are significantly altered.  As the filler content increases, the volume fraction of the interaction region increases too. The polymer chains in the region are under constraint because of the interfacial polymer-particle interaction, and therefore the $T_g$ of the composites have been shifted to higher temperature. Base on the concept of free volume, the increase of the filler content increases the free volume due to loosened molecular packing of the chains. This extra created free volume assists the large-scale segmental motion of the polymer. As a result $T_g$ of the composites decreases as the filler loading increases.

Table 2 shows that a higher $T_g$ value is obtained for PVC/ENR/kenaf core powder composites with and without the addition of the PE-g-MA for all kenaf core powder loading. Based on the concept of free volume, the addition of PE-g-MA decreases the free volume at the interface and improved the compatibility, and therefore slightly increased the $T_g$.
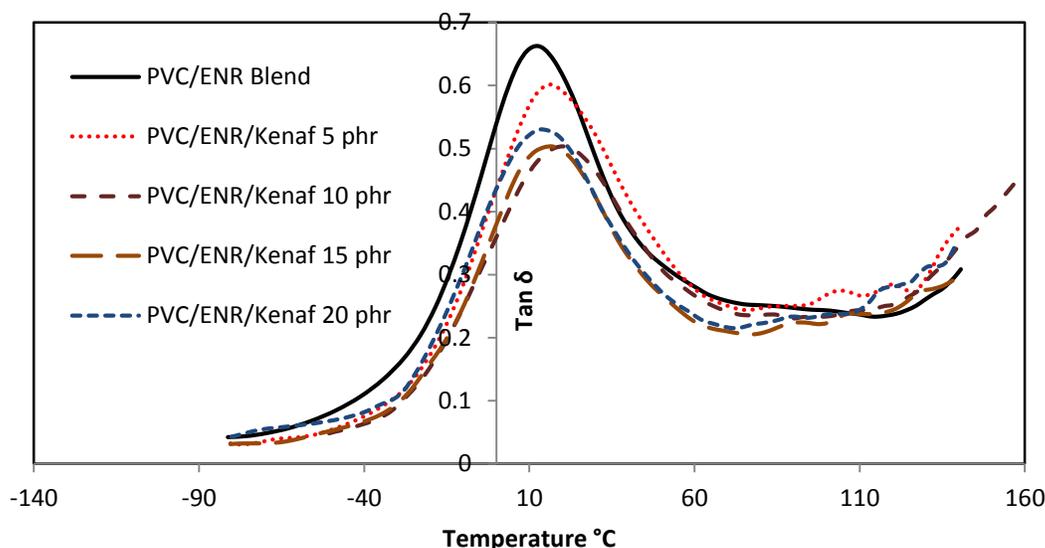


**Fig. 11.** Tan δ  for PVC/ENR/kenaf core powder composites without PE-g-MA as a function of temperature

**Table 2.** Effect of Kenaf Core Powder Loading and PE-g-MA on the Tan $\delta_{max}$ and the Glass Transition Temperature of PVC/ENR/Kenaf Core Powder Composites

| Sample codes | Tan $\delta_{max}$ | | $T_g$ (°C) with respect to Tan $\delta_{max}$ | |
| --- | --- | --- | --- | --- |
| | Composites without PE-g-MA | Composites with PE-g-MA | Composites without PE-g-MA | Composites with PE-g-M |
| PVC/ENR blend | 0.66 | | 12.09 | |
| PVC/ENR/Kenaf 5 phr | 0.6 | 0.66 | 16.61 | 22.10 |
| PVC/ENR/Kenaf 10 phr | 0.5 | 0.55 | 20.11 | 22.31 |
| PVC/ENR/Kenaf 15 phr | 0.5 | 0.5 | 16.19 | 20.15 |
| PVC/ENR/Kenaf 20 phr | 0.53 | 0.52 | 13.81 | 19.74 |

## Swelling Properties

Swelling tests were conducted for 24 h using toluene. According to Fig. 12, the swelling index decreased with the incorporation of PE-g-MA. Better interfacial adhesion that appeared in the composites with PE-g-MA reduced the ability of the solution to penetrate the composite. The diffusion mechanism in rubber is associated with the ability of the polymer to provide pathways for the solvent to progress in the randomly generated voids that are formed (Ismail *et al.* 2001; 2005; Jacob *et al.* 2004; Nabil *et al.* 2013). In this study, it has been clearly shown that the cross-linking reduced the pathway for the toluene solution. In addition, with increasing kenaf core powder loading, the amount of matrix was reduced and the composite became stiffer and less penetrable by the toluene.
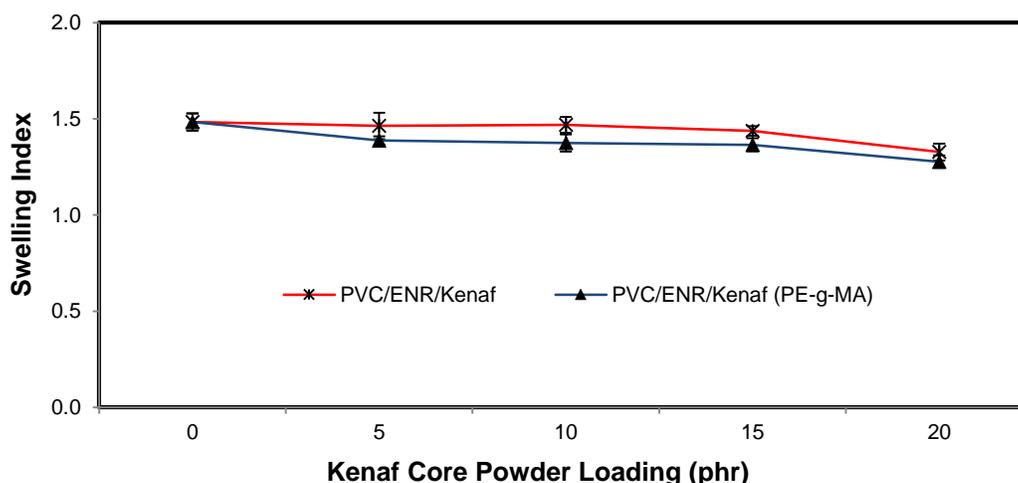


**Fig. 12.** Swelling index for PVC/ENR/kenaf core powder composites with and without PE-g-MA

## CONCLUSION

1. The incorporation of kenaf core powder in PVC/ENR/kenaf core powder composites reduced the tensile strength and elongation at break of the composites due to poorly dispersed filler in the matrix and poor compatibility.

2. With the incorporation of PE-g-MA, the tensile properties improved due to better interfacial adhesion between the kenaf and the matrix, which improved the stress transfer.

3. Young's modulus increased with increasing kenaf core powder loading due to the increase in the stiffness of the composites.

4. Results from the SEM micrographs demonstrated better dispersion of kenaf core powder and less fiber pulled out after the addition of PE-g-MA.

5. DMA analysis indicated that the $T_g$ for PVC/ENR/kenaf with the addition of PE-g-MA was higher than that of PVC/ENR/kenaf without PE-g-MA.

6. The swelling index indicated that the incorporation of the PE-g-MA in the composite reduced the swelling properties of the composite.

## ACKNOWLEDGMENTS

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