Preparation and Characterization of Rubber Composites using Sawdust as Filler

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ABSTRACT

The research work aims to prepare, characterize and apply the vulcanized rubber composites by using sawdust as filler. Sawdust was used as filler in the process of preparation of vulcanized rubber composites. The composite of natural rubber-sawdust (NR-SD) was prepared by being replaced the percent ratio (100:0; 75:25; 50:50; and 25:75 parts/weight) of natural rubber (NR) with SD vulcanized for rubbery goods. The surface-morphology, elemental analysis and thermal degradation of the prepared NR-SD composites were determined by SEM, EDXRF and TG-DTA, respectively.

2. Proposed Methods

2.1. Preparation of Sawdust

Sawdust samples were washed with distilled water to remove dust and impurities for optimization of the process. Then, it was dried for 8 hr at 100 °C and cooled in a desiccator. This sawdust was heated for 3 hr at 700 °C. The sawdust sample was sieved to 0.9 - 1.7 mm by using respective British Standard Sieves. The separated sawdust sample was stored in airtight container.

2.2. Preparation of NR-SD Composites

The vulcanization of NR-SD composites was carried out for efficient vulcanization system. Natural rubber smoked sheet and reclaimed rubber were first rolled about 5 min by a roller to break out the fibrous bond of rubber polymer chain. This process is called mastication. The mercaptobenzothiazole (MBT) was added and rolled about 30 min. Stearic acid, zinc oxide were added simultaneously and continuously rolled about 4 min. Then PCC (Precipitated Calcium Carbonate) and clay were added in order to make the vulcanite harder and to develop resistant. It was then rolled continuously for about 10 min with sulphur to obtain a two millimetre thickness sheet. The total mixing time was approximately 20 min. During the mixing, water was passed through the roller to control the generated heat. The flow diagram of preparation of NR-SD composites are shown in Figure (2.1).

Keywords: Sawdust, Composite, Physicomechanical.
2.3. Physic mechanical Measurements of Prepared NR, NR-SD

2.3.1. Determination of Hardness
The test piece was placed on the hardness tester. The operating wheel was turned by hand to lower a flat ended circular foot onto the surface of the test piece. After 5 seconds, the weight of the hardness tester was pre-set to apply the correct force to foot. The operating wheel was further to apply a known contact face onto the foot, followed by a known test force. Hardness is based upon the indentation caused by the test forced. After 30 seconds, the hardness was directly measured in IRHD on the micrometer gauge.

2.3.2. Determination of Specific Gravity
The test piece was suspended on a needle form an arm at one end of the beam, which was zeroed by means of quickly adjustable sliding weights. The test pieces were then immersed in water contained in a glass beaker locked on a friction-clamped platform. This platform can be raised and lowered easily and remained in position without an additional clamping. When the test piece was immerged, the specific gravity was calculated.

2.3.3. Determination of Tensile Strength and Elongation at Break
The prepared NR-SD composite sheets were cut off according to JISK 7127 and the shape and the dimension of test pieces were described. The 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 were shown at the recorder. This procedure was repeated for three times for each result.

2.3.4. Determination of Tear Strength
The specimen to be tested was cut only by the die from the above sheets. Specimen was cut with a single nick (0.05mm) at the entire of the inner concave edge by a special cutting device using 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 can be calculated. The procedure was repeated three times for each result.

2.3.5. Determination of Abrasion Resistance
The flat end of a cylindrical test piece was abraded against the surface of a rotating drum covered with an abrasive cloth, while the test piece was traversed from one end of the drum to the other to reduce contamination of the cloth. The abrasion resistance can be calculated.

2.4. Energy Dispersive X-Ray Fluorescence (EDXRF) Analysis of Prepared NR, NR-SD
The chemical constituents of the prepared NR, NR-SD composites were detected by using energy dispersive X-Ray fluorescence. The procedure was followed according to the catalogue.

2.5. Scanning Electron Microscopic (SEM) Analysis of Prepared NR, NR-SD
The instrument used in setting the specimen onto the brass stubs was coated. The carbon double tape was covered on the brass stubs and the sample was placed onto the covered double tape. The stubs were inserted into the ion sputter for platinum coating on the sample. Then, the platinum-coating stub was placed in the sample holder and put into the scanning electron microscope.

2.6. Determination of Swelling Percent of Prepared NR, NR-SD
The test piece with uniform thickness and volume was used. The test pieces were weighted to the nearest milligram and then the initial weight of all pieces were nearly the same before swelling.

Each piece was placed in each of screw-tight metal capped test bottles (100mL) 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 weighted the sample. The weight gains were measured during 3, 6 and 9 days.

2.7. Thermogravimetry-Differential Thermal Analysis (TG-DTA) of Prepared NR, SD25
Thermogravimetric analyses of the prepared composites were performed by thermogravimetric analyzer with argon atmosphere.

3. Results and Discussion
The focus of this research was to investigate the effect of filler on rubber compounding. The investigation was found on different parts of filler in the rubber compounding. The comparisons of the physicomechanical properties of NR-SD composites were also performed. Moreover, the effective usage of NR-SD composites in rubber goods were also investigated.

3.1. The Role of Sawdust
Apart from its role as a cheapening extender, sawdust in many cases aids processing considerably by reducing nerve. Batches containing sawdust are mixed more easily and rapidly. Where high physical properties are not necessary, it is possible to use large quantities of sawdust.

3.2. Determination of Physicomechanical properties of NR-SD
The physicomechanical properties of NR:SD composites were compared. The results are summarized in Table (3.1). Comparison of hardness was found that hardness of composites increased with an increase in SD. It was found that tensile strength, elongation, tear strength are decreased and specific gravity, average mass loss on increased.

| Properties | Rubber composites |
|------------|------------------|
|            | NR | SD25 | SD50 | SD75 |
| Hardness (IRHD) | 43 | 57  | 84  | 98.7 |
| Specific gravity | 1.22 | 1.34 | 1.46 | 1.55 |
| Tensile strength (MPa) | 13.0 | 5.3  | 3.1  | 2.6 |
| Elongation at break (%) | 614 | 363  | 86  | 18  |
| Tear strength (kN/m) | 36.6 | 19.1 | 19.0 | 20.7 |
| Abrasion resistance (mg) | 272 | 492  | 391 | 542 |

3.3. Surface Morphology of NR, NR-SD
Surface morphologies of NR 100% composites, different NR-SD composites were investigated by scanning electron microscopic technique. The SEM micrographs of each sample are presented in Figure. Figure (3.1) shows the surface...
nature of 100 % NR composites. Its surface has almost smooth texture particles of ingredients used in rubber compounding and dispersed homogeneously through the rubber matrix.

Figure (3.1) indicates the surface images of NR 100% and SD25 composites. These two images are very different from each other. According to images of SD25, 25% of sawdust particles are uniformly throughout the entire matrix of natural rubber. The particles size (less than 10 μm) are in the range of nano size. The particles are arranged orderly so that it enhances the quality of rubber composite. On the other hand, the surface image of NR75-SD25 shows randomly orient nature of particles no homogeneous smooth texture can be seen on it. This means the mechanical properties of its not as good as SD25. According to surface image of SD75 composite rough texture of surface can be seen. The particles are not orderly arranged and clusters of particles on its surface make poor quality of composite.

The average swelling percent of NR and SD composites toward ethanol, it can be seen that the composites with SD 25% filler indicates the swelling properties compared to the other composites. 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, NR and SD composites with NR 75% and SD 25% filler can be absorbed the ethanol and the test piece were swelled. The test pieces become SD saturated with selected oil that were no increasing in weight after 6 days. The composites with any filler were used, the composites has nearly same swelling percent.

The average swelling percent of composites toward gasoline, the NR 75 composites with SD filler can absorb the highest amount of gasoline. The SD 25% filler show the more absorption than the SD 25% filler. Hence, rubber goods and tyre made up the composites with SD 25% filler is the best gasoline resistance.

Table (3.2) shows the average swelling percent of composites toward diesel oil. The composites can absorb diesel. The composites with NR 75% and SD 25% filler is more swelling than SD 25% filler. Therefore, the composites with SD 25% filler being the oil resistance can produce rubber goods and tyre.

The swelling percent of the composites toward engine oil, the composite with SD filler has a little swelling. Although NR is a good oil sorption material, the composites with SD filler can absorb a little amount. The composites with NR 75% and SD 25% fillers indicate more swelling than the composites with SD 25% filler. Hence, the composite with SD 25% filler has the best engine oil resistance. Based on this fact, SD25 composite might be used in making the O'ring.

The swelling percent of the composites toward used engine oil, the composite with SD filler has a little swelling. Although NR is a good oil sorption material, the composites with SD filler can absorb a little amount. The composites with NR 75% filler indicates more swelling than the composites with SD 25% filler. Hence, the composites with SD 25% filler has the best used engine oil resistance in Table (3.2).

### Table 3.2 Swelling properties of SD25 (NR 75%-SD 25%) composite

| Solvent        | Before wt. (g) | After wt. (g) | Swelling percent (%) |
|----------------|---------------|--------------|----------------------|
|                | 3 day | 6 day | 9 day | 3 day | 6 day | 9 day |
| EtOH           | 1.08  | 1.11  | 1.10  | 1.11  | 2.8   | 1.9    | 1.9    |
| Gasoline       | 0.73  | 1.48  | 1.51  | 1.51  | 102.7 | 106.8  | 106.8  |
| Diesel         | 1.06  | 1.63  | 1.81  | 1.84  | 53.8  | 70.8   | 73.6   |
| Engine oil     | 0.83  | 0.94  | 0.99  | 0.94  | 13.3  | 19.3   | 13.3   |
| Used engine oil| 1.44  | 1.59  | 1.67  | 1.71  | 10.4  | 16.0   | 18.8   |

### 3.4. Studies on the Swelling Properties of NR, SD

The results for swelling of prepared rubber composites in selected solvents such EtOH, gasoline, diesel, engine oil and used engine oil in Table (3.2).

### 3.5. Thermogravimetry-Differential Thermal Analysis (TG-DTA) of Prepared NR, SD25

Thermal methods of analysis measure chemical and physical changes that a material undergoes as it is heated. The changes include weight gain or loss, change in dimension or strength and release or absorption of energy. The thermogram of natural rubber composites (NR, SD25) are presented in Table (3.3).

According to thermogram, it has about 9.059 % loss in weight between the temperature range of 40 °C and 341 °C. This may be due to the loss of surface water, absorbed water and bounded water. The second stage between about the temperature range of 341 °C and 518 °C show loss in weight about 43%. It has been corresponding to the burning of carbon and sulphur. It has been observed that decomposition of natural rubber occurred.

The thermogram profiles of SD25 composite was similar and degrading temperature were nearly the same. Therefore, SD25 composite has the lowest thermal stability.
Table 3.3 Thermal analysis of prepared Rubber composites (NR, SD25)

| Composites | TG thermogram | DTA thermogram | TG-DTA remark |
|------------|---------------|----------------|---------------|
|            | Break in temperature | Weight loss (%) | Temperature (ºC) | |
| NR (100 %) | 59-240        | 1.69           | 220.44        | Thermally stable up to 200 ºC, loss of surface water |
|            | 240-405       | 42.71          | 372.93        | Degradation of polysioprene units, loss of some ingredients of rubber compounding, loss of some fragments |
|            | 405-585       | 16.27          | 520.61        | Depolymerization of rubber backbone |
| SD25       | 40-341        | 9.059          | -             | Withstand up to 300 ºC |
|            | 341-518       | 42.679         | 450           | Decomposition of fragments of polymer and backbone of polymer |

4. Conclusion
The results of investigation have shown that the durable and flexible rubber composites can be produced to prepare useful items such as tyre. The optimum conditions of NR-SD was found to be NR 75%: SD 25% (NR:SD) composites was made by mixing the ratio as (NR75%:SD25%) (SD25). It was found that the ratio of (NR75%: SD25%) (SD25) gives the best composites for making flooring and O’ring.

According to SEM, it was found that NR75%:SD25% (SD25) are smooth textures. From TG-DTA analysis, it was found that the loss of mass in NR75%:SD25%(SD25) showed more improved resistance to thermal decomposition over that of NR 100%. Using sawdust (SD) as substitutes in rubber goods, it reduces the cost of production.

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