

STUDIES ON REACTIVE COMPATIBILIZATION AND DYNAMIC VULCANIZATION OF POLYPROPYLENE/EPOXIDIZED NATURAL RUBBER BLENDS FILLED WITH CARBONIZED DIKA NUTSHELL

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ABSTRACT

Carbonized Dika nutshell filled – polypropylene (PP)/epoxidized natural rubber (ENR) blend was prepared and its morphological, rheological and mechanical properties were studied. A combination of maleic anhydride-modified PP (PP-g-MA) and poly (lactic acid) (PLA) was used as compatibilizer, which act as interfacial agents between PP and ENR phases. The effectiveness of compatibilization was suggested from viscosity and storage modulus, determined by rheological measurements. Compatibilization produced increases in complex viscosity, storage modulus and impact strength. Dynamic vulcanization also resulted in increases in these properties, which were higher than those observed for non-compatibilized/non-vulcanized blends. The increase in complex viscosity, storage modulus and impact strength with compatibilization and vulcanization is due to molecular build-up arising from chemical reactions that occurred during the melt blending process, as well as increase in network formation caused by vulcanization. Outstanding mechanical performance, especially tensile strength and impact strength (higher values), and elongation at break (lower values) were obtained with increase in filler content.

Keywords: reactive compatibilization, dynamic vulcanization, carbonized dika nutshell, polypropylene, epoxidized natural rubber

INTRODUCTION

Over the years, elastomer-thermoplastic blends have become technologically useful as thermoplastic elastomers (Morris, 1979; Kresge, 1978; Kresge, 1984). They exhibit many of the properties of elastomers but are process able as thermoplastics (O'Connor and Faith, 1982). Thus, they offer a substantial economic advantage with respect to the fabrication of finished products. For many applications, an essential requirement of elastomer-thermoplastic blends with good mechanical properties is that the elastomer component be finely dispersed in a relatively small amount of the thermoplastic. The elastomer particles should also be cross linked to promote elasticity. Again, the favourable morphology should remain during the fabrication of the blend into finished parts and in use. Because of these essential requirements, the usual methods of preparing elastomer-thermoplastic blends by melt mixing, solution blending or latex mixing are not sufficient (Coran and Patel, 1980).

The best way to produce thermoplastic elastomers comprising vulcanized elastomer particles in melt process able thermoplastic matrices is the method called dynamic vulcanization. It is the process of vulcanizing the elastomer phase using a suitable vulcanizing agent, during its melt mixing with the molten thermoplastic giving rise to a thermoplastic vulcanizate (TPV) (Margolis, 2004). Property improvements obtained from dynamic vulcanization include reduced permanent set, improved mechanical properties, greater resistance to attack by fluids, higher temperature characteristics, greater stability of phase morphology, and more reliable thermoplastic fabricatability.

Polypropylene (PP) is a commodity crystalline thermoplastic polymer with excellent properties, such as electric insulation with extremely low dielectric loss, and chemical resistance, and therefore has wide applications in the electronic and textile industries. However, the impact strength of PP is poor, especially at low temperatures, and at 0°C the polymer is virtually brittle. Another major disadvantage of PP is the susceptibility of its methyl groups to thermo-oxidative degradation (Joel, 2005). For these

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reasons, the applications of PP have been limited. While antioxidant is a necessary component in all PP compounds, in view of the susceptibility of PP to thermo-oxidative degradation, significant improvement in impact strength can be achieved by blending with elastomers, so that the elastomer particles act as effective stress dissipating agents (Cimmino *et al.*, 1985; Coppola *et al.*, 1986). The impact modification of thermoplastics has been an important area of polymer blend technology for many years, and continues to be a challenging field of research. Commercial products which have proven to be important include high-impact polystyrene, rubber-modified styrene/acrylonitrile copolymers, toughened poly (vinyl chloride), and elastomer-modified polyamides (Bucknall, 1977; Keskkula, 1982). These blends contain between 6 and 12 percent rubber and have notched Izod impact strengths ranging from 32 to 192 Nm/m (Ku, 1988).

Polymers from renewable resources have attracted an increasing amount of attention over the last two decades, predominantly due to two major reasons: firstly environmental concerns, and secondly the realization that our petroleum resources are finite. The development of synthetic polymers using monomers from natural resources provides a new direction to develop biodegradable polymers from renewable sources. One of the most promising polymers in this regard is poly(lactic acid) (PLA), because it is made from agricultural products and is readily biodegradable. PLA is hydrophilic, and its hydrophilic character increases degradability. However, its moisture sensitivity, poor gas barrier, brittleness and high cost limit its application (Long *et al.*, 2006). Yet, PLA can be conveniently utilized in relatively small amount as a compatibilizer in polymer blends. In PP/ENR blends, the presence of carboxyl group in PLA backbone provides an effective anchorage between the phases through the reaction between the carboxyl groups of PLA and epoxide groups of ENR.

Prevailing economic realities in developing countries like Nigeria with respect to increasing debt burden and low Gross Domestic Product (GDP), urgently call for reduction in import dependence and the encouragement of locally sourced raw materials. Bulk of the particulate fillers, such as carbon black, calcium carbonate, mica, etc, used as reinforcing or cost-reducing additives in polymer compounding operations in Nigeria are imported, and huge amount of foreign exchange can be saved if some of these materials can be sourced locally. Thus, the use of agricultural by-products to partly or wholly replace these additives has attracted significant attention in recent years, particularly for low-cost/high volume application (Onyeagoro, 2012). Nigeria is endowed with vast agricultural resources, which are largely under-utilized. Dika nutshell, like, many other agricultural by-products in Nigeria are currently being discarded as waste. However, these rejects can be harnessed as potential substitute fillers for imported particulate fillers in polymer products, thereby saving huge foreign exchange spent on importation of these particulate fillers.

Due to large difference in surface energy and polarity, blending PP with ENR should be phase separated. Yet, blend of the two is a good choice in application areas where excellent oil and impact resistance, gas barrier, good wet grip, and high damping characteristics are required. In this regard, a combination of maleic anhydride-grafted polypropylene (PP-G-MA) and poly (lactic acid), which acts as interfacial agents between PP and ENR phases, is considered to be a potential compatibilizer for this blending system.

Dynamic vulcanization technique has been employed in the development of several thermoplastic vulcanizates (TPV'S). Most of them are made using polypropylene matrix, such as nitrile rubber (Zhang *et al.*, 2002; Naderi *et al.*, 1999; George *et al.*, 1999; Coran and Patel, 1983; Soares *et al.*, 2006), ethylene-propylene-diene rubber (EPDM) (Gupta *et al.*, 2007; Chatterjee and Naskar, 2007; Manchado *et al.*, 2002) and natural rubber (Thitihammawong *et al.*, 2007), among others. However, to the best of our knowledge, there is no report in the accessible literature dealing with reactive compatibilization and dynamic vulcanization of carbonized Dika nutshell powder-filled PP/ENR blends using a combination of maleated polypropylene and poly (lactic acid) as a compatibilizer.

Therefore, the aim of the present study is to investigate the morphological, rheological and mechanical properties of dynamically vulcanized Dika nutshell powder-filled PP/ENR blends, using maleated polypropylene/poly (lactic acid) as compatibilizer. Because of the high incompatibility between PP and ENR, compatibilization is necessary in order to improve the mechanical performance of the blend for commercial applications.

MATERIALS AND METHOD

Materials

Polypropylene (density = 0.9g/cm³ and melt flow index = 3.5g/10min at 230⁰C/2.16kg), and maleic anhydride-graft-polypropylene (PP-g-MA) (Polybond 3200) (melt flow index = 115g/10min at 190⁰C/2.16kg; 1wt. % of maleic anhydride) were purchased from Rovet Chemicals Ltd, Benin City, Nigeria. Epoxidized natural rubber (density = 0.95g/cm³ and epoxide content of 33% was obtained from a chemical store in Lagos, Nigeria. Rubber grade additives used were zinc oxide (ZnO), stearic acid, and benzoyl peroxide (BPO).

Preparation of Carbonized Dika nutshell powder (C-DNS)

Dika nutshells were washed and dried in air to remove sand particles and moisture. The dried Dika nutshells were milled to fine powder and sieved through a mesh size of 150 μ m. The fine particles that passed through the mesh were collected and carbonized at 600⁰C for 3hours (Ishak and Baker, 1995). The carbonized C-DNS was kept in a desiccator and allowed to cool to room temperature until required.

Blend preparation and morphological features

Blends of PP and ENR were prepared by melt mixing, carried out in a Brabender Plasticorder model PLE 331. PP was first melted for 2min at 230⁰C followed by the addition of ENR. Mixing was continued for 6min. In dynamically vulcanized samples, the curatives and compatibilizer system were added 2min after the rubber (ENR) had been introduced, followed by the addition of C-DNS filler. Mixing was stopped after 10min, and the blend was cooled under N₂ atmosphere and then stored in drying oven at room temperature. Blend composition of PP/ENR was fixed at 50/50 by weight, while varying filler loadings of 0, 4, 8, 12, and 14 wt.% were used. The formulations used in preparing the non-vulcanized blends (NVB) and the vulcanized blends (VB) are given in Tables 1 and 2, respectively. The amounts of the curatives are based on the rubber phase only.

To compare the morphological features of each blend, the tensile fracture surfaces of the blends were examined using scanning electron microscopy (SEM, JEOL, and JSM – 6300 model). All blend samples were coated with a thin layer of gold-palladium to reduce any charge build-up on the fracture surface.

Table 1. Compounding recipe for non-vulcanized PP/ENR blends

Ingredients (by weight)	NVB1	NVB2	NVB3
PP	50	50	50
ENR	50	50	50
PP-g-MA	0	5	5
PLA	0	0.1	0.4
C-DNS	0, 4, 8, 12, 14		

Table 2. Compounding recipe for vulcanized PP/ENR blends

Ingredients (by weight)	VB1	VB2	VB3
PP	50	50	50
ENR	50	50	50
PP-g-MA	0	5.0	5.0
PLA	0	0.1	0.4
ZnO	0	5.0	5.0
Stearic acid	0	2.0	2.0
BPO	0	1.0	1.0
C-DNS	0, 2, 4, 6, 8,10		

Measurement of Rheological Properties

The rheological behaviour of the blends was carried out on a rheometric dynamic spectrometer using a parallel- plate geometry ($R = 25$ mm) (Utracki, 1988). The measurements were done at 230°C . For strain sweep measurements, a strain range of 0.1 to 300% and an angular frequency of 7.5 rad/s were used. The measurements with frequency sweep were done at a constant strain of 2.5% and a frequency range of 0.1 to 100 rad/s.

Tensile Test

Tensile properties were determined on a Monsanto Tensile Tester (Model 1/m) with a crosshead speed of 500mm/min using a dumb bell test pieces measuring 45mm x 5mm x 2mm according to ASTM D412-87 method A. The recorded value for each tensile parameter is the average of three measurements.

Measurement of Impact Strength

Impact strengths of the PP/ENR blends were measured by carrying Izod impact tests. All specimens for Izod impact test were stored in a dessicator until required for test to avoid moisture absorption. Izod bars were notched and tested at room temperature according to ASTM D256 method. The values of impact strengths of each blend sample are the averages of 5 runs.

RESULTS AND DISCUSSION

Blend Morphology

To confirm the effects of compatibilization and vulcanization on dispersed phase morphology, the morphologies of the non-vulcanized PP/ENR blend (NVB1) and the vulcanized PP/ENR blend (NVB2) were compared. Figure 1 shows the SEM micrographs of the tensile fracture surfaces of the non-vulcanized and vulcanized PP/ENR blends. For the non-vulcanized PP/ENR blend (Figure 1(a)), the ENR domains displayed large size distribution. Also, there is little adhesion between the phases, resulting in many pull-out of dispersed phase from the matrix. This is a typical morphology of incompatible polymer blend. For the non-vulcanized blends, the size of the dispersed phase is dominated preferentially by the interfacial tension between the blend components (Chen and White, 1993). Large interfacial tension between the two phases leads to large size of dispersed phase and poor interfacial adhesion. However, unlike non-vulcanized PP/ENR blends, vulcanized PP/ENR blends (Figure 1(b)) displayed different morphological features. As shown in Figure 1 (b) compatibilization and vulcanization resulted in considerable reduction in the large interfacial tension or increase in the interfacial adhesion.

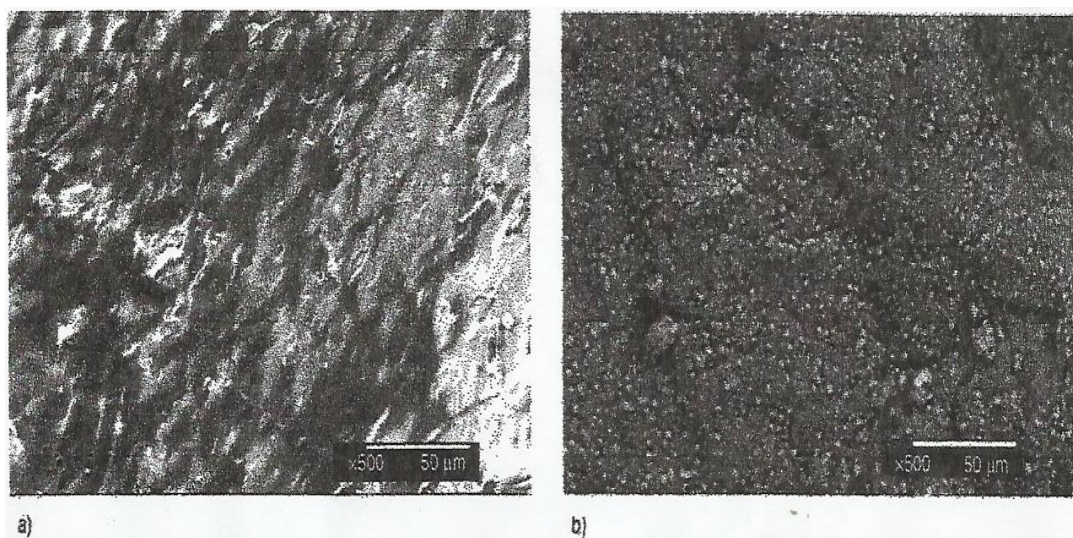


Figure 1. SEM micrographs of PP/ENR (50:50 wt%) blends: (a) non- vulcanized and non-compatible; (b) vulcanized and compatibilized with PP-g-MA/PLA (5.0/0.1).

Rheological Properties

In a study by Ayo et al, 2011 on the effect of filler carbonization temperature on mechanical properties of natural rubber/groundnut shell filler composites, optimal mechanical properties were reported at 600°C filler carbonization temperature. In the present study, the rheological properties of vulcanized and non-vulcanized PP/ENR blends filled with carbonized Dika nut shell obtained at 600°C carbonization temperature were investigated. In both cases, the filler content was kept constant at 10 phr. Plots of complex viscosity, η , versus angular frequency, ω , for the vulcanized and non-vulcanized PP/ENR blends are presented in Figure 1. In the case of non-vulcanized blends (Figure 1 (a)), the viscosity of the compatibilized blends (NVB3 and NVB2) is higher than that of the non-compatibilized blend (NVB1). The same trend is shown by the vulcanized blends (Figure 1 (b)). It has been reported that increase in complex viscosity of a polymer blend generally occur when there is either a specific interaction between the phases (Han, 1976 ; Han, 1981) or chemical bonding between the blend composition (Utracki, 1988). Such a chemical bonding will also induce a strong interaction between the phases. Consequently, the matrix will exhibit greater resistance to flow, resulting in a high viscosity. On the other hand, when there is no interaction or chemical bonding between the phases, low viscosity results because the domain can easily be elongated in the matrix. Thus, the higher complex viscosity of the compatibilized PP/ENR blends over non-compatibilized blends is due to reactive compatibilization arising from chemical reaction between the acid (-COOH) groups of PP-G-MA and the epoxide groups of ENR during melt blending. For both non-vulcanized and vulcanized blends, the complex viscosity and storage modulus of blend containing 0.4 wt% PLA (NVB3 and VB3) were lower than one containing 0.1 wt% PLA (NVB2 and VB2), respectively because of increased lubricating effect of PLA with increase in PLA content.

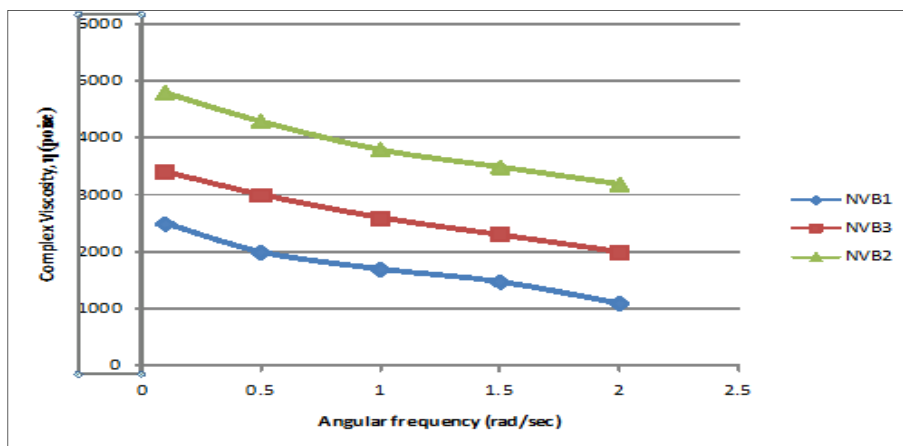


Figure 1(a). Plot of Complex Viscosity versus Angular frequency for non-vulcanized PP/ENR blends

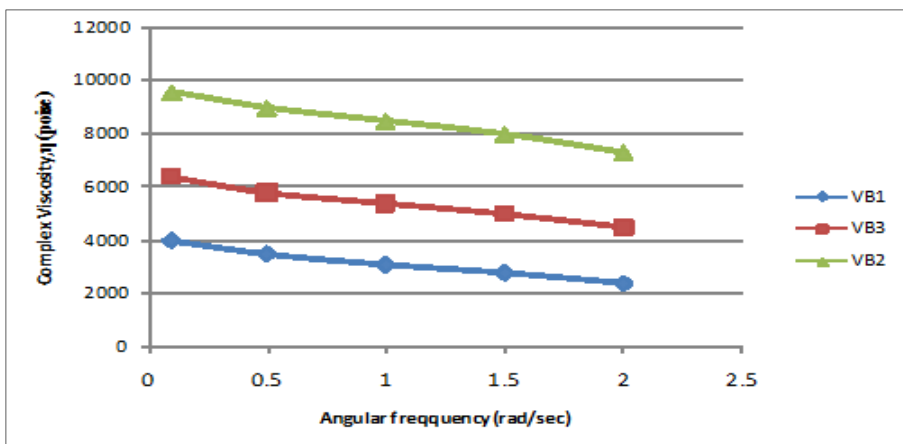


Figure 1 (b). Plot of Complex Viscosity versus Angular frequency for vulcanized PP/ENR blends

Like the complex viscosity, the compatibilized blends (Figure 1 (b)) showed a higher increase in storage modulus, G , than non-compatibilized blends due to molecular build-up resulting from chemical reactions that occurred during the melt blending process (Bagrodia et al, 1986).

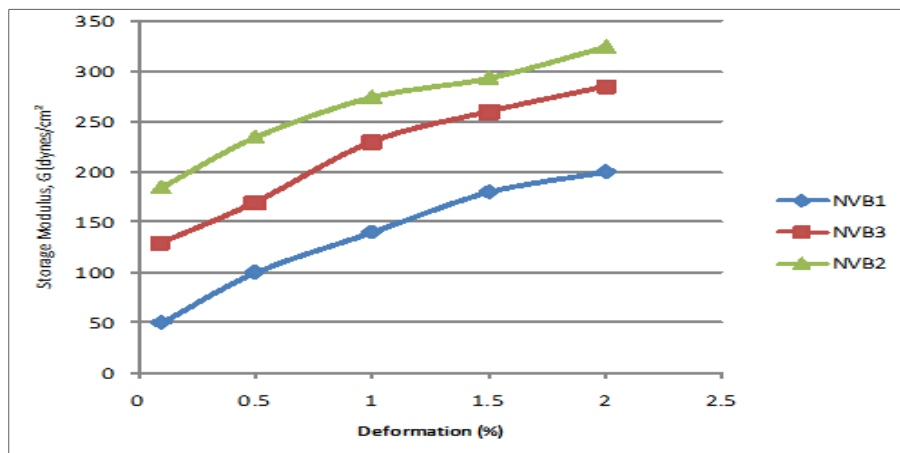


Figure 2 (a). Plot of Storage Modulus versus Deformation for non-vulcanized PP/ENR blends

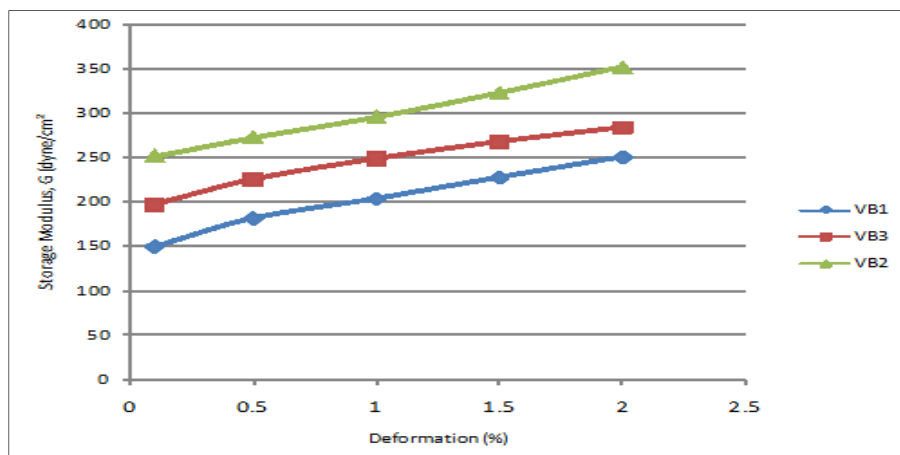


Figure 2 (b). Plot of Storage Modulus versus Deformation for Vulcanized PP/ENR blends

Tensile Properties

The effects of filler loading on mechanical properties of re-inforced elastomers have been extensively reported in the literature (Findik et al, 2004). In a recent study (Onyeagoro, 2012) on the influence of carbonized Dika nutshell (C-DNS) filler loading on vulcanizate properties of natural rubber/acrylonitrile-butadiene rubber blend, optimal vulcanizate properties were reported at 10 phr filler loading. In the present study, non-vulcanized and vulcanized blends of PP/ENR filled with C-DNS were prepared. The tensile properties of the blends as a function of filler loading were investigated and the results are presented in Figure 3 and Tables 3 and 4. As shown in Figures 3 (a) and (b) tensile strength increased with filler loading for both non-vulcanized and vulcanized blends. The vulcanized blends displayed higher tensile strength than non-vulcanized blends. In the case of non-vulcanized blends (Figure 3 (a)), the tensile strength of the compatibilized blends (NVB3 and NVB2) is higher than that of the non-compatibilized blend (NVB1). The same trend is shown by the vulcanized blends (Figure 3 (b)). For both non-vulcanized and vulcanized blends, the tensile strength of blend containing 0.4 wt% PLA (NVB3 and VB3) was lower than one containing 0.1 wt% PLA (NVB2 and VB2), respectively because of increased lubricating effect of PLA with increase in PLA content.

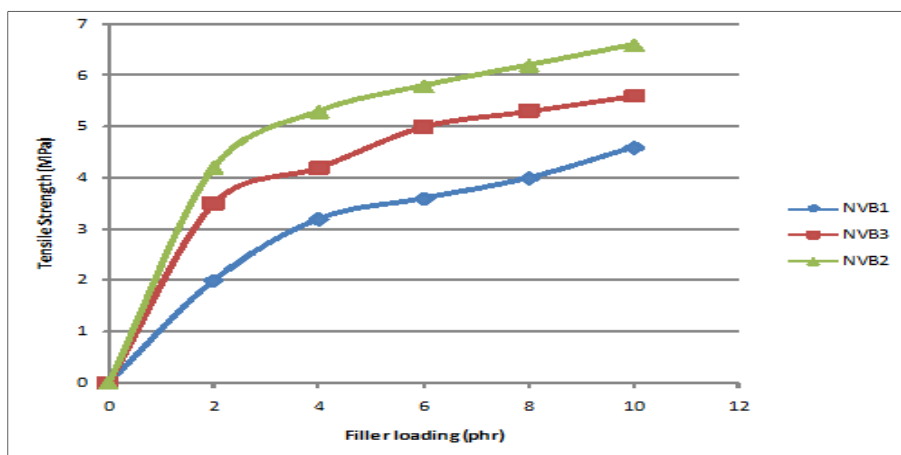


Figure 3 (a). Plot of Filler loading versus Tensile Strength for non-vulcanized PP/ENR blends

On the other hand, both non-vulcanized blends (Table 3) and vulcanized blends (Table 4) show a decreasing trend in elongation at break with filler loading due to decrease in chain mobility arising from formation of physical bonds between filler particles and polymer chains. However, the vulcanized blends displayed a greater decrease in elongation at break due to network formation and consolidation of these physical bonds with vulcanization. As shown in Tables 3 and 4, the elongation at break of blend containing 0.4 wt% PLA is higher than the one containing 0.1 wt% PLA because of increased lubricating effect of PLA with increase in PLA content.

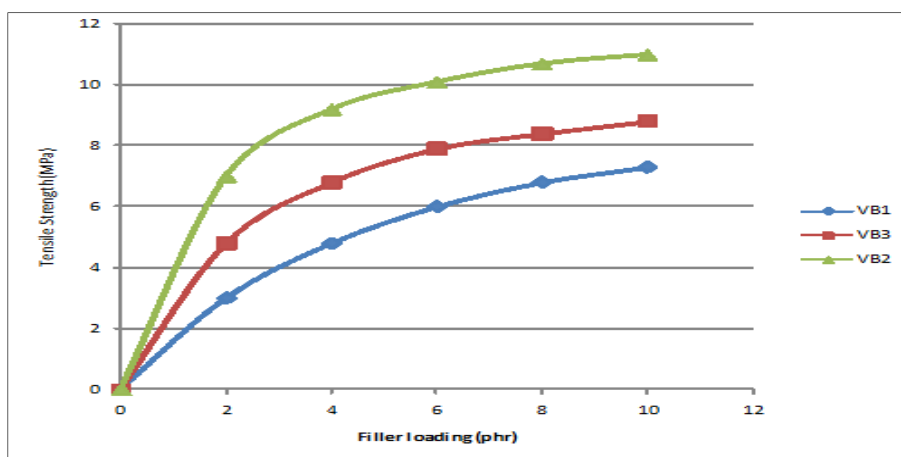


Fig. 3 (b). Plot of Filler loading versus Tensile Strength for Vulcanized PP/ENR blends

Table 3. Effect of filler loading on Elongation at Break for non-vulcanized PP/ENR blends

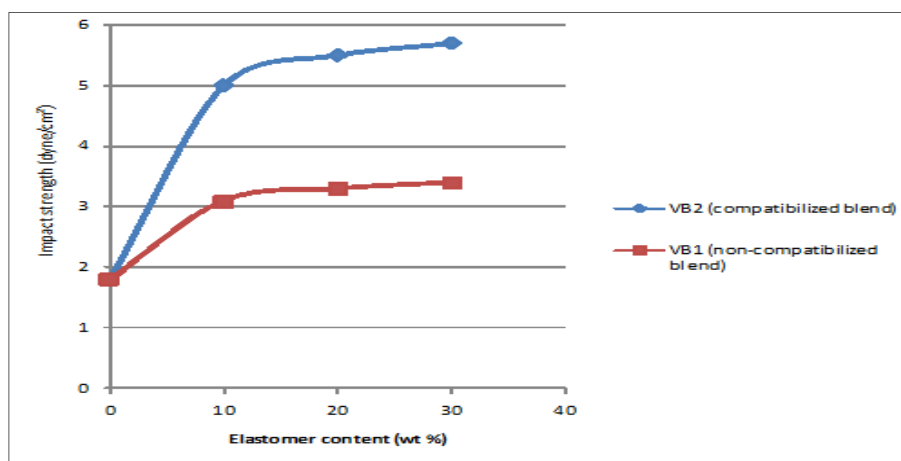
Filler Loading Phr	Elongation at Break (%)		
	NVB1	NVB2	NVB3
0	245	285	347
2	220	260	325
4	208	248	304
6	185	215	288
8	150	196	255
10	125	164	226

Table 4. Effect of Filler Loading on Elongation at Break for Vulcanized PP/ENR Blends

Filler Loading	Elongation at Break (%)			
	Phr	VB1	VB2	VB3
0		355	395	425
2		338	376	405
4		315	354	396
6		290	330	375
8		275	315	358
10		256	297	334

Impact Strength

In order to study the impact strength of PP/ENR blends, blend samples VB1 (non-compatible) and VB2 (compatible) were prepared with varying PP/ENR compositions of 100/0, 90/10, 80/20, and 70/30 by weight, respectively. The impact strength of each blend was measured as a function of elastomer content. The results are presented in Figure 4. In both cases, the impact strength increased with increase in elastomer content. However, the impact properties of compatible blends were higher than those of non-compatible blends. These observations are in agreement with the morphologies of the blends (Figures 1 (a) and (b)). For non-compatible blends, the dispersed phase displayed broad size distribution and poor adhesion to the matrix phase (Figure 1 (a)) so that the dispersed elastomer phase would not be able to act as an effective stress dissipating agent, resulting in poor impact properties. On the other hand, with the compatible blend, the morphology of the domain became smaller (Figure 1 (b)), and the stresses applied to the matrix (PP) will be transferred to the elastomer domains more effectively. The result is an improvement in impact strength as shown in figure 4.

**Figure 4. Variation of Impact strength with Elastomer content**

CONCLUSION

The following conclusion can be drawn from this work:

1. PP was melt blended with ENR using PP-g-MA/PLA and carbonized Dika nutshell as compatibilizer and filler, respectively. While one portion was non-compatible/non-vulcanized, the other was compatible/vulcanized. The morphological, rheological and mechanical properties of the non-compatible/non-vulcanized and compatible/vulcanized blends were compared. Compatibilization produced increases in complex viscosity, storage modulus and impact strength. Dynamic vulcanization also resulted in

increases in these properties, which were higher than those observed for non-compatibilized/non-vulcanized blends. The increase in complex viscosity, storage modulus and impact strength with compatibilization and vulcanization is due to molecular build-up arising from chemical reactions that occurred during the melt blending process, as well as increase in network formation caused by vulcanization.

2. Outstanding mechanical performance, especially tensile strength and impact strength (higher values), and elongation at break (lower values) were obtained with increase in filler content.
3. The moisture sensitivity of poly (lactic acid) (PLA), as well as its brittleness and high production cost greatly limit its application. However, PLA is a biodegradable polymer. In this study, it was used in very small quantity as a coupling agent to increase the interfacial adhesion between PP and ENR as well as to confer biodegradability to PP/ENR blends. Again, Dika nutshell used as filler in this work is a biodegradable agricultural by-product which improves biodegradability of the filled blends. Biodegradable PP/ENR blends will reduce the impact of non-biodegradable polymeric materials on our solid waste stream. This is in line with global environmental concerns which have suggested a need for materials that are biodegradable because of the decreasing availability of landfill space as well as increasing cost of municipal solid waste disposal. Furthermore, as particulate filler obtained from a renewable resource, Dika nutshell offers reduction in production cost advantage over the use of conventional, but costly petroleum-based reinforcing fillers such as carbon black. Thus, cheap, process able and biodegradable PP/ENR blends with improved mechanical properties were prepared.

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