Studies on Physico - Mechanical Properties of Chloroprene Rubber Vulcanizate for Belting Application

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Abstract— Among the vulcanized elastomers, the chloroprene rubber (Neoprene) possesses a good performance being one of the most used in the current days. Compounding was carried out in a two-roll mill and vulcanized at 150°C. However, this kind of polymer is seriously playing a vital role for the manufacture of power transmission belts in the automotive industry. A worldwide method that has been used and that is an important tool in the rubber vulcanization in hydraulic press curing at high temperature in which the chloroprene compound has virtual physical and mechanical properties. In this work, the chloroprene samples were prepared according to ASTM standards. The Rheological and the physico-mechanical properties of CR vulcanizate were studied.

Key words: Chloroprene Rubber, Compounding, Belting, Vulcanization

I. INTRODUCTION
Thermosetting materials such as rubbers upon processing and molding are cross linked and cannot be softened or remolding by heating process again. The three-dimensional network of the thermo set polymer system must be broken either through the cross link cleavage or through the carbon–carbon linkage of the chain backbone [1].

Commonly rubbers are used in many more applications such as tires, footwear, toys, clothing, etc. The wide use of these materials has lead to great generation of post-consumer waste. A reason for the interest in rubber recycling is that the rubber waste represents a source of significant raw material for the rubber industry [2].

Among all types of rubber the Chloroprene – CR (Neoprene – DuPont) is a prominent material. This rubber was the first market synthetic elastomer (1932) due to advantageous properties.

It is very versatile and can be cured with sulfur or organic peroxides and has the following characteristics: good elasticity, allows perspiration, resistance to oil, solvents, weather ageing, heat, oxygen, ozone and flame [3]. Vulcanized CR has excellent oil resistance [4].

The effects of carbon blacks and mineral fillers on the processing and vulcanizate properties of polychloroprene are generally similar to their effects on other elastomers. Like natural rubber and other polymers with pronounced crystallization tendencies, polychloroprene gum vulcanizates have high tensile strength [5,6]. The need for reinforcement, therefore, is less than for such elastomers as SBR, butyl rubber and nitrile rubber [7].

Oil-resistant rubbers are special rubbers such as chloroprene (CR) and acrylonitrile butadiene rubber (NBR), and polysulphide rubber [8]. Oil-resistant polymeric materials are very important for the automotive industry, since a variety of spare parts may come into contact with oils and greases. The chemical composition of rubber and namely the electro-negativity and crystallinity plays the important role in the production of oil resistant products [9].

In CR/PVC blends, the polar nature of CR and PVC enhances the oil resistance of such blends [10,11]. Omran et al. studied different rubber blends based CR and PVC and found that CR/PVC blends loaded with N990 type carbon black had excellent engine and hydraulic oils resistance [12].

The present work is aiming to elaborate high performance oil resistant rubber products based on CR loaded with N990 carbon black and also by varying the processing oil which is to be utilized in automobiles industry. The CR vulcanizate is evaluated by rheological, physical and mechanical characteristics.

II. MATERIALS AND METHODS

A. Materials
Other materials used for the preparation of the compounds were:

1) Magnesium oxide
2) Stearic acid
3) Zinc oxide
4) Calcium Carbonate
5) Carbon Black (N990)
6) Antiozonant Wax
7) Antioxidant
8) Polyethylene Wax
9) Aromatic Oil
10) Zinc Oxide
11) MBTS

All materials were commercially obtained from local market.

To study the effect of loading of carbon black with chloroprene rubber a basic formulation was prepared (Table 1), based on standards for the automotive industry. Chloroprene rubber compound was prepared in an open two-roll mixing mill with 2 kg of capacity at temperature between 50°C and 60°C. For a comparative study, the loading of carbon black (N990) and also the processing oil (aromatic oil) are fixed as the variables.

B. Methods

1) Compounding
The raw rubber such as CR (Neoprene) was first masticated on the laboratory open two roll mill (Keesa X14) for ten minutes. The required amount of MgO was incorporated with CR on a two roll mill at first. Zinc oxide, stearic acid and other rubber additives were added sequentially in masticating CR rubber.

Finally, CR rubber with magnesium oxide was mixed with the rubber compounds for another ten minutes. The mixing mill was cooled by cold water circulation during the mixing of all rubbers and rubber additives. The
compounds were prepared as per formulation given in Table 1. According to ASTM method D 3182.

<table>
<thead>
<tr>
<th>S. No</th>
<th>Additives</th>
<th>Loading, Phr Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>C1</td>
</tr>
<tr>
<td>1</td>
<td>Neoprene w</td>
<td>100</td>
</tr>
<tr>
<td>2</td>
<td>Magnesium Oxide</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>Stearic Acid</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>Calcium Carbonate</td>
<td>25</td>
</tr>
<tr>
<td>5</td>
<td>Carbon Black (N990)</td>
<td>20</td>
</tr>
<tr>
<td>6</td>
<td>Antioxidant</td>
<td>4</td>
</tr>
<tr>
<td>7</td>
<td>Polyethylene Wax</td>
<td>5</td>
</tr>
<tr>
<td>8</td>
<td>Aromatic Oil</td>
<td>15</td>
</tr>
<tr>
<td>9</td>
<td>Zinc Oxide</td>
<td>6</td>
</tr>
<tr>
<td>10</td>
<td>TMTD</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 1: Base formulation of CR.

### Rheo properties of CR vulcanizate

<table>
<thead>
<tr>
<th>Rheo Properties @ 180°C</th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
<th>C5</th>
</tr>
</thead>
<tbody>
<tr>
<td>ML (dNm)</td>
<td>0.57</td>
<td>0.63</td>
<td>0.95</td>
<td>0.86</td>
<td>0.78</td>
</tr>
<tr>
<td>MH (dNm)</td>
<td>1.73</td>
<td>1.94</td>
<td>2.97</td>
<td>2.43</td>
<td>2.38</td>
</tr>
<tr>
<td>t50 (mint)</td>
<td>18.34</td>
<td>18.89</td>
<td>19.47</td>
<td>18.91</td>
<td>17.80</td>
</tr>
<tr>
<td>t52 (mint)</td>
<td>2.79</td>
<td>3.46</td>
<td>4.76</td>
<td>6.78</td>
<td>8.48</td>
</tr>
<tr>
<td>CRI</td>
<td>5.74</td>
<td>6.38</td>
<td>7.54</td>
<td>8.68</td>
<td>9.37</td>
</tr>
</tbody>
</table>

Table 2: Rheo properties of CR vulcanizate

2) Curing

The aromatic characteristics, the minimum torque (ML), maximum torque (MH), delta torque (ΔH), Cure rate index (CRI), the cure time (t90) and scorch time (t52) were determined using a Monsanto Moving Die Rheometer (MDR 2000) according to ASTM method D 2084. Samples of about 10 g of the respective compounds were tested at a vulcanization temperature of 150°C. The cure rate index is a parameter which indicates the speed of curing reaction was determined from rheometric data. CRI is calculated by using the following relation[4].

Cure rate Index (CRI) = 100 / cure time- scorch time (1.1)

### Vulcanization Process

Sheet of 3 mm thickness was compressed and molded at 150°C ± 5°C with 10 MPa force by using a hydraulic press at the respective cure time (t,90) determined with the MDR 2000.

### Characterization of CR Vulcanizate

1) Tensile Strength and Elongation at break:

Tensile strength (TS) and percentage of elongation at break (% EB) were carried out in a tensile testing machine (ZWICK load tester 4301) according to ASTM D 412 test method using dumb-bell shaped test specimens at a uniform speed 10 mm/sec[13]. The sample failure properties were calculated by using the following formulation.

Tensile Strength, Kg/cm² = Load failure / Area of Cross section of the specimen (1.2)

2) Hardness

The hardness of the Chloroprene vulcanizate was measured as per ASTM D 2240 test method. Reported hardness values are the average of reading taken at five different locations on each sample at room temperature. It is expressed in terms of IRHD units.

### Abrasion Loss

Abrasion test was conducted using an ASTM D5963 test method. The abrasion resistance has been expressed as relative volume loss in cubic millimeter. The test was conducted in a DIN abrader.

### RESULTS AND DISCUSSION

The effect of loading of N990 and the loading of processing oil on the CR vulcanizate results were discussed here. Various Physical and Mechanical properties data of Chloroprene vulcanizate were described in Table 3: ASTM Method B, Hot press curing @150°C, 20 minutes.

<table>
<thead>
<tr>
<th>S. No</th>
<th>Mechanical Properties of CR Vulcanizate</th>
<th>Compounds</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>C1</td>
</tr>
<tr>
<td>1</td>
<td>200% Modulus, MPa</td>
<td>3.24</td>
</tr>
<tr>
<td>2</td>
<td>Tensile Strength, MPa</td>
<td>15.61</td>
</tr>
<tr>
<td>3</td>
<td>Elongation, %</td>
<td>710</td>
</tr>
<tr>
<td>4</td>
<td>Hardness, IRHD</td>
<td>54</td>
</tr>
<tr>
<td>5</td>
<td>Compression Set, %</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>DIN Abrasion Loss, mm³</td>
<td>140</td>
</tr>
</tbody>
</table>

Table 3: Mechanical Properties of CR
and ozone conditions, dynamic stress such as belting applications to transmit the power at the drastic conditions.

C. Effect of carbon black (N990) on compression set of CR vulcanizate

The result of compression set of CR vulcanizate is shown in Fig. 4. Compression set is a measure of the ability of rubber vulcanizates to retain their elastic properties after prolonged compression at constant strain under a specific set of condition and its permanent set of rubber vulcanizes. The poor performance of rubber compounds in terms of compression set is attributed to uncross-linked chains. Therefore it does not contribute to the permanent network and are able to relax during the compression stage. The results of N990 filled CR vulcanizate show no significant variation in their results. It is due to the curing or crosslink or effective network chain in the deformed state. The lower values of compounds indicate the best material for the compression state. Among the C1-C5 compounds of CR, the compression set values are obtained in the range of 30% - 34%. However, the compound C5 showed the lower compression set value whereas the C2 exhibited the value is 34% corresponding to the content of carbon black and processing oil at the level of 30 Pph and 20 Pph respectively. Also C2 showed the poor mechanical and rheological properties when the compound is analyzed at its service temperature. Compression set property of rubber compounds commonly plays an important role in the application of hoses and tubing particularly in oil resistance applications. Therefore it is more prominent to study the mechanical properties of the various Chloroprene rubber based compounds (C1-C5) by varying the black fillers and processing oils.

D. Effect of processing oil on Rheological Properties of CR Vulcanizate

From the previous studies it was found that the incorporation of aromatic oil in virgin rubber matrix and rubber blends the oil molecules must penetrate into the rubber matrix and go around the CR rubber compound which is in semisolid phase [16]. This phenomenon urged us to study the rheological properties such as cure rate index, tS2, t90, ML and MH in CR rubber vulcanize. The various rheological properties of CR compound were studied at 180°C and their results are showed in Table 2. Fig.5. shows that the cure rate index of C1 to C5 compounds is linearly increased to the various loading of processing oil. C5 exhibits the highest CRI value as 9.37 helped us to get an idea about the curing characteristics of CR vulcanizate at 180°C. Similarly the compounds C1, C2 and C3 showed the tS2, t90, ML and MH are in an increasing order unto the
addition of 25 Phr of aromatic oil. This illustration describes how the dispersion of the carbon black particles into the chloroprene rubber matrix phase could exhibit the significant rheological properties at its processing temperature. The dispersed CR phase could lead to a very-tortuous path for oil molecules travelling through the rubber matrix. This behavior is similar to that between flow and mechanical properties of two-phase system (Rubber matrix-processing oil) and the permeation models of chloroprene rubber composite materials.

The experimental results showed that the rheological properties of the evaluated rubber compounds after the substitution of conventionally used aromatic oils have not been deteriorated significantly. The CRI values indicate that major processing characteristics of the compounds remained almost unchanged by using the same process oils at various quantities. The use of ecofriendly process oil causes small variations in the curing behavior of CR vulcanizate.

From the results obtained, the followings are concluded.

(i) In N990 filled CR vulcanize, the physico-mechanical properties and the rheological properties such as tensile strength, hardness, elongation at break, abrasion loss, scorch time, optimum cure time and cure rate index were nicely discussed. (ii) The cure characteristics and mechanical properties of N990 filled CR compounds mainly depend on the content of carbon black particles and the processing oils. (iii) The values show the opposite trend with increasing the carbon black content in CR compounds. (iv) The minimum torque and maximum torque of the CR compound also improved the mechanical properties. (v) The filler to processing oil ratio did not much influence on compression set properties. (vi) Accelerated aging behavior of N990 filled CR compound also improved the mechanical properties. (vii) The study shows that the CR based C4 and C5 compounds are particularly recommended for use in making driving belts, timing belts or conveyor belts because of their excellent mechanical properties.

IV. CONCLUSIONS

REFERENCES


