Effect of Modified Liquid Rubber on Increasing Toughness of Epoxy Resins

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ABSTRACT

Epoxy resins are considered as one of the most important classes of thermosetting polymers for many industrial applications, but unfortunately they are characterized by a relatively low toughness. In this respect, many efforts have been made to improve the toughness of cured epoxy resins by the introduction of rigid particles, reactive rubbers, interpenetrating polymer networks and thermoplastics within the matrix. In this work, hydroxyl-terminated polybutadiene (HTPB) as a modifier, firstly, was cross-linked by variable content of divinylbenzene (DVB) in the presence of epoxy resin (ER) and then the modified matrix was cured with 1-methylimidazole as a curing agent. Infrared spectra showed the existence of a chemical reaction between modified HTPB (MHTPB) and the ER. Most of the tensile properties attained a peak at an approximately 20 phr (part per hundred rubber) DVB content, where the toughening reached its maximum. For both notched and un-notched specimens, a two-fold increase in izod impact strength was obtained by the addition of just 20 phr DVB compared to the neat resin. On the addition of DVB, the Izod impact strength varied from 0.54 to 0.71 kJ/m² for notched specimens and from 1.43 to 6.66 kJ/m² for un-notched specimens. Whereas, KIC varied from 1.35 to 2.59 MPa.m¹/² with increasing DVB content. By SEM analysis the average diameter was found to be about 5 µm (corresponding to maximum of toughness) for modified rubber particles. The overall results have shown that it is possible to obtain an excellent impact strength and good mechanical properties with the use of MHTPB as a toughening agent for the epoxy resins.

INTRODUCTION

Epoxy resins, are characterized by the presence of a three-membered ring known as the epoxy, epoxide, oxirane or ethoxyline group. Commercial epoxy resins contain aliphatic, cycloaliphatic or aromatic backbones. The most widely used one is epichlorohydrin and bisphenol-A derived resins. The outstanding performance characteristics of these resins are conveyed by the bisphenol-A (toughness, rigidity and elevated temperature performance), the ether linkages (chemical resistance) and

Key Words:
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rubber particles;
toughness;
modification;
toughening.
the hydroxyl and epoxy groups (adhesive properties and formulation latitude, or reactivity with a wide variety of chemical curing agents). Epoxy resins consist of three-dimensional networks or agglomerates of moderate molecular weight. As the molecules polymerize through cross-linking, their rotational and transitional freedoms are reduced, which reduces the chances for primary bonds to be set up with adjacent molecules. The tensile and compressive strengths of epoxy resins are increased if the distance between cross-links is shortened. Shorter cross-link distance implies a high volume concentration of epoxy groups and hence a higher probability of chemical reaction of all the epoxy groups [1-5].

Epoxy resins are considered as one of the most important classes of thermosetting polymers and are extensively used for their many good properties, which include high stiffness and strength, creep resistance, chemical resistance and good adhesion to many substrates. These resins have found wide uses in various fields of coatings, high performance adhesives, advanced composites for the aerospace, electronic industries and the other engineering applications. Once cured, they are characterized by high chemical and corrosion resistance as well as good mechanical and thermal properties. However, in many applications they have one major drawback, they are very brittle with poor resistance to crack propagation and low impact strength (IS), and therefore, they exhibit low toughness [1-2, 5-6].

The aim, when attempting to toughen brittle polymers, is to increase their toughness without any significant reduction other important properties such as modulus and heat distortion temperature. Most of the reported studies have been carried out using glass beads, alumina trihydrate, silica [7], and one of the most successful involves the addition of a suitable rubber such as liquid amine-terminated (ATBN) [8-10], carboxyl-terminated (CTBN) [9, 11], hydroxyl-terminated (HTBN) [12], epoxy-terminated (ETBN) [13] copolymers of butadiene and acrylonitrile, hydroxyl-terminated [14-15] and epoxy terminated [16] polybutadiene, more recently through the use of thermoplas-tics (polyphenyl oxide, PPO) [17] and interpenetrating polymer networks (IPN) structures [18-19].

The first approach to modify epoxy resins is by addition of rigid particle, which generally leads to a significant reduction in cost and a considerable improvement in the resins mechanical, thermal, and electrical properties, namely, the elastic modulus, heat-distortion temperature, and dielectric strength. Considerable work relating to the effect of particulate fillers on the mechanical properties of thermoplastic and thermosetting polymers has been reported in literature. Parameters such as the volume fraction of the filler, particle size, modulus and strength of the filler, resin-filler adhesion, and toughness of the matrix have been extensively studied. The variation of some of these parameters leads to improved toughness of the filled material while increasing its strength and modulus [20].

The second method used to improve the toughness of epoxy resins involves an addition to the uncured epoxy resin of suitable rubbers that are copolymers with variable acrylonitrile contents. The effects of the acrylonitrile content of the copolymer type, the molecular weight, the concentration, and the solubility parameter of the rubber and its functional end groups on the fracture toughness or on the impact strength of the epoxy resin have been studied. In these studies, epoxy resins were modified with CTBN [9, 11], HTBN [12], or epoxy-terminated [13] copolymers of butadiene and acrylonitrile.

The degree to which a highly cross-linked epoxy resin can be toughened by rubber particles is very limited because its ability to deform by shear yielding is reduced with increasing cross-link density; therefore, a third technique has emerged. Recently, a highly cross-linked thermosetting system has been toughened by tough, ductile, and chemically and thermally stable engineering thermoplastics such as polyethersulfones, polyetherimides, phenolic hydroxyl-terminated bisphenol-A polysulphone, and PPO [17, 20].

Finally, IPNs are a novel type of polymer alloy intended, in the case of epoxy resins, to modify impact resistance. Interpenetration, or permanent entanglement, usually leads to improved compatibility, which means that a decreased degree of phase separation is attained. Morphology, which plays a major role in affecting IPN properties, is controlled by the chemical compatibility of the polymers, interfacial tension and cross-linking densities of the networks, polymerization method, and IPN composition [18-19].

The main objective of the present work is to evaluate the use of modified HTPB (MHTPB) with divinylbenzene (DVB) as an impact modifier for diglycidyl
ether of bisphenol-A (DGEBA) epoxy resin. It is well known that the reactivity and selectivity of the elastomer play an important role in the process. Functional groups must be present in the chain of the rubber in order to promote the formation of chemical or physical bonds with the epoxy matrix, which are necessary for an efficient stress transfer between the rubber particles and the matrix [21].

Also, the existing of hydroxyl groups-contained compounds in epoxy matrix will participate in curing reactions and increase the rate of the reactions [22], as it is observed in Scheme I. Therefore, the presence of HTPB causes the reactions to go forward furthermore and increasing the toughness of the matrix.

It is well known [23] that in order to toughen an epoxy resin with a liquid polymer, first, the liquid polymer should form a rubbery second phase which is dispersed throughout the matrix and second, the rubbery second phase should be bounded to the matrix through the functional groups of the liquid polymer.

In the present work, a series of HTPB/DVB-toughened epoxy materials, with different levels of DVB are evaluated for affecting most physico-chemical properties of an epoxy resin. It attempts to improve the toughness of DGEBA epoxy resin using 1-methylimidazole as a curing agent. The modifying agent is MHTPB rubber. The chemical reactions that possibly take place during the modification of the epoxy resin were monitored using a Fourier transform-infrared (FTIR), while glass transition temperature (Tg) was measured using a dynamic-mechanical thermal analyzer (DMTA). The mechanical behaviour of modified epoxy resins was evaluated in terms of the Izod impact strength, the critical stress intensity factor (KIC) at different modifier contents. The fractured surfaces were studied using a scanning electron microscope (SEM) to elucidate the mechanisms of deformation and toughening as well as other morphological features. Finally, stress-strain curves of the modified epoxy resin were evaluated in terms of studying tensile properties.

**EXPERIMENTAL**

**Material**

Epoxy resin system: A liquid epoxy resin system based on DGEBA (Scheme II) Araldite GY 6010, from Jana Co. with equivalent weight of epoxy groups equal to 182-192 g/eq, used in all experiments and it was dried at 80°C under vacuum for 1 h before use throughout this work. A liquid 1-methylimidazole, from Fluka Co. was supplied as curing agent of epoxy resin and used throughout this work.

Liquid rubber, Scheme II, and DVB: The elastomer employed was a liquid HTPB with Mn = 2900 contain-

<table>
<thead>
<tr>
<th>Table 1. Composition of epoxy resin /rubber/DVB systems for each component in the prepolymers and in the final materials.</th>
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</thead>
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<tr>
<td><strong>Materials code</strong></td>
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<tr>
<td>BV0(0)</td>
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<td>BV5(1)</td>
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<td>BV5(40)</td>
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ing terminal allylic OH groups and having equivalent weight of hydroxyl equal to 1280 g/eq, was supplied from the market and used as an epoxy modifier. Divinylbenzene (DVB), from Merck Co. as modifier of HTPB and azobisisobutyronitrile (AIBN), from Merck Co., as initiator of cross-linking reactions was employed in all the experiments.

Sample Preparation
Rubber and DVB (1-20 phr) as shown in Table 1, were first mixed together with AIBN (6% wt). Next, the mixture was added to epoxy resin with the weight ratio of 100:5 for epoxy resin/HTPB. The mixture was heated for 60 h at approximately 50-60°C, while stirring in N₂ atmosphere. Then, the mixture was cooled to 40°C and the curing agent of epoxy resin was then added prior to hand mixing and as drop by drop (10-15 min). Next, the resin mixture was kept under vacuum about 30 min, poured into a silicon mould. The prepared sample, first remained at room temperature for 2 h and then were cured for 24 h at 60°C and finally post-cured at 120°C for 3 h. At this stage the cured specimens were allowed to cool slowly in the mould at room temperature.

Characterization Methods
Infrared Spectroscopy (FTIR)
FTIR Spectroscopy was used to monitor the extent of chemical reactions of the epoxy resin/HTPB/DVB systems and the reactions which occurred when such modifiers were used. For the infrared (IR) studies a small portion of the epoxy system was attritioned on potassium bromide (KBr) pellet.

This spectroscopy method, was also used for studying the product of the reaction between DVB and HTPB that was separated from epoxy resin as powder. For the measurements, a small portion of the HTPB/DVB powder was ground to a fine powder, mixed with KBr powder and pressed into a pellet. FTIR Spectra were recorded on an Equinox model 55 spectrometer (Bruker Co., Germany).

Dynamic Mechanical Thermal Analysis (DMTA)
The glass transition temperature (T_g) of cured and modified DGEBA resins with various DVB contents were evaluated at a heating rate of 10°C/min and a bending mode using a PL dynamic-mechanical thermal analyzer (Polymer Laboratories Co., England).

Mechanical Properties Evaluation
Tensile Properties
The load-elongation curve was obtained using an axial extensometer coupled to an Mechanical Tester with a 10 kN load cell and a cross-head speed of 2 mm/min. All tests were performed at room temperature according to ASTM D638 method with an MTS tensile machine model 10/M (MTS Co., America). Using the load-elongation curves tensile strength and modulus of elasticity with different DVB contents were obtained (the tensile modulus is taken at 0.2% extension).

Izod Impact Strength
Izod impact strength was evaluated according to ASTM D256 using a standard notched and un-notched specimen. Tests were performed on a Zwick Model 5102 impact tester type machine equipped with a hammer (Zwick Co., Germany). Six samples from each formulation were broken. Average impact strength values were reported and their Izod values are calculated as follows:

\[
IS = \frac{U_1 - U_2}{(W-a)t} \quad (kJ/\text{m}^2)
\]

where \(U_1\): the impact energy (kJ), \(U_2\): the residual energy (kJ), \(W\): the specimen width (m), \(t\): the specimen thickness (m), and \(a\): the notch length (m).

Critical Stress Intensity Factor (KIC)
The fracture toughness was measured in terms of \(K_{IC}\). Fracture toughness values were determined with un-notched specimens in three-point bending with a
span of 50 mm (ASTM D790). These tests were performed with an Instron model 6025 (Instron Co., England) at a cross-head speed of 2 mm/min and a 1 kN load cell at room temperature. KIC was determined according to ASTM E399, with the following relationship [24]:

\[ K_{IC} = \frac{3PSa^{1/2}Y}{2tw^2} \]

where P: the critical load for crack propagation (N), S: the length of the span (mm), a: the specimen length (mm), t: the specimen thickness (mm), w: the specimen width (mm), and Y is a geometrical factor given by:

\[ Y = 1.93 - 3.07 \left( \frac{a}{w} \right) + 14.53 \left( \frac{a}{w} \right)^2 - 25.11 \left( \frac{a}{w} \right)^3 + 25.80 \left( \frac{a}{w} \right)^4 \]

**Morphology Analysis**

*Scanning Electron Microscopy Analysis*

Fractured surfaces of samples of epoxy resin modified with different HTPB/DVB contents were coated with a thin gold layer by BioRad, ES200 Auto Sputter Coater and examined using a scanning electron microscope, SEM (Cambridge 5360, 20KV).

**RESULTS AND DISCUSSION**

**Characterization**

*I Infrared Spectroscopy*

Series of FTIR spectra have been obtained to confirm the occurrence of any chemical interaction between epoxy resin, HTPB and DVB in first step of their reaction.

First, the cross-linking reaction between C–C double bonds of DVB with HTPB (radical polymerization), in reaction temperature about 50-60°C for 60 h, in presence of epoxy resin was run. In this step, a prepolymer of HTPB/DVB was formed, where DVB and HTPB were bound through C-C linkages as illustrated in Figure 1.

The FTIR spectra of the products formed in this step exhibited an absorption band at 1700-2000 cm⁻¹, indicating the formation of the benzene substitution. The FTIR spectrum for HTPB/DVB mixture showed the typical absorption band of C=C of benzene, thus...
confirming the above result (Figure 2).

In this work, the FTIR spectra of cured samples with hardener were also evaluated until the reaction of epoxy resin/hardener system and its probable interaction with HTPB/DVB systems were understood (Figure 3).

**Dynamic-mechanical Thermal Analysis**

The glass transition temperature ($T_g$) was obtained from DMTA thermograms of epoxy resin against HTPB/DVB content. Addition of HTPB/DVB causes a very slight decrease in the glass transition temperature which is attributed to an increase in the dissolved rubber within the epoxy matrix, but the main amount of HTPB/DVB system reduces as separating phase and therefore, it indicates a glass transition temperature separately at approximately -60°C. The pure epoxy resin indicated $T_g$ at approximately 160°C (Figure 4).

**Mechanical Properties**

**Tensile Properties**

Table 2, shows that the tensile properties of epoxy resin vary with the MHTPB content.

Rubbers are generally well known to affect tensile properties depending on their compatibility with the epoxy matrix, the surface area of contact, particle size, shape and content as well as the intrinsic strength of the rubber phase [8].

Figure 5, represents the stress versus strain curves of neat epoxy resin and modified epoxy resin (Table 1). Brittle fracture is evidenced for the neat epoxy resin, i.e. absence of yielding, whereas, the modified epoxy resins exhibit a ductile deformation with the appearance of an upper yield stress. A similar behaviour is noticed for the remaining concentration as presented in Table 2.

An increase in stress-at-break accompanied by an increase in elongation-at-break as well as the appearance of yielding are observed. As expected, the tensile modulus gradually increases with increasing HTPB/DVB content; this increase may be due to the decreasing effect of the soft segment structure of HTPB/DVB. Similar explanation holds true for the increase in the stress-at-break as a consequence of DVB addition.

In agreement with many studies [25], the tensile modulus increases with decreasing soft rubber content.

**Table 2. Effect of HTPB/DVB contents on the tensile properties of epoxy resin.**

<table>
<thead>
<tr>
<th>HTPB/DVB</th>
<th>E (GPa)</th>
<th>Stress-at-break $\varepsilon_B$ (MPa)</th>
<th>Strain-at-break $\sigma_B$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neat epoxy, BV0(0)</td>
<td>1.85</td>
<td>34.37</td>
<td>3.52</td>
</tr>
<tr>
<td>BV5(1)</td>
<td>1.91</td>
<td>35.43</td>
<td>3.51</td>
</tr>
<tr>
<td>BV5(5)</td>
<td>2.08</td>
<td>36.94</td>
<td>3.46</td>
</tr>
<tr>
<td>BV5(10)</td>
<td>2.21</td>
<td>38.96</td>
<td>3.34</td>
</tr>
<tr>
<td>BV5(15)</td>
<td>2.40</td>
<td>39.72</td>
<td>3.69</td>
</tr>
<tr>
<td>BV5(20)</td>
<td>2.42</td>
<td>41.50</td>
<td>3.81</td>
</tr>
<tr>
<td>BV5(30)</td>
<td>2.70</td>
<td>33.64</td>
<td>3.80</td>
</tr>
<tr>
<td>BV5(40)</td>
<td>2.75</td>
<td>30.43</td>
<td>2.35</td>
</tr>
</tbody>
</table>
It is interesting to note that the elastic modulus increase from 1.85 to about 2.75 GPa with increasing DVB content (Table 2). This might be related to the fact that the modulus of the HTPB/DVB system is much more than that of the epoxy phase.

**Izod Impact Strength**

Figure 6, represents the Izod impact strength of the notched and un-notched epoxy modified samples as function of DVB content (amount of HTPB has been fixed on 5 phr). The impact strength results for the materials prepared with concentrations of DVB varying from 1 to 40 phr have been summarized in Table 3. The impact strength of these materials increased with DVB content, up to 20 phr (BV5(20)), but decreased with higher DVB content. The Izod impact strength of both notched and un-notched specimens exhibited a bell-shaped curve with a maximum at 20 phr DVB content.

Notched samples showed lower values than the un-notched ones. The main reason behind this behaviour was that notches act as stress concentrators, which lead to lower impact strength. Furthermore, the sensitivity of a material to notches is also affected by the fact that fracture is a process that involves both crack initiation and propagation. In notched specimens, an apparent crack is already initiated, so the amount of energy absorbed is dependent primarily on the energy to propagate the crack. However, in un-notched specimens, the energy to initiate the crack is added onto that required for its propagation [20].

For notched and un-notched specimens, the impact strength reached maximum values of 0.71 and 6.66 kJ/m², respectively, at 20 phr before decreasing with increased DVB content. This corresponded to a twofold increase in impact strength caused by the addition of just 20 phr DVB compared to the neat resin. This further showed the beneficial effect of using DVB. In fact, at 20 phr, before increase in cross-linking between HTPB and DVB, there is a change from liquid rubber to solid state (cured rubber). After 20 phr increase in cross-linking there is a destruction of elastic behaviour. The formation of air bubbles and voids

**Table 3.** Effect of HTPB/DVB contents on the Izod impact strength of epoxy resin.

<table>
<thead>
<tr>
<th>Material code</th>
<th>Izod impact strength (kJ/m²) (notched specimens)</th>
<th>Izod impact strength (kJ/m²) (un-notched specimens)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BV0(0)</td>
<td>0.5477</td>
<td>3.7584</td>
</tr>
<tr>
<td>BV5(1)</td>
<td>0.5355</td>
<td>3.8132</td>
</tr>
<tr>
<td>BV5(5)</td>
<td>0.6259</td>
<td>4.4125</td>
</tr>
<tr>
<td>BV5(10)</td>
<td>0.6565</td>
<td>4.8096</td>
</tr>
<tr>
<td>BV5(15)</td>
<td>0.68</td>
<td>5.41</td>
</tr>
<tr>
<td>BV5(20)</td>
<td>0.7133</td>
<td>6.6641</td>
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<td>BV5(30)</td>
<td>0.6283</td>
<td>2.2426</td>
</tr>
<tr>
<td>BV5(40)</td>
<td>0.5706</td>
<td>1.4269</td>
</tr>
</tbody>
</table>

IS: impact strength
was practically unavoidable. The voids not only reduced the stress-bearing area but acted as stress raisers, thus initiating in formation of cracks.

Critical Stress Intensity Factor \( (K_{IC}) \)

Figure 7 shows the effect of DVB content on the \( K_{IC} \). A bell-shaped trend was obtained with a maximum at 20 phr (BV5(20)). At this level (20 phr of DVB), a three-fold increase in the \( K_{IC} \), from 1.35 to 2.59 MPa.m\(^{1/2}\), was observed (Table 4).

Fellahi et al. [26], reported similar results when modifying the same epoxy resin type as a matrix but used 30 phr of polyurethane with a chain extender as a modifier.

A trend similar to that for impact strength was observed, that is a maximum was reached at 20 phr (BV5(20)). This result demonstrates the observation of Izod impact strength and its results.

The appearance of a difference between these two tests might be attributed to the fact that the tests were done under different conditions. \( K_{IC} \) was realized at a

![Figure 7. Effect of DVB content on the \( K_{IC} \) of epoxy resin.](image)

**Table 4.** Effect of HTPB/DVB contents on the \( K_{IC} \).

<table>
<thead>
<tr>
<th>Material code</th>
<th>( K_{IC} ) (MPa.m(^{1/2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>BV0(0)</td>
<td>1.35</td>
</tr>
<tr>
<td>BV5(5)</td>
<td>1.6439</td>
</tr>
<tr>
<td>BV5(10)</td>
<td>1.8712</td>
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<tr>
<td>BV5(15)</td>
<td>2.3700</td>
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<td>BV5(20)</td>
<td>2.5985</td>
</tr>
<tr>
<td>BV5(30)</td>
<td>2.2727</td>
</tr>
<tr>
<td>BV5(40)</td>
<td>1.6399</td>
</tr>
</tbody>
</table>

![Figure 8. SEM micrographs of HTPB/DVB-modified epoxy resins: (a) neat epoxy resin, (b) 20% DVB/HTPB (BV5(20)) and (c) 40% DVB/HTPB (BV5/40)) at the same magnification (1500 ×), showing differences in particle size.](image)
relatively low speed, whereas the impact was performed at a high speed. In other words; when tested at a high speed (impact), the molecules would not have had time to relax or respond to this type of loading, and consequently, only a small amount of filler could be tolerated, whereas a higher filler loading could be tolerated when molecules were tested at a slow speed [27].

**Morphology and Fracturography**

The impact behaviour of the toughened networks can be explained by considering both toughening and flexibility effects. The flexibility effect is caused by the presence of dissolved rubber inside the epoxy matrix, whereas the toughening process is related to the cavitations in rubber particles dispersed inside the epoxy matrix.

In order to correlate the mechanical properties of the modified epoxy resin with the morphology, particularly, searching for a correlation between particle size of the elastomeric phase and impact strength results, the fracture surface of rubber-modified epoxy networks was analyzed by SEM. The SEM micrographs of some of the HTPB/DVB modified epoxy resin presented in Figure 8 show the large difference in rubber particle size that exists between different materials. The micrographs of epoxy resin modified with HTPB/DVB show distinct separated particles of rubber, indicating a heterogeneous system. This heterogeneous morphology resulted in opaque samples. The holes observed in the micrograph are related to the rubber particles, indicating no chemical bond between the blend components.

Materials that show maximum impact strength have an average particle diameter in the range from 2 to 5 µm.

Two factors which could be responsible for the variation in the size of elastomeric particles are: one due to the agglomeration of molecular chains of the pre-polymer, due to low compatibility with epoxy resin matrix; a second one, would be due to further polymerization of the HTPB chains through its reaction with DVB, before being linked to epoxy resin. In both cases, the formation of big particles would be favoured in a large stage.

**CONCLUSION**

A HTPB/DVB system has a potential as a toughening agent for epoxy resin, although the procedure used in this work may be imposing a limit to the amount of HTPB and DVB that can be incorporated. This limit is dependent on the compatibility between HTPB/DVB system and epoxy resin, which is affected by the ratio employed.

Based on the results obtained and discussed previously the following conclusions may be drawn: the values of rubber/DVB content have an influence on the cure temperature of these systems. The IR analysis confirmed the occurrence of a chemical reaction between HTPB and DVB and also epoxy resin leading to improved compatibility. The DMTA analysis of samples indicated different Tg for HTPB/DVB and epoxy resin in mixture, explaining phase separation between the rubber and matrix phases. This matter was confirmed by centrifugation. On adding DVB the stress-at-break increased accompanied by an increase in elongation-at-break with an approach to yielding point. The tensile modulus increases from about 1.85 to 2.75 GPa with increasing DVB content. Maximum impact strength is obtained by adding 20 phr of DVB (BV5(20)) and finally, SEM analysis showed modified rubber particles cavitation and morphology of fractured surface.

**ACKNOWLEDGEMENT**

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