Application of recycled rubber powder (RRP) in NR / SBR compounds

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This study presents tire recycling process to produce rubber powder (particle size, 0.1-1.00 mm), and explores use of recycled rubber powder in NR / SBR compounds as substitution for black fillers (soot). A number of tests and measurements of rubber compound properties were used to find the best ratio of soot and rubber powder. Guidelines to what percentage of recycled rubber powder should be added in a compound without affecting mechanical properties of finished rubber products are also provided.

Keywords: Fillers, Mechanical properties, Recycled rubber powders, Rubber

Introduction
Polymeric materials, including tires, account for only 10% of total waste, but their disposal as landfills occupy much space due to low density and high voluminosity1-6. A number of studies are available on recycling processes of waste tires7-10. Among tires collected for recycling, 76% are processed into shreds, granules and powders1-3. Recycling of waste tires into powder requires considerable economic and technical expenses, because tires are made from multi-layered materials, which require a clean separation of each component by type3-6. This study presents a suitable process for using recycled tires in powder form.

Experimental Section
Materials
Recycled rubber powder (RRP) was produced from tires (age, 3 y old; particle size, 500 µm; fiber, 0.5%; steel, 0.1%) using ground recycling process. Black filler or N-220 was replaced with RRP (5, 15 and 40%). Characteristics of NR / SBR compounds (0% RRP) were compared with characteristics of other compounds (Table 1).

Preparation of NR / SBR Compounds (Mixing and Vulcanization Parameters)
Compounds were mixed in a laboratory-size mixer with a rotor speed of 50 rpm at 60°C for 6 min. After discharging the mixer, compounds were mixed on a two-roll mill, and 8 end-roll passes were made before eventually being drawn into sheets. Time and length of curing process was determined by Monsanto Rheometer 100S according to ASTM D 2240-93, with vulcanization time of 10 min and vulcanization temperature of 150°C.

Testing Mechanical Properties
All measurements were carried out before and after aging, which was conducted in aging oven for 7 days at 100°C. Hardness measurements (5 for each sample) were performed in accordance with ISO 7691-1, using a manual durometer type Shore A. Testing of wear resistance was performed in accordance with ISO 4649 using a Shopper cylindrical device with 5 measurements per sample. Tensile strength was determined in accordance with ISO 37 at the measuring point on dumbbell specimen type 2 (thickness 3 mm). Tear resistance was analyzed in accordance with ISO 34. Three angular type A tubes (thickness 3 mm) were strained to break in measuring point, where speed of clamp separation was constant at 350 mm / min. Testing equipments both for tensile strength and tear resistance were universal testing machine, AD-converter HBM Spider 8, displacement transducer HBM WK 200, force transducer HBM U9B (5kN) and software HBM Easycatman AP for data processing (sample rate 800Hz). Clamp separation speed was 350 mm / min2,5,7,10-14. Dumbbell specimens were elongated to 100% and left in the aging oven for 7 days at 70°C in accordance with ISO 37.
Results and Discussion

For each test sample, five test samples were made and five measurements were conducted, after which measurement results were averaged. Hardness of all vulcanizates decreases (Fig. 1) with increase in RRP, with only a negligible decrease between 0-15% of RRP. However, hardness decreases more significantly when RRP reaches 40%, resulting in 9% of initial hardness, may be due to decrease in cross-link density when RRP filler is used. After aging, hardness decreases significantly with increase in RRP.

Tensile properties fall into one of the most important properties of rubber compounds (tensile strength and modulus of elasticity). Tensile strength (Fig. 2) decreases slightly (5-15%) with increase in RRP. Tensile strength decreases significantly when amount of filler reaches 40% \(^{12,16-18}\), may be due to weak bonds between particles and polymer chains, as well as irregular networking, which leads to a decrease in tensile strength. With increase in RRP, possibility of improper particle dispersion also increases, causing more frequent occurrence of errors in the material. These results comply with reported results\(^{12}\).

As expected, NR / SBR compounds (RRP, 5-15%) give highest modulus of elasticity (Fig. 3), while this value decreases significantly for compounds with 40% of RRP. Thus RRP behaves like a rigid filler since it has a higher modulus of elasticity than natural rubber matrix, which is fully in accordance with reported results\(^{12,15,20}\).

RRP has no effect on wear resistance either before or after aging (Fig. 4). Residual elongation after unloading

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>NR/SBR compounds with different RRP</th>
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<tbody>
<tr>
<td>g</td>
<td>0%  5%  15%  40%</td>
</tr>
<tr>
<td>SMR-10</td>
<td>225  225  225  225</td>
</tr>
<tr>
<td>SKS-30</td>
<td>870  870  870  870</td>
</tr>
<tr>
<td>ZnO</td>
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<tr>
<td>Stearin</td>
<td>15   15   15   15</td>
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<tr>
<td>4010 Na</td>
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<tr>
<td>TMQ</td>
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<tr>
<td>SOLAR-3</td>
<td>70   70   70   70</td>
</tr>
<tr>
<td>N-220</td>
<td>580  550  490  350</td>
</tr>
<tr>
<td>CBS</td>
<td>14.6 14.6 14.6 14.6</td>
</tr>
<tr>
<td>MS</td>
<td>1.0  1.0  1.0  1.0</td>
</tr>
<tr>
<td>S</td>
<td>24.2 24.2 24.2 24.2</td>
</tr>
<tr>
<td>PVi</td>
<td>1.8  1.8  1.8  1.8</td>
</tr>
<tr>
<td>RRP</td>
<td>0    30   90   230</td>
</tr>
</tbody>
</table>

Fig. 1—Hardness of rubber sample

Fig. 2—Stress-strain diagram of NR / SBR compounds

Fig. 3—RRP impact on elastic moduli of NR / SBR compound

Fig. 4—Wear resistance test samples
increases with increase in RRP (Fig. 5), as well as breaking elongation ((Fig. 2), may be due to poor cross-linking between RRP particles and NR / SBR compounds, which leads to weakened bonds, as well as decrease in the ability of rubber compounds to absorb stresses. This is also fully in accordance with reported results\textsuperscript{12,15-19}.

A significant decrease in tearing force was recorded after adding 5% of RRP (Fig. 6), which is followed by a slight decrease with increase in RRP\textsuperscript{12-22}, may be due to poor distribution of particles in cross-linked chains of RRP and varying particle size, which negatively affect resistance to fragmentation. These results comply with reported results\textsuperscript{14,16-19}.

Since properties degraded, especially tear resistance, the structure was examined using an optical microscope \textit{KONUS DIAMOND 5420} magnification x60, power 10W. Before examination, samples were immersed in \textit{N}_2, after which they were broken so as to observe the structure. As assumed, structural non-homogeneity appeared due to mixing RRP into original mixture. With increase in RRP, large structure flaws appeared (Fig. 7d), which resulted due to grouping of RRP particles and their unequal dispersion in the mixture.
Conclusions

Use of RRP in NR / SBR compound depends on required characteristics of rubber products. Hardness can be achieved by replacing black fillers (soot) by a maximal amount of RRP (15%). As far as tensile strength and permanent elongation are concerned, black fillers can also be replaced by RRP (up to 15%), where no significant deviation occurs. Wear resistance shows slight sensitivity to application of RRP. The best properties are achieved by applying 5% of RRP, yet optimal ratio from economic and technical analysis is 15% of RRP. However, for products requiring tear resistance, use of RRP is not recommended since it leads to significant reduction of tearing forces even at values lower than 5% of RRP.

References