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The Potential of O-MMT as a Reinforcing Filler for Uncured and Dynamically Cured PVC/XNBR Composites

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Organic montmorillonite modified with quaternary ammonium (O-MMT) was compounded with uncured and dynamically cured poly(vinyl chloride)/carboxylated nitrile butadiene rubber (PVC/XNBR) composites, using a Brabender Plasticorder at 130°C and 50 rpm rotor speed. The reinforcing efficiency of the O-MMT was investigated in the uncured PVC/XNBR composite and the dynamically cured PVC/XNBR counterpart. Mixing and dynamic curing of the composites were monitored by typical torque-time curves derived from a Brabender internal mixer. The torque-time curves revealed that the dynamic curing process was successful and the incorporation of O-MMT has no adverse effect on the processibility of the composites. It has been found that the introduction of crosslinks within the rubbery phase in the presence of the O-MMT has further improved the tensile properties. DMA studies revealed that dynamically cured composite with O-MMT showed higher storage modulus than the composite without O-MMT. Furthermore, a one-step tensile modulus vs. temperature curve and a related one peak tensile loss modulus vs. temperature curve were obtained, consequently, both are characteristics of a miscible polymers system. Further evidence on the composite miscibility was purchased by thermal scans from DSC, which showed a single glass transition temperature of PVC/XNBR composites. This claim was further supported by ATR-IR spectra which revealed that hydrogen bonding is extensively involved in PVC/XNBR composites. This evidence unveiled the exact nature of the specific interactions responsible for miscibility and hence, enhanced mechanical properties. Furthermore, we proved in our studies the reinforcing role played by layered clay due to better dispersion, as well as improved interactions.

Keywords: dynamic vulcanization; hydrogen bond; tensile strength; miscibility; composites

1. Introduction

Polymer blends, particularly prepared through melt blending, became a highly demanding class of materials with a balance of properties replacing many conventional materials where a pristine polymer cannot support (1, 2). Blends based on rubber and plastic could be either simple thermoplastic polyolefin or elastomeric alloys where the rubber phase is partially cured (3, 4). Apart from their dual characteristics of cured rubber and thermoplastic properties, the possibility of adjusting their properties by different routes makes elastomeric alloys a versatile class of materials. Incorporation of various additives is one of these routes. Fillers are widely used additives in significant ratios in such alloys. In this regard a considerable amount of work was reported (5, 6). Incorporation of a filler into a polymer alloy enhances some properties at the cost of

some other properties. They might influence the rheological, curing and elastomeric properties of such alloys, additional influences include the compatibility of alloy components. Nevertheless, implementation of fillers into these type of alloys has not gained much interest and is rarely reported. In the light of increasing number of polymer alloys, it is interesting to study a new class of polymer blends in the form of polymer composites via the incorporation of fillers into the blends as extra cutting down the materials cost with various properties. In this regard, various filler types are in use for plastic and rubber (7, 8). One of these fillers is clay being used as a rubber filler for many years. The reinforcing ability of clay is poor due to its particle size and low surface activity. Recently, a new way to improve the reinforcing ability was found by incorporation of layered clay in various polymers. In order to achieve an influential property improvement, the platelets of the clay should be made accessible for the polymer chains. The compatibility of the clay with polymers is also tailored by the ion exchange reactions using organic quaternary ammonium salts with long chains. Melting intercalation can be applied to most polymers, but it needs a polymer that has good processing properties in the melting state.

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Thus, the resulting organophilic clay platelets can be well dispersed into a range of commodity polymers from essentially non-polar polystyrene, to weakly polar poly(ethylene terephthalate) to moderately polar poly(vinyl chloride) to strongly polar nylon, exploiting the shear stress field generated in the mixing equipment, albeit other ways can be applied such as *in situ* and solution polymerization. Several investigations on the influence of O-MMT on the mechanical, thermal and barrier properties of thermoplastics (9, 10) and thermosets are extensively reported in the literature (11, 12). However, there were few studies on thermoplastic elastomers. Therefore, the innovation in this study is the incorporation of the layered clays within the uncured and dynamically cured thermoplastic polyolefin based PVC/XNBR formulations. The effects of dynamic curing and O-MMT on the processibility, mechanical performance, dynamic mechanical and IR properties are reported in this article.

2. Experimental

2.1 Materials

Carboxylated nitrile rubber Krynac X 740, was supplied by LANXESS Deutschland GmbH. Suspension poly(vinyl chloride) (PVC) grade in powder form with a k-value of 67 and stabilized with lead salt, quaternary ammonium salt modified natural montmorillonite, Cloisite 30B were obtained from IPF Dresden e.v. Stearic acid and zinc oxide powder were supplied by ACROS Organics, Belgium, MBTS and Sulfur were supplied by Continental AG, Hanover. All chemicals were used as received.

2.2 Formulation

The recipe used to fabricate PVC/XNBR filled O-MMT composites are presented in Table 1.

2.3 Sample Fabrication

The blends were fabricated by melt compounding in a Brabender Plasticorder Model PLE 331. The instrument was equipped with a torque rheometer and a thermocouple for continuous measuring of the melt torque and temperature, respectively. The composites were melt mixed at 130°C and a rotor speed of 50 rev. min⁻¹ for 6 min. The cure

characteristics were determined at 160°C using an Ekktron oscillating disc rheometer at 1.66 Hz frequency and 0.5 arc. The obtained blends were sheeted into 2 mm thickness by preheating at 160°C for 2 min followed by compression for 6 min at 12 MPa pressure. Attenuated total reflection spectroscopy experiments were carried out on a Bruker FT-IR spectrometer at room temperature on pure samples and composites, with a 4 cm⁻¹ resolution and 32 scan signal from 400–4000 cm⁻¹ in the transmission mode.

2.4 Testing

2.4.1 Tensile Tests

Tensile properties were carried out according to ASTM D638 on Zwick 1456. The dumbbell specimens were cut from molded PVC/XNBR filled O-MMT composites. Five specimens were tested and the median value was taken for each formulation.

2.5 Hardness

Circular discs of 2 mm thickness were tested on a ASTM shore A hardness tester in International Rubber Hardness Degrees (IRHD) according to ISO 48(1979).

2.6 Failure Mode

The surfaces of the prepared materials were inspected for evidence of miscibility using a scanning electron microscope (SEM) type (geol Toleyo, Japan). The specimens were sputtered with Au-Pd alloy prior to scanning.

2.7 Thermal Analysis Process

A dynamic mechanical thermal analysis (DMTA) test was run with a Rheometric scientific thermal analysis system in the temperature/time scan mode. Temperature/time scanning was performed from -80–100°C at a heating rate of 5°C/min in a dual cantilever mode at frequency of 1 Hz. Storage modulus and glass transition temperature T_g were determined. T_g was taken as the temperature of maximum peak in the loss modulus vs. temperature curve. Further evidence on the miscibility of the composites was overwhelmed by a Perkin-Elmer DSC-6 differential scanning calorimeter (DSC) in the range of -100 to 100°C at a heating rate of 10°C/min.

Table 1. Recipes used to produce PVC/XNBR blends (phr)

Composition	1	2	3	4
PVC	70	70	70	70
XNBR	30	30	30	30
MMT	—	3	—	3
St. acid	2	2	2	2
Sulfur	—	—	0.6	0.6
TMTD	—	—	0.2	0.2

3. Results and Discussions

3.1 Torque-Time Curves

Figure 1 depicts the torque–time curves for pristine PVC/XNBR composites at 130°C and 50 rpm. The curve can be interpreted as follows. XNBR is charged into the mixing chamber in the region A, hence, the torque increased to B. This is due to the resistance exerted by the XNBR

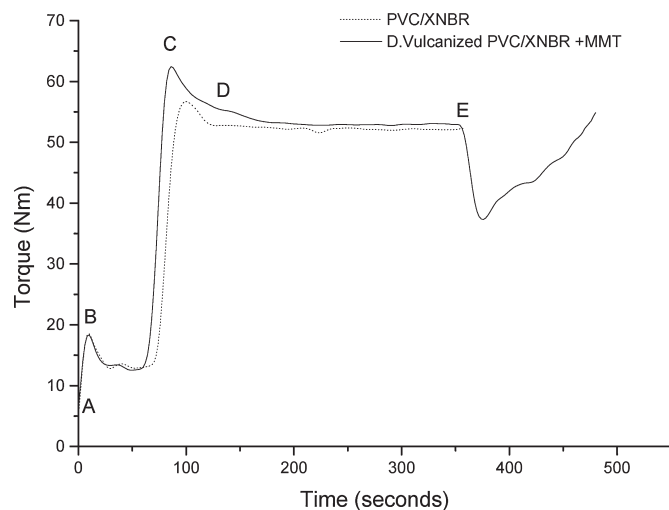


Fig. 1. Brabender plastograms for PVC/XNBR plain blend and dynamically vulcanized PVC/XNBR filled MMT.

against the rotors. Once the homogenization process is complete the torque starts to decrease, mostly due to the temperature rising. The thermoplastic (PVC in this case) was added at the 2nd minute and torque increases again. The torque increment should be attributed to the fusion process of the PVC consequently, the viscosity will increase, recall that viscosity is a direct function of the torque. The increase in viscosity, coupled with shearing, generates a frictional heat build up which is expected to increase the melt stock temperature.

The increase in temperature is coupled with a decrease in torque until complete fusion of PVC was observed as indicated by fusion peak at C, fusion continues until D. Then, the melt temperature stabilizes giving rise to a constant torque. The melt was allowed to homogenize by mixing till E. Figure 1 also shows the influence of O-MMT and dynamic curing on torque-time curve of PVC/XNBR composites. More or less similar torque-time curve to that of the pristine composition is observed. However, examination of torque values further reveals that final near equilibrium torque values of the O-MMT filled composites showed only a slight rise than the unfilled system. This indicates that the melt viscosity of the composites at the processing temperature was not significantly affected by the incorporation of the O-MMT, albeit the melt strength in the presence of the filler has increased due to the expected physical linkage between the O-MMT layers and the PVC/XNBR composites, as well as the distribution process of the filler, i.e.,

intercalation/exfoliation in this case. Furthermore, it can be seen that the processibility or the fusion characteristics of the PVC compounds are influenced principally by the formula composition. Note that the fusion peak of the PVC has shifted to a lower value as compared to control sample, simultaneously, the gelation speed of composites has increased tremendously with O-MMT incorporation as summarized in Table 2. This could be due to the fact that the interlayer of the platy O-MMT may take the role of the plasticizer for the PVC chains resulting in a decreased interaction forces between the PVC molecule and lead to a loose PVC/XNBR based O-MMT compounds with lower fusion temperature. Similar claims were reported earlier on PVC/O-MMT composites (13). Figure 1 also reflects the influence of dynamic curing on PVC/XNBR filled O-MMT composite. The curing ingredients including sulfur were added at E region, i.e. the 6th minute of mixing. Note that torque starts to increase and stabilizes at higher values than corresponding uncured counterpart composites after the addition of the curatives. This was the first indication that dynamic curing was affected with a higher energy requirement for the melt processing of the dynamically cured blend than the plain blend.

3.2 MDR Studies

The elastic torque at minimum value (ML) and the maximum elastic torque (MH) are shown in Figure 2. It can be seen that the uncured filled O-MMT formula has recorded the minimum ML value compared to the control and the dynamically cured formulation. It has been reported that the reduction of ML is a clue on an improved processibility leading to reduced melt viscosity of the blend (14). The minimum value should be traced to the role of O-MMT where the interlayer of the O-MMT may act as a plasticizer, increasing the distance between PVC/XNBR chains leading to the observed trend (15). Hence, it can be inferred that the incorporation of O-MMT has improved the processibility of the blend by reducing the value of ML, since ML is commonly considered as representative of the uncured elastic modulus (16). On the other hand, the dynamically cured blend showed a higher ML value even in the presence of O-MMT. This scenario could be related to the influence of dynamic curing. The dynamically cured rubber particles encapsulated by the thermoplastic matrix are expected to increase the viscosity of the blend and hinder the flow characteristics of the compositions, hence higher ML values are recorded. Figure 2 shows MH for different

Table 2. Fusion behavior of PVC with and without MMT as derived from Brabender plasticorder

Composite	Fusion time (Minutes)	Gelation speed (Nm/min)	End torque (Nm)
PVC/XNBR	1.30	233.4	44.8
PVC/XNBR/MMT (D. Vulcanizate)	1.18	270.0	55

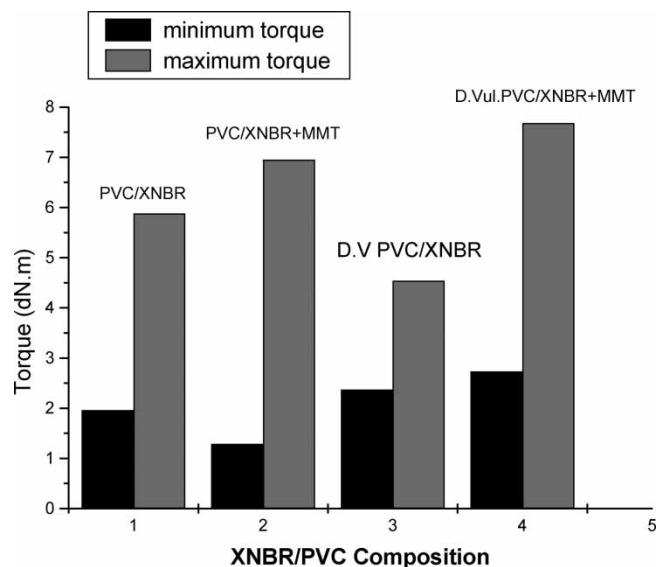


Fig. 2. Maximum torque of PVC/XNBR with different compositions as derived from MDR at 160°C.

four compositions as well. Remember that MH is a measure of the degree of interactions and the stiffness of the composite. In this regard, the degree of interactions has increased after the incorporation of the O-MMT without any curing as shown in Figure 2. This could be due to the physical interactions between the polar O-MMT and the polar PVC/XNBR system. In addition, it is believed that the smaller the particle size, the greater the surface area, which implies the greater rubber-filler interactions. This tends to impose extra resistance to flow as a higher restriction to the macromolecular motion of the polymeric composition (17). Furthermore, it can be seen that MH decreased in the case of dynamically cured formulation without O-MMT as demonstrated in Figure 2. This observation could be related to the type of curing system implemented in this investigation (TMTD/Sulfur) without zinc oxide. One should not forget that zinc oxide, in combination with TMTD, forms a highly sulfuring complex which acts as a precursor of an ultimate matured network. So the ultimate strength very much depends upon the initial curing reaction. After the incorporation of the O-MMT into the dynamically cured blend a dramatic increase in MH value was recorded as reflected in Figure 2. Obviously, this should be attributed to a matured curing reaction in the presence of O-MMT. The O-MMT has facilitated the curing reaction due to the amine functionality of the O-MMT. Earlier investigations (18–20) revealed that certain organic substances containing nitrogen atoms promote the curing of olefinic rubber comprising sulfur and accelerators through the formation of complexes. These complexes were made responsible to promote the break up of sulfur molecules and to form crosslinks between the leaner rubber chains. In the context of the present investigation it is highly possible that the ammonium salt in the O-MMT facilitated the curing of

XNBR phase. This observation is in agreement with recent findings on the application of O-O-MMT in SBR vulcanizates and hydrothermally decomposed polyurethane in natural rubber and nitrile rubber, respectively (20, 21). Thus, it can be concluded that organoclay is not only a filler but also acts as a co-curing agent which could facilitate the curing reaction. Table 2 compares $\tan \delta @ ML$ derived from MDR for the four different composition. It is obvious that the incorporation of the O-MMT has slightly increased $\tan \delta$ as compared to the pristine composition, while dynamic curing has significantly decreased $\tan \delta$ even in the presence of O-MMT. The observed trend could be related to the absence of chemical crosslinks in the case of the plain blend and the O-MMT filled composites, and to the presence of chemical crosslinks in the case of dynamic curing process. This agrees quite well with a previous work on the cure characteristics of XNBR vulcanizates (22). Hence, it can be predicted that dynamic curing and O-MMT has reduced the hysteresis behavior of the PVC/XNBR composites with improved resilience. Similar results were recorded in the case of $S'' @ MH$ which is also a damping indicator. This observation concurs a previous work on poly(vinyl chloride)/epoxidized natural rubber blends (23).

3.3 Infrared Spectroscopy (AT-IR)

In IR spectra of polymers, the hydrogen bonding peak normally occurs at 3200 to 3500 cm^{-1} depending on the molecular environment (24). For example, Cowie (25) in his work on specific interactions in poly-blends of polystyrene, observed the hydrogen bonding peak at 3500 cm^{-1} in blending with poly(alkylmethacrylates). A similar shift is observed in PVC and PVC/ENR blends by other workers (26). Several researchers deduced hydrogen bonding from IR spectra of blends (26, 27). Figure 3 shows that both polymers, PVC and XNBR, are individually capable of hydrogen bonding, as well as O-MMT.

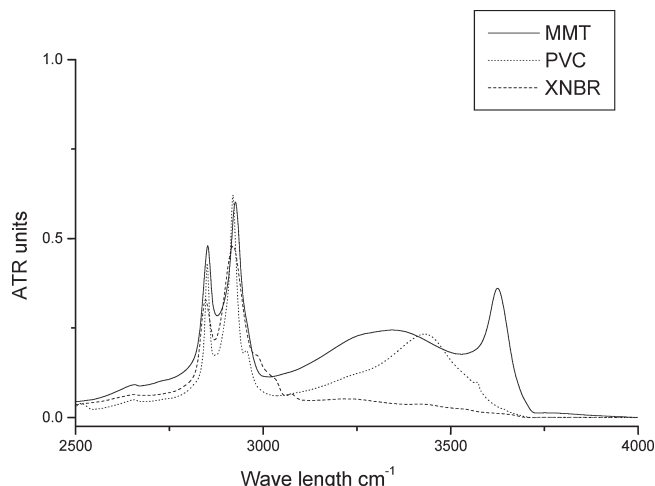


Fig. 3. AT-IR spectra of the PVC, XNBR, and O-MMT.

Figure 4 shows the IR spectra of the PVC/XNBR composites with and without O-MMT. The broad peak prominent at 3303 cm^{-1} in the blend absorption is a very clear signature of hydrogen bonding. Note, that the incorporation of O-MMT has slightly increased the peak intensity of hydrogen bonding at 3303 cm^{-1} as illustrated in Figure 4. This indicates the important role played by hydrogen bonding in PVC/XNBR filled O-MMT composites. Based on this, it could be inferred that hydrogen bonding is involved in this system. Shortly, the presence of hydrogen bonding could be used to account for miscibility and enhanced mechanical performance as will be elaborated later.

The difference spectrum is basically the spectrum of interactions which, in this case, is the spectrum due to curatives concentration and the subsequent interaction between the curatives and the XNBR to yield crosslinks. This notion is clearly indicated in Figure 5. It is obvious that the addition of the curing system results in new peaks as a function of curatives. In this regard, the absorption at 1697.9 cm^{-1} is indicative of the carbonyl of the stearic acid. The absorption from $1600\text{--}900\text{ cm}^{-1}$ could be related to the C=S bond of the TMTD which is attached to nitrogen atom. The spectra appear as a series of bands which could be related to the interactions between C=S and C-N stretch. The S-H stretch appears at 2500 cm^{-1} , while the C-S bond is indicated at 650 cm^{-1} . The difference IR spectra for the dynamically cured composite filled with O-MMT minus the pristine one is presented in Figure 5 as well. It is shown that similar spectra to the previous one is recorded. The only difference is the peak around 3625 cm^{-1} of this material. The observed absorption could be due to the presence of hydrogen bonding which is the contribution of -OH on the surface of organoclay. Peaks at 2920 cm^{-1} and 2849 cm^{-1} are coming from the aliphatic $\text{-CH}_2\text{-}$ group in the rubber backbone and also in the organic modifier of the O-MMT. The characteristic peak for Si-O-Si group appeared at 1002 cm^{-1} and also at 1040 cm^{-1} .

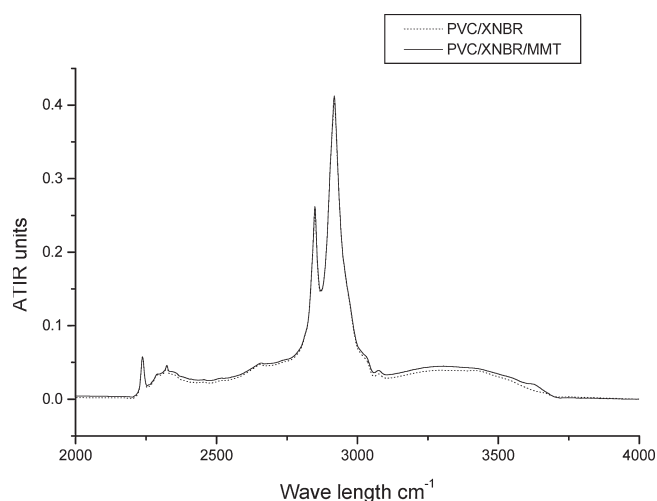


Fig. 4. AT-IR spectra of PVC/XNBR with and without O-MMT.

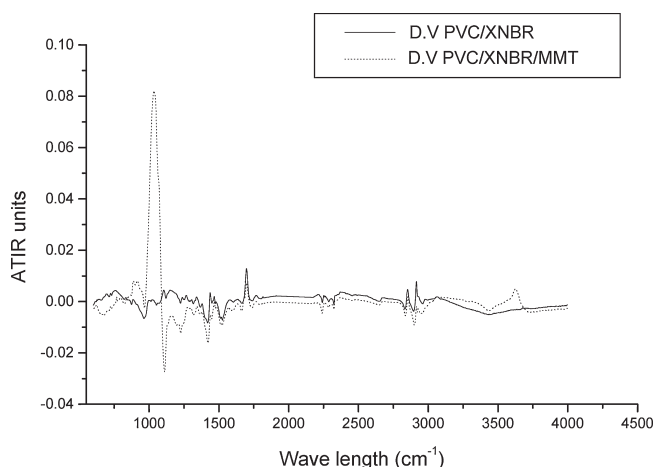


Fig. 5. AT-IR difference spectra of dynamically cured VC/XNBR with and without O-MMT.

3.4 Thermal Analysis

The effect of O-MMT and dynamic curing on the dynamic mechanical properties were studied in the temperature range between $-80\text{--}100^\circ\text{C}$. The storage modulus is shown in Figure 6.

The incorporation of O-MMT and dynamic curing increased the storage modulus in comparison to the pristine formula. Such observation is additional evidence for successful dynamic curing of O-MMT filled composites. Although the storage modulus of the composites decreases with temperature, it demonstrates that dynamically cured PVC/XNBR composites filled with O-MMT exhibited higher storage modulus than the control. It is believed that O-MMT has the ability to absorb heat and retard the softening of the polymers, as mineral fillers are known to enhance the dimensional stability (28). This observation is in line with previous work on elastomers filled nanoclay (29). Note that

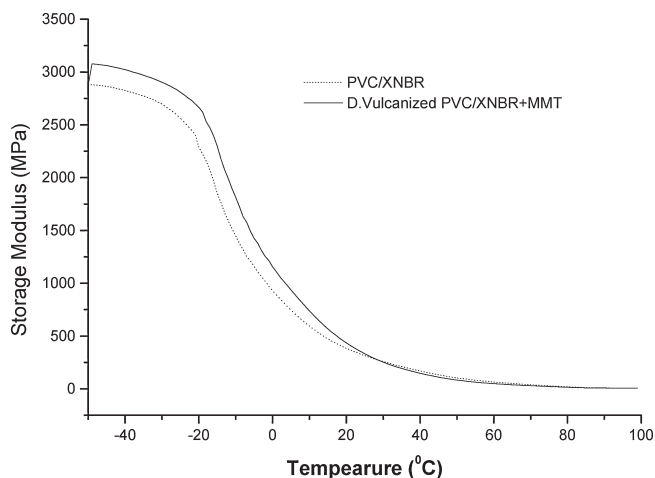


Fig. 6. Storage modulus of PVC/XNBR at various compositions as derived from DMTA at broad temperature scan.

a one-step storage modulus vs. temperature curve and a related one peak loss modulus vs. temperature curve shown in Figures 6 and 7, consequently, both characteristics of miscible polymers were observed. The miscible nature of the composites should be due to the specific interactions (namely, the hydrogen bond) between the $\sim\text{C-Cl}$ of the PVC and the $\sim\text{OH}$ and $\sim\text{NH}$ of the elastomer and the O-MMT, respectively, reflected by the IR spectra and elaborated in the previous section. Also, it can be seen that the O-MMT loading has slightly increased the intensity and the position of $\tan \delta_{\text{max}}$. The former could be correlated with the plasticizing role exerted by the layered nanoclay, while the latter is related to the dynamic vulcanization of the elastomer of rubbery composites, as the introduction of crosslinks hinder the flow characteristics of the composites. The lubricating effect of the layered O-MMT is expected to enhance the molecular motion of the composites and hence, increased magnitude of T_g (30). As a further support on the miscible nature of the composites, the DSC scan was run from sub-ambient to ambient temperatures as shown in Figure 8. It can be observed that a single peak was detected. This is similar to our notice from DMTA which supports our claims of the miscible nature of the composites.

3.5 Tensile Properties

Figure 9 compares the effect of O-MMT and dynamic curing on the tensile strength of the PVC/XNBR composites with the uncured counterpart. It can be seen that the incorporation of the O-MMT has increased the tensile strength of the material as compared to the control sample. This should attribute to the interaction between O-MMT and the PVC/XNBR composites, thus highlighting a good reinforcing efficiency of the O-MMT. The small particle size provides a larger surface for the interaction between the filler and the polymer matrix. In this regard, one should not forget the polar nature of both, O-MMT and the PVC/XNBR matrix, which is expected to

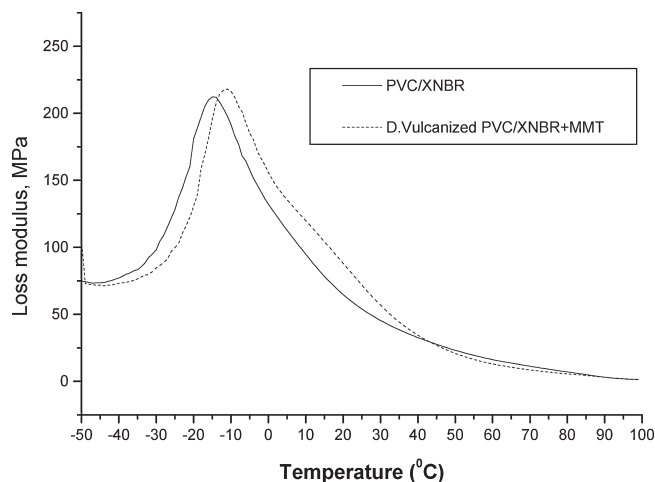


Fig. 7. Loss modulus of PVC/XNBR at various compositions as derived from DMTA at broad temperature scan.

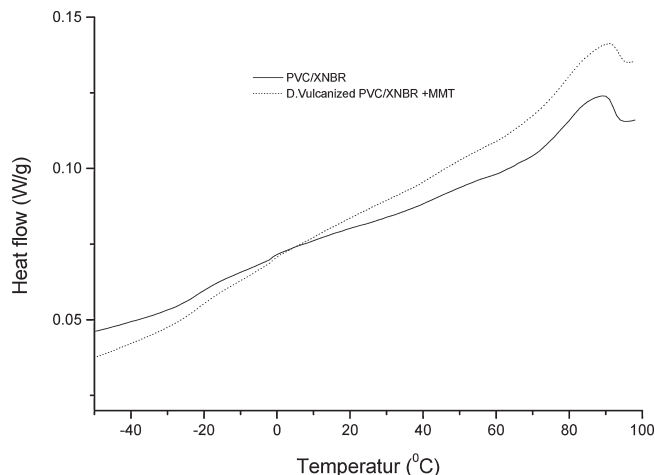


Fig. 8. DSC traces of undured and dynamically cured PVC/XNBR with and without MMT.

facilitate such interactions. On the other hand, the improved tensile strength could be due to the high aspect ratio of the platelet structure of the O-MMT. This is in line with previous work on the role of O-MMT on several rubber stocks such as EPDM, NBR/SBR blends ENR compatibilized NR vulcanizates (30, 31, 17). Further, observe that the dynamically cured blend obtained lower tensile strength as compared to the control formulation with and without O-MMT. Again, this scenario could be related to the un-matured curing reaction in the absence of zinc oxide as elaborated earlier. Recall that due to the incorporation of the O-MMT into the dynamically cured blend a significant increase in tensile strength value was recorded as depicted in Figure 9. This should be attributed to a matured curing reaction in the presence of O-MMT. The O-MMT has facilitated the curing reaction due to the amine functionality of the O-MMT (20–22). Thus, it can be again concluded that organoclay is not only a filler, but also acts as a co-curing agent, which could facilitate the curing reaction. The enhanced tensile strength of the cured blend over the uncured are well explained in the literature for the unfilled plastomer/elastomer blends (32). The cured rubberized particles in the

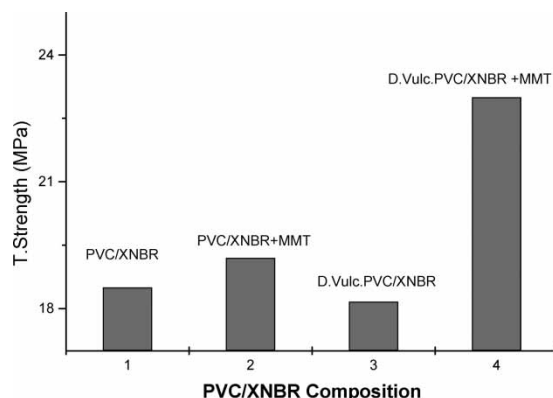


Fig. 9. Tensile strength of PVC/XNBR at various compositions.

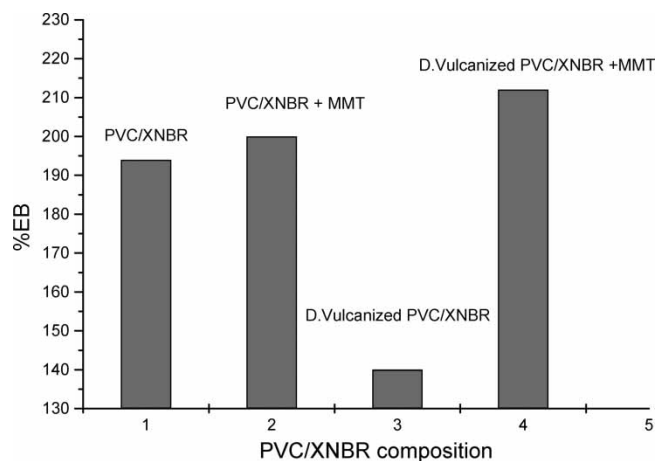


Fig. 10. % Elongation at break of PVC/XNBR at various compositions.

composites, facilitating stress transfer is the clear factor contributing to such enhancement. Recall that the incorporation of O-MMT decreased the elastomer portion, which implies that formation of crosslinks is not the only factor that governs such improvements. As evidenced by SEM studies, dynamic curing has resulted in the interruption of filler agglomeration and, as a consequence, enhanced filler dispersion. Well dispersed and finer filler particles contribute to enhanced mechanical properties. Similar trends were recorded in the case of %EB and tensile modulus presented in Figures 10 and 11, respectively. The same reasons hold to explain the trend observed in the case of %EB. As for the tensile modulus, it can be inferred that O-MMT with dynamic curing has enhanced the stiffness of the materials. Fillers are known to increase modulus, provided that the modulus of the filler is higher than that of polymer matrix. These results show that the tensile moduli of the prepared composites follow the same trend as tensile strength and elongation at break. The effect of O-MMT and dynamic curing on the hardness is illustrated in Figure 12. The trend here is similar to that recorded in the case of tensile

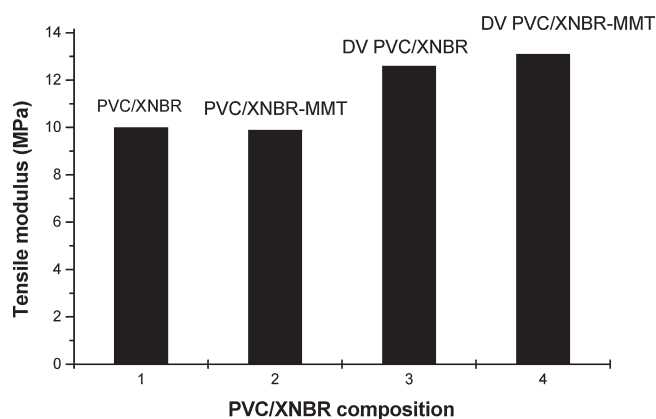


Fig. 11. Tensile modulus of PVC/XNBR at various compositions.

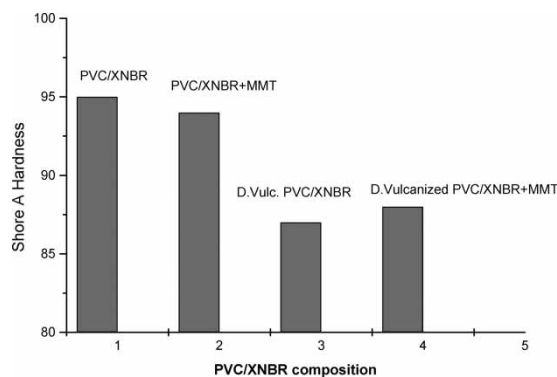


Fig. 12. Shore a hardness of PVC/XNBR at various compositions.

strength. This could be attributed to the plasticization role of O-MMT interlayers and the immature curing reaction in the absence of ZnO and the co-curing functionality of O-MMT. The density vs. composition is shown in Figure 13. The increase or decrease in density could be reasonably attributed to closer packing or better spatial arrangement of the polymer chains due to increased interaction between the composite components. These observations are in strong agreement with an earlier report (33) which postulated that the improved properties might be due to the observed increase in packing density on blending. The observations from this investigation tend to favor these views as indicated in Figure 13. Again, this confirms our findings on miscibility of the composites derived from AT-IR and thermal analysis presented in Figures 3–8 respectively.

3.6 Scanning Electron Microscope

The scanning electron micrographs for the control sample and the dynamically cured filled O-MMT counterpart are presented in Figure 14(a and b), respectively. Evidence of uniform dispersion is shown at this level of magnification. The obvious difference compared with the uncured sample is that the cured blend has a rugged phase surface over the

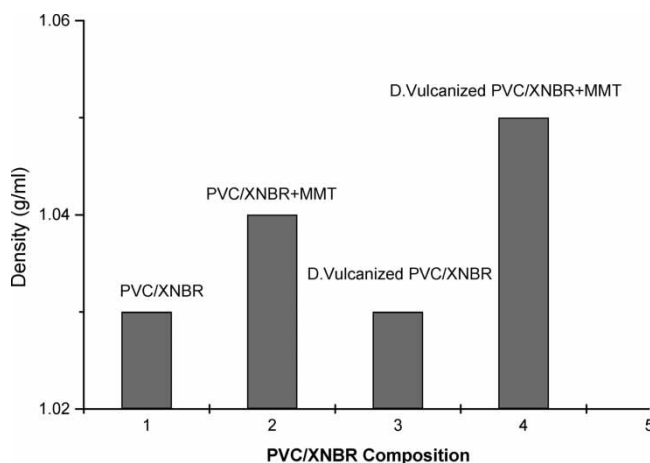
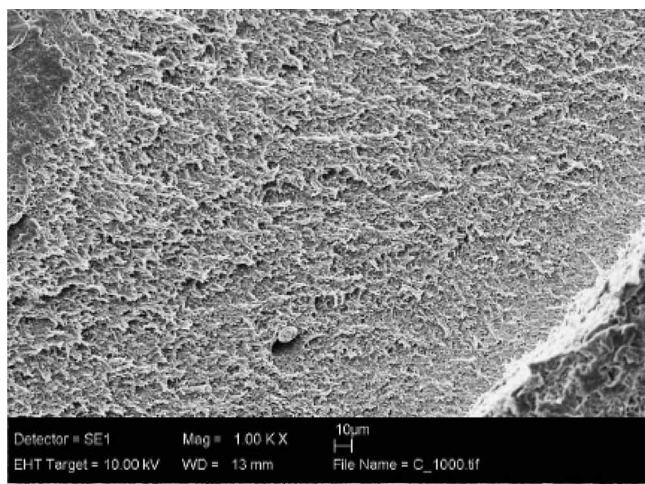
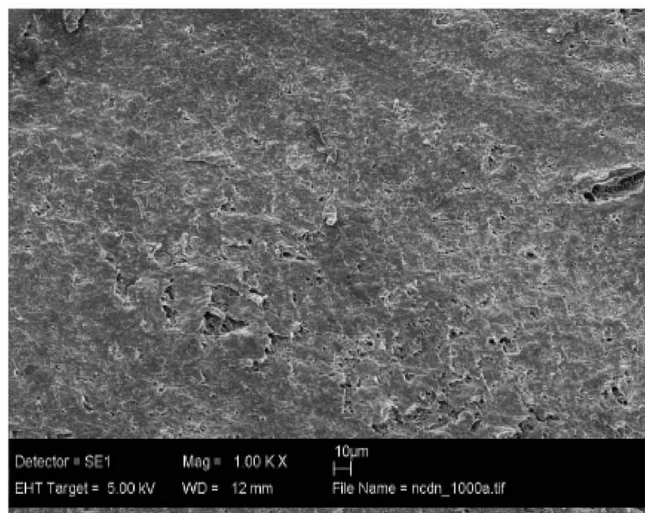


Fig. 13. Density of PVC/XNBR at various compositions.



(a)



(b)

Fig. 14. Scanning electron microscope pictures of (a) uncured and, (b) dynamically cured PVC/XNBR with O-MMT composites.

uncured counterpart. Furthermore, the cured blend filled with O-MMT has retained a more uniform surface which could be attributed to the influence of dynamic curing. However, it was difficult to distinguish the individual PVC or XNBR phases in both formulas, i.e. the uncured and cured one. This might suggest a good miscibility of the blend components as demonstrated by Figure 14(a and b), respectively.

4. Conclusions

The prevailing work was devoted to study the effect of O-MMT as a reinforcing filler in uncured and dynamically cured PVC/XNBR. Based on the presented results, the following conclusions can be drawn: Torque rheometer curves showed that O-MMT has reduced the fusion time, which was attributed to the plastization role of the platy-like structure of O-MMT. Further, the equilibrium torque of

formulation has increased with O-MMT loading, thus highlighting stiffer compounds. The plastograms showed that dynamic curing of the XNBR phase during melt mixing was affected. Cure characteristics of the materials derived from MDR have shown that the O-MMT can act as a co-curing agent in addition to reinforcing filler. New evidence from AT-IR studies point to the fact that hydrogen bonding is involved in PVC/XNBR systems which could be used to account for enhanced properties, pointing out the role of hydrogen bonding in this system. Dynamic mechanical thermal analysis investigations demonstrated that a single phase system exists even after the incorporation of O-MMT. The tensile properties were improved with increasing clay content due to the improved degree of interaction, highlighting the efficient reinforcing role practiced by O-MMT.

5. References

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