STUDY ON THE PREPARATION OF RUBBER COMPOSITE FROM WASTE TYRE, RUBBER AND NEUTRALIZED WASTE LEATHER PARTICLES

Ko Ko Aung¹, Khin Aye May², Kyaw Myo Naing³

Abstract

Scrap tyre and waste leather products are major environmental problem for disposal. To overcome this problem, it is needed to reuse into another purpose. Therefore in this research, waste tyre and waste leather were used as filler in rubber compounding which also reduced the cost of production. Waste tyre and waste chrome tanning leather sample (neutralized with ammonia) were collected from tyre shop and leather mill. In this research 6 types of rubber composites RLC I – VI were made by using different amounts of waste tyre and fixed amount of waste leather. All these composites were investigated their physicochemical and physicomechanical properties. In the series of RLC I – VI, RLC VI (100 phr of NR, 100 phr of waste chrome leather neutralized with NH₃ and 600 phr of waste tyre) had the highest tensile strength 6.63 MPa, elongation at break 232 %, tear strength 62.0 N/mm and 100 % modulus 6.63 %. RLC VI was more resist to strain and stress. Among the prepared flooring RLC VI was selected due to its highest 100% modulus which resists the pressure and the neutralizing agent (NH₃) used in leather waste did not cause adverse effect on environment. The comparison of surface morphology and thermal resistance of RLC VI and commercial flooring was performed. The surface of RLC VI was smoother than that of commercial flooring. Thermal resistances of both were nearly the same. Weight loss of RLC VI was 65.86 % and commercial was 71.896 %. The cost of prepared flooring was 2710 kyats per 4’×7’ sheet while the commercial was 7500 kyats per sheet. Therefore this prepared rubber composite flooring can be substituted in commercial flooring.

Keywords: waste tyre, waste leather, composites, TG-DTA, SEM, Flooring

Introduction

Mankind has been aware composite materials since several hundred years before Christ and applied innovation to improve the quality of life. Although it is not clear has to how man understood the fact that mud bricks made sturdier houses if lined with straw, used them to make buildings that lasted (Wang, 2011).

Composites that forms heterogeneous structures which meet the requirements of specific design and function, imbued with desired properties which limit the scope for classification. However, this lapse is made up for, by the fact new types of composites are being innovated all the time, each with their own specific purpose like the filled, flake, particulate and laminar composites (Anderson, 2012). Another reason for used as composites materials were high resistance to fatigue and have been high corrosion degradation, high strength or utility ratio (Miracle and Donaldson, 2000).

Materials and Methods

All chemicals used in this research were procured from British Drug House (BDH), England. The chemicals were used as received unless state otherwise. All specific chemicals used were cited detail in each experimental section. The apparatus consist of conventional lab wares, glass wares and modern equipment.

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Sampling

Representative rubber smoked sheets were purchased from local markets. Scrap tyre and waste chrome tanning leather were collected from Hlaing Tha Yar Industrial zone, Yangon Region.

Preparation of Scrap Rubber

Vehicle tyre (no more used) was cut with different types of cutting machines to obtain rubber belts. Then, belts were chipped with chipping machine. The rubber chips were ground in the grinding mill to obtain scrap rubber powder.

Preparation of Neutralized Leather Particles

Waste chrome leather shavings, obtained from a local industry, contain chromium (2.5 %) and nitrogen (11.21 %). The untreated leather fibers were dried at 100 °C for 15 min in an air oven and after cooling to ambient temperature, they were shredded into fine particles. The untreated leather particles were acidic in nature which would interfere with the vulcanization of rubber compound (Langmaier et al., 2005). To overcome the acidic nature ammonia was used for neutralizing agent because it was available in local markets and do not affect for environment. After neutralization, the leather particles were separated by filtration and the excess water was removed. The resultant cake was dried in sunlight for 2 days followed by drying in an oven at 100 °C for 15 min (Buljan and Karal, 2011).

Preparation of Rubber Leather Composite

Compounding was done on a 100 phr of natural rubber, 100 phr of neutralized leather particles and 100 to 600 phr of 40 mesh size scrap rubbers. Composite series I to VI are rubber leather composite (RLC) using waste leather neutralized with NH₃. Natural rubber was masticated and after a smooth band was formed on the mill, it was mixed with premix waste leather tyre composites. Softening agent such as aromatic process oil was added to the mixture followed by the addition of antioxidant santoflex 13 (2 phr), zinc oxide and steric acid as activator. It was rolled with roller mill at 60°C to complete mixing. After complete mixing and band formation were ensure, accelerator dibenzothiazlisdisulpide (MBTS), tetra methyl thiuramdisulphite (TMTD), N- (1,3 dimethyl) –N- phenyl –p-phenylenediamane (6 PPD) and sulphur were added. Appropriate nip gaps were maintained and 3/4th cuts were made during the mixing process in order to get uniform compound quality. The schematic diagram for preparation of rubber leather composite is shown in Figure 1 (Ravichandran and Natchimuthu, 2010).
**Figure 1** Schematic diagram for preparation of rubber leather composite

**Determination of Physicomechanical Properties**

**Determination of tensile strength and elongation at break**

The prepared composite sheets were cut off according to JISK 7127 and the shape and the dimension of the test pieces were prepared. Both ends of the test pieces were firmly clamped in the jaw of tensile strength testing machine. One jaw was fixed and other was movable. The movable jaw moved at the rate of 10 mm/min. the resultant data was recorded on the recorder. This procedure was repeated three times for each composite. The calculation of tensile strength and elongation at break are described in Table 1.

**Determination of 100 % modulus**

The procedure was the same as procedure for tensile strength and the results are shown in Tables and Figures.

**Determination of tear strength**

Specimen was cut with single nick (0.05 mm) at the entire of the inner concave edge by a special cutting device using a razor blade. The clamping of the specimen in the jaw of test machine is aligned with travel direction of the grip at the rate of 100 mm/min. The recorder of the machine showed the highest force to tear from a specimen nicked. Tear strength was calculated and described in Table 1. The procedure was repeated three times for each result.
Determination of hardness

The test pieces were placed on the table of instrument. The operating wheel was turned by hand to lower a flat ended circular foot onto the surface of the test piece. After 5 s, the weight of the instrument was pre-set to apply the correct force to foot.

The operating wheel was turned further to apply a known contact force onto foot, followed by a known test force. Hardness is based upon the indentation caused by the test forced. After 30 s, the hardness was directly measured in IRHD on the micrometer gauge. This experiment was repeatedly three times for each composite. The hardness was calculated and described in Table 1 (ASTM, 2000).

Physicochemical Characterization of the Prepared Composites
Determination of swelling percent of the prepared composites

The test piece with uniform thickness and volume were used. For the determination of swelling, the test pieces were cut as possible as the same size and weighed. Each piece was placed in each of screw-tight metal capped test bottles (100 mL) containing 50 mL of the selected solvents such as ethanol, gasoline, diesel, engine oil and used engine oil at room temperature. The test piece was taken off from the bottle and blotted with filter paper to remove any adhering oil on sample surface and weighed the sample. The weight gains were measured at 3rd, 6th and 9th days. The swelling percent was calculated and described in Table 2.

Furthermore the prepared composite RLC V and commercial floor were investigated by SEM for surface morphology and TG DTA. All of these techniques were performed at Universities’ Research Center, Yangon.

Results and Discussion

The focus of this research was to investigate the effect of filler percent on rubber compounding. The investigation was found on different amounts of filler percent change the physicomechanical and physicochemical properties of composites. To compare the mechanical properties, rubber leather composites were also prepared by using different amount of scrap tyre as filler. The comparisons of physicomechanical properties of 6 types of rubber-leather composites (RLC) were also performed. From the experimental data, composites obtained from scrap tyre as filler showed good oil resistance and heat resistance. Moreover, based on these comparisons, the effective usages of these composites in flooring were also investigated.

Physicomechanical Properties of RLC I to VI

RLC I to RLC VI were prepared by using 100 phr of natural rubber, different amounts (100 – 600 phr) of scrap tyre particle and fixed amount (100 phr) of waste leather (neutralized with NH3). According to physicomechanical properties RLC VI has the highest tensile strength (6.63 MPa) and elongation at break percent was 232. Thus, it is more resist to strain and stress. Furthermore RLC VI was good 100 % modulus (6.43 %) and the most favorable condition tear strength (62.0 N/mm) (Figure 2).
Table 1 Physicomechanical Properties of Prepared RLC I - VI Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Composition (phr)</th>
<th>Tensile strength (MPa)</th>
<th>Elongation at break (%)</th>
<th>100 % Modulus</th>
<th>Tear strength (N/mm)</th>
<th>Hardness (IRHD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RLC-I</td>
<td>Leather 100, NR 100, Waste tyre 100</td>
<td>5.30</td>
<td>135</td>
<td>4.89</td>
<td>26.9</td>
<td>76</td>
</tr>
<tr>
<td>RLC-II</td>
<td>Leather 100, NR 100, Waste tyre 200</td>
<td>5.68</td>
<td>156</td>
<td>5.19</td>
<td>34.8</td>
<td>72</td>
</tr>
<tr>
<td>RLC-III</td>
<td>Leather 100, NR 100, Waste tyre 300</td>
<td>5.94</td>
<td>179</td>
<td>5.51</td>
<td>43.9</td>
<td>67</td>
</tr>
<tr>
<td>RLC-IV</td>
<td>Leather 100, NR 100, Waste tyre 400</td>
<td>6.21</td>
<td>200</td>
<td>6.01</td>
<td>52.7</td>
<td>61</td>
</tr>
<tr>
<td>RLC-V</td>
<td>Leather 100, NR 100, Waste tyre 500</td>
<td>6.57</td>
<td>225</td>
<td>6.35</td>
<td>61.5</td>
<td>57</td>
</tr>
<tr>
<td>RLC-VI</td>
<td>Leather 100, NR 100, Waste tyre 600</td>
<td>6.63</td>
<td>232</td>
<td>6.43</td>
<td>62.0</td>
<td>56</td>
</tr>
</tbody>
</table>

* Leather neutralized with NH₃

NR = Natural Rubber
RLC = Rubber leather composite

Natural rubber, NH₃ neutralized leather and 100 phr waste tyre composites for RLC I and II, III, IV, V and VI for 200, …, 600 respectively

Figure 2 (a) Tensile strength (b) Elongation at break (c) 100 % modulus (d) Tear strength (e) Hardness of prepared composites RLC I to VI versus amount of waste tyre particle
Physicochemical Properties of RLC I to VI

Swelling percentage

For determining the swelling properties, the ethanol, gasoline, diesel, engine oil and used engine oil. Previously weighed sample RLC-VI was soaked in above solvents separately. After soaking for 3 days it was weighed and continued soaking for 6 days and 9 days. Then it was weighed again and again. Finally swelling percents were calculated.

Table 2 shows the results for swelling of prepared rubber composite in selected solvents. This can be attributed to the highly rigid cross-linked polymer nature of composites and non-polar nature of ethanol.

During 9 days of swelling duration, all the composites can absorb the ethanol and the test pieces were swelled. The test pieces become saturated with selected oil that were no increasing in weight after 6 days.

The swelling percent shows, that it can resist spoiling the given solvent on the floor. The swelling percents of these composites after 9 days of duration are also presented in Table 2.

Table 2  Swelling Percent of RLC VI in Different Solvents

<table>
<thead>
<tr>
<th>Solvents used</th>
<th>Weight (before swelling) (g)</th>
<th>Weight (after swelling) (g)</th>
<th>Swelling percent (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3 day</td>
<td>6 day</td>
<td>9 day</td>
</tr>
<tr>
<td>Ethanol</td>
<td>2.01</td>
<td>2.03</td>
<td>2.04</td>
</tr>
<tr>
<td>Gasoline</td>
<td>2.02</td>
<td>2.21</td>
<td>2.37</td>
</tr>
<tr>
<td>Diesel</td>
<td>2.01</td>
<td>2.25</td>
<td>2.44</td>
</tr>
<tr>
<td>Engine Oil</td>
<td>2.03</td>
<td>2.15</td>
<td>2.25</td>
</tr>
<tr>
<td>Used Engine Oil</td>
<td>2.01</td>
<td>2.10</td>
<td>2.12</td>
</tr>
</tbody>
</table>

Comparison between TG-DTA thermogram of prepared and commercial floor

The weight lost percent of commercial floor (71.896 %) was greater than prepared floor (65.954 %). Moreover the degradation temperature of commercial floor was 550 °C and the prepared composite was 580 °C. It was shown that the commercial floor is less heat resistance than the prepared floor (Figure 3). Therefore the prepared composite resisted to cigarette and mosquito coil burn.
Figure 3  TG-DTA thermogram of (a) prepared floor and (b) commercial floor

Comparison of SEM image between prepared floor and commercial floor

Figure 4 show the surface image of prepared floor and commercial floor composite. The particle size of prepared floor is smaller than commercial floor. 90% of particle size was less than 10μm in prepared floor and in the commercial floor it was has 10μm. The surface of commercial floor composite was found to have has smooth texture particles and ingredients used in the compound were dispersed homogeneously.

Figure 4  SEM images for (a) prepared floor and (b) commercial floor

Table 3 Estimate Cost for Rubber Floor (for 100 sheets of 4’×7’ size)

<table>
<thead>
<tr>
<th></th>
<th>For 2800 sqft</th>
<th>Kyats</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw materials</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(i) Natural rubber (200 lb x 1200)</td>
<td></td>
<td>240000</td>
</tr>
<tr>
<td>(ii) waste leather (200 lb x 15)</td>
<td></td>
<td>3000</td>
</tr>
<tr>
<td>(iii) waste tyre (1000 lb x 3)</td>
<td></td>
<td>3000</td>
</tr>
<tr>
<td>Chemicals</td>
<td></td>
<td>9000</td>
</tr>
<tr>
<td>Electrical cost</td>
<td></td>
<td>1000</td>
</tr>
<tr>
<td>Labor cost (3000 x 5)</td>
<td></td>
<td>15000</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td></td>
<td><strong>271000</strong></td>
</tr>
</tbody>
</table>

Table 3 shows the estimated cost for 1 rubber sheet is 2710 Kyats. In market the cost of commercial floor (4’×7’) sheet is 7500 Kyats. Therefore prepared floor composite is 4790 Kyats saved than commercial floor.
Conclusion

From the overall research, the following conclusion could be drawn.

The physicomechanical properties, such as tensile strength, elongation at break, 100% modulus, tear strength and hardness of 6 composites and physicochemical method (swelling) were examined. Mechanical properties are those physical properties that related to strength, toughness and durability. This RLC-VI has moderately hardness 56 IRHD. Hardness of flooring is the resistance of that flooring to deform under an applied load. RLC-VI has good tensile strength 6.63 MPa. The 100% modulus of RLC-VI has 6.43%. If the low modulus (soft) the floor may be good strain, stress or elongation and if the high modulus (hard) the floor may be good stress, load or tensile force. In elongation at break for RLC-VI has 232%. RLC-VI has high tear strength 62.0 N/mm. So all composite can be used for flooring. For floor decoration, the swelling property of RLC-VI was determined with different solvents such as ethanol, gasoline, diesel, engine oil and used engine oil for rigid cross link polymer nature. Modern techniques were comparatively studied with prepared flooring and commercial flooring. The prepared flooring cost 2710 kyats per (4’ × 7’) sheet which is found to be more cost effective than the price of commercial flooring 7500 kyats.

Acknowledgement

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