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## **Recycling of Scrap Tyres**

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### **Abstract**

De-vulcanization of rubber waste poses a challenging economical, environmental and social problem. In the present study, we propose a new de-vulcanization process to reuse the rubber waste called mechano-chemical process (MCP), where the waste were de-vulcanized through the application of mechanical shearing, heat (1100C) and de-vulcanizing agent (Di-benzyl disulfide). A new look into the de-vulcanization mechanism and the influence of de-vulcanizing agent on mechanical properties of re-vulcanized rubber was presented. One of the most interesting observations is that the retention of tensile strength of re-vulcanized rubber with respect to original tyre is 35.1% and 75.6%, when de-vulcanized in absence and in presence of disulfide respectively. The formation of extra cross-link bond in re-vulcanized rubber containing disulfide was confirmed from cross-link density data and TGA result. DMA analysis revealed that the storage modulus increased for re-vulcanized rubber containing disulfide. T

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### **Introduction**

The disposal of the large amount of tyre waste generated worldwide, becoming an increasing environmental and economic problem, because of their three-dimensional chemical network. As a result it is neither fusible nor soluble and consequently, it cannot be remolded into other shapes without serious degradation. Large amounts of rubbers are used as tyres for trucks, cars, aeroplanes, two wheelers etc. After long use when these tyres are not serviceable they are discarded. During long run only few grams of rubber are abraded out from the tyre. Almost entire amount of rubber from a worn out tyre is discarded, which takes very long time for natural degradation<sup>1,2</sup>. Many attempts<sup>3-7</sup> were made (combustion, burying under ground and recycling) for reuse the used and waste tyre of which the most important method is recycling<sup>8-10</sup> as per the environmental pollution concern. However, for recycling the important obstacles is economical problem. For recycling various methods<sup>5,9,11-16</sup> have been developed. In most of these method ground rubber was used as filler or blended with virgin rubber or other polymers<sup>17</sup>. There are only a few papers<sup>10,13,18,19</sup> discussing about the total conversion of waste tyre rubber to new rubber product. Unfortunately no techniques can yet recover more than 40% of the original tyre rubber properties.

The main object of this work is to de-vulcanize the waste tyre by a mechanochemical process (MCP), where we used the mechanical shearing (cracker-cum mixing-mill), thermal energy (115<sup>0</sup>C) and de-cross-linking chemicals (diaryl disulfide) simultaneously to induce de-vulcanization. The de-vulcanized rubber mass was then re-vulcanized with sulfur, CBS and retarder at 145<sup>0</sup>C and compared their properties with those of original tyre. The main advantage of this process is that it is eco-friendly and economically viable process and it gives fine elastic rubber powder, unlike cryoground rubber. It is very difficult to disperse the curative thoroughly into the cryoground rubber<sup>20</sup>, but the elastic rubber powder produced by our process can accept the curatives and they can disperse uniformly throughout the mass in the next re-vulcanization step.

### **Experimental**



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### **Materials**

Worn out tyre tread (natural rubber (NR) based) was supplied by Birla Tyres (India), and N-cyclohexyl- 2-benzothiazyl sulfenamide (CBS) was supplied by Bayer (India) Ltd. The diaryl disulfide was supplied by Aldrich Chemical, USA and used together with aromatic process oil.

### **Mechanochemical de-vulcanization process**

This vulcanized rubber compound was ground with the help of an open cracker-cum-two roll mixing mill at 115<sup>0</sup> C for 10 minutes, and then milled in an open two-roll mixing mill with simultaneous addition of the diaryl disulfide and the process oil at the same temperature. (The two-roll open cracker-cum-mixing-mill is a type of mixing mill having both the cracking zone and the mixing zone on the same roll; it was developed in-house. The main function of this mill is to reduce lump size. Its flight length, angle and land and sea ratio are so adjusted that the bigger lumps are automatically transferred, after being cracked, to the mixing zone, where we place the de-vulcanizing agent). This de-vulcanized rubber was re-vulcanized with sulfur, CBS and cure retarder (thiophthalimide type) at 145<sup>0</sup>C, up to the optimum cure time.

### **Cure characteristics of rubber compound**

The cure characteristics of the rubber vulcanizate were studied using a Monsanto Rheometer, R-100 at 145<sup>0</sup>C. From the graph the minimum torque, ML (dN.m); the maximum torque, MH (dN.m); the scorch time,  $t_2$  (min) (i.e. the time to two units of torque increases above the minimum); optimum cure time,  $t_{90}$  (min) (i.e. the time to 90% of maximum torque developed); state of cure  $\{t_{max} - t_{min} \text{ (dN.m)}\}$  were determined.

### **Mechanical characterization**

Vulcanized slabs were prepared by compression moulding, and the dumbbell shaped specimens were punched out. The tests were carried out by means of a universal tensile testing machine, Hounsfield H10KS. The moduli at 100%, 200% elongation, tensile strength, tear strength and elongation at break were measured at room temperature. The initial gauge length of each specimen was 25mm and the speed of the jaw separation was 500mm/minute.

Five samples were tested for each set of conditions, at the same elongation rate. The values of the tensile strength, modulus at 100% and 200% elongation, and elongation



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at break were averaged. The relative error was below 3%. The Shore A hardness was measured.

**Determination of cross-link density**

The cross-link density was determined by immersing a small amount of sample in 100ml benzene to attain equilibrium swelling. After this the sample was taken out from the benzene and the solvent was blotted from the surface of the sample and weighed immediately. This sample was then dried out at 80<sup>0</sup>C to constant weight. Then the chemical cross-link density was calculated by the Flory Rehner equation<sup>21</sup>.

**Sol-gel determination**

Small pieces of rubber samples were placed in benzene for five days at room temperature for sol-gel determination. Gel was filtered, washed with benzene and dried to constant weight. The sol fraction was taken as an indication of degree of the re-vulcanization and it was calculated from the expression:-

$$\text{Sol fraction (\%)} = (W_0 - W_1) 100/W_0$$

where  $W_0$  was the dry sample mass before swelling, and  $W_1$  was the dry sample mass after swelling.

The gel fraction can be calculated as

$$\text{Gel fraction (\%)} = 100 - \text{Sol fraction}$$

**Thermal characterization**

Thermo-gravimetric analysis (TGA) was carried out using a Universal V1.12E TA instrument in the range 30<sup>0</sup>C-900<sup>0</sup>C at a heating rate of 20<sup>0</sup>C/min in a nitrogen atmosphere.

**Dynamic mechanical analysis (DMA)**

Dynamic mechanical properties of the vulcanizate were analysed using a TA Instrument DMA 2980 dynamic mechanical analyser in tension mode. The samples were subjected to a sinusoidal displacement of 15 $\mu$ m at a frequency of 1 Hz from -70<sup>0</sup>C to 165<sup>0</sup>C, with a heating rate of 5<sup>0</sup>C/min.

**Results and Discussion**

The compounding formulation used for de-vulcanization studies of the Automobile tyre are shown in Table I. For re-vulcanization sulfur and CBS are used based on 100gm of virgin rubber (Table II), because according to De<sup>22</sup> and Gibala et al.<sup>23</sup>



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the de-vulcanized rubber does not cause vulcanization alone. The same results also obtained in our present study, the rheometric plots for de-vulcanized rubber (Fig. 1 graph 'DEVUL') were almost straight.

### **Cure characteristics**

The cure characteristics of de-vulcanized and re-vulcanized rubber are presented in Fig. 1. The parameters studied as a function of diaryl disulfide concentration including minimum torque, maximum torque, scorch time and cure time are shown in Table III. Generally the minimum torque in a rheograph is presented as the minimum viscosity and the maximum torque is presented as the maximum viscosity value. The minimum torque for re-vulcanized rubber GR obtained by de-vulcanization in presence of disulfide was lower than re-vulcanized rubber in absence of disulfide (KR) and the minimum torque decreases consequently with increasing disulfide concentration (HR). The increasing concentration of disulfide effectively dissociates more cross-links in the main polymeric chain, and this results in a decrease in viscosity. The decrease in minimum torque also indicates that the processability of the de-vulcanized rubber increases with increasing disulfide concentration. On the other hand torque maximum for re-vulcanized rubber KR was higher than GR but for re-vulcanized rubber HR the value again higher than KR. The re-vulcanized rubber KR shows the high torque maximum as the in absence of disulfide small amount of cross-links dissociation occurs due to mechanical milling, so large number of cross-links originally present in the original tyre still persist and these account for the high maximum torque. For disulfide containing vulcanizate large amounts of cross-links were dissociated and on re-vulcanization though the new cross-links are formed, the number is less than that was dissociated. This is supported by the cross-link density data (Table IV). This may be due to some side reaction (formation of cyclic sulfidic and pendent group and other non active group) was occurred in the time of re-vulcanization. The torque maximum and torque difference increases with increasing concentration of disulfide. This is due to the fact that the contribution of original undissociated cross-links to the maximum rheometric torque is negligible for higher amounts of disulfide containing vulcanizate HR. Another important observation is that



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for re-vulcanized rubber KR the reversion reaction is more than the re-vulcanized rubber GR and HR.

The scorch time and cure time for re-vulcanized rubber KR is very low compared to re-vulcanized rubber GR and HR. In absence of de-vulcanizing agent (diaryl disulfide) very small amount of cross-links are dissociated. At lower concentration lower amount of cross-links are dissociated and this number increases with increase in concentration of disulfide. Again for re-vulcanized rubbers the ratio of sulfur/CBS are constant but for disulfide containing re-vulcanized rubber this ratio to sulfur radicals decreases as more new sulfur cross-links are formed, hence large time is required for re-vulcanization. Similar results were also observed by Ishiaku et al.<sup>17</sup> using De-Link R as de-vulcanizing agent.

#### **Mechanical properties**

The stress-strain curves for original tyre and re-vulcanized rubber are shown in Fig. 2, to assess the effect of de-vulcanizing agent (diaryl disulfide) on the tensile properties (tensile strength, elongation at break, 200% and 300% modulus) of the re-vulcanized rubber. The tensile properties of the re-vulcanized rubber are lower than the original tyre. The reduction of tensile properties of the re-vulcanized rubber may be due to chain scission during mechanical shearing. This will result the de-vulcanized rubber having the lower molecular weight than the original tyre rubber. Also on re-vulcanization some amount of newly formed cross-links will be broken due to heat and pressure in another vulcanization step. Further more, on re-vulcanization, all the cross-links, which was broken in the de-vulcanization step, do not recombine again and there is a possibility of desulfuration, decomposition and side reaction that may produce cyclic sulfides, sulfidic pendent groups. Another factor, which may be responsible for decrease in tensile strength, is the presence of greater amount of cross-linked gel in re-vulcanized rubber compared to original tyre. The cross-linked gel is not dispersed in the continuous matrix of re-vulcanized rubber and it remains as weak sites for stress transmission for re-vulcanized rubber and lowers the tensile strength. The Fig. 2 also shows that the tensile strength of re-vulcanized rubber obtained by de-vulcanization in presence of disulfide (GR and HR) was higher than in absence of disulfide (KR). This may be due to the



disulfide, which breaks tremendously the sulfur cross-linking present in original tyre, which on re-vulcanization with sulfur and CBS forms more new cross-links. Again the tensile strength of re-vulcanized rubber increases significantly with the increase in diaryl disulfide concentration, only 0.3phr increase in disulfide concentration the tensile strength increases about 13.2% compared to original tyre. The modulus (200% and 300%) elongation at break, tear strength and hardness of re-vulcanized rubber KR was lower than the re-vulcanized rubber GR and HR (Table IV). Enhancement of these mechanical properties manifests itself in Table IV, by a sharp increase in cross-link density and gel-fraction.

#### **Effect of diaryl disulfide on mechanical properties of re-vulcanized rubber**

Fig. 3(A), (B), (C) and (D) compares the tensile strength, elongation at break, tear strength and cross-link density data of original tyre and re-vulcanized rubber both in presence and absence of disulfide. The retention of tensile strength of re-vulcanized rubber with respect to original tyre was 35% when de-vulcanized in absence of disulfide (KR) and 67.5% in presence of lower amount of disulfide (GR), 80.7% in presence of higher amount of disulfide (HR) respectively. Similarly the retention in cross-link density of re-vulcanized rubber was 44.5%, 82.6% and 96.7%, for KR, GR and HR respectively. Many of these mechanical properties of re-vulcanized rubber (GR and HR) shown in Fig. 3(A), (B), (C) and (D) were comparable with those of industrially manufactured vulcanized natural rubber and it is believed that the de-vulcanized rubber produced by this mechanochemical process may be able to replace virgin natural rubber in numerous applications.

#### **Thermal properties**

Thermo-gravimetric analysis (TGA) was used to investigate the thermal decomposition behaviour of the rubber. Fig. 4A shows the thermo-gravimetric of original tyre and re-vulcanized rubber. Fig. 4B shows the DTG curves from the same TGA analysis. The corresponding thermal parameters obtained from those curves are given in Table 6 where  $T_{50}$  and  $T_{90}$  are the temperature at 50% and 90% weight loss. It can be seen from the table that the addition of disulfide in the de-vulcanization step increases the onset degradation temperature ( $T_1$ ) and the temperature of 50% and 90% decomposition



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for corresponding re-vulcanized rubber. The delayed degradation for GR and HR suggests that it is thermally more stable because of more cross-linking (Table IV). Table 6 is again indicates that the onset degradation temperature,  $T_{50}$  and  $T_{90}$  increases with increasing disulfide concentration in the de-vulcanization step. This once again supports the previous hypothesis that the diaryl disulfide predominantly breaks the cross-links in the original tyre, resulting the more stable vulcanizate on re-vulcanization. From Fig. 4B it is cleared that the temperatures where the maximum degradation occurs ( $T_{max}$ ), of re-vulcanized rubber GR and HR is higher than the KR. This may be due to the re-vulcanized rubber GR and HR has more cross-link density (Table IV). Cross-linking increases the rigidity of the system, which in turn increased the thermal stability<sup>25,26</sup>.

#### **Dynamic mechanical properties**

Fig. 5A and 5B shows the temperature dependence of storage modulus ( $E'$ ) for original tyre and different re-vulcanized rubber. Each curve of the vulcanizate has three distinct regions, glassy region, leathery region and rubbery region. With increase in temperature the storage modulus of the vulcanizates sharply decreases. It is due to the decrease in stiffness of the vulcanizate. Below the glass transition temperature (Fig. 5A) of the vulcanizates (glassy region), the storage modulus of the re-vulcanized rubber was higher than the original tyre and it is highest for re-vulcanized rubber HR containing higher amounts of disulfide. In the glassy state molecular motion are stopped. This leads to lower entanglement, which in turn increased the modulus for re-vulcanized rubber compared to the original tyre. At above ambient temperature (Fig. 5B) the storage modulus of re-vulcanized rubber (KR, GR and HR) was lower than original tyre (OR) and it increases with increasing the concentration of disulfide. Fig. 5B shows that the storage modulus of OR, KR, GR and HR at room temperature ( $27^{\circ}\text{C}$ ) are approximately 8.8 MPa, 5.9 MPa, 7.4 MPa and 8.6 MPa respectively. The increase in storage modulus for disulfide containing re-vulcanized rubber is again suggests that the degree of cross-linking of the re-vulcanized rubber significantly improved when diaryl disulfide was employed in the original tyre as a de-vulcanizing agent.

Fig. 6A and 6B shows the loss modulus ( $E''$ ) verses temperature plots of the vulcanizates. The glass transition temperature ( $T_g$ ) of the vulcanizates was taken at the





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maximum of  $E''$  and  $\text{Tan}\delta$  value and is shown in Table 6. For re-vulcanized rubber the  $T_g$  value (taken at the maximum of  $E''$ ) shifted towards higher temperature side compared to original tyre. This is may be due to the increase in molecular chain mobility (due to main chain degradation) and decrease in cross-link density. The improvement of cross-linking is again observed for disulfide containing re-vulcanized rubber. From the value of loss modulus, a shift in peak towards the low temperature region is detected. The  $T_g$  taken from  $E''_{\text{Max}}$  is lower than that taken from  $\text{Tan}\delta_{\text{max}}$ , but in both cases the  $T_g$  is higher in disulfide containing re-vulcanized rubber (GR and HR) than the non-disulfide containing re-vulcanized rubber (KR)(Table VI). This fact supports that the disulfide tremendously breaks the sulfur cross-links in de-vulcanization steps, as a result the cross-linking increases on re-vulcanization. Above the room temperature the loss modulus of re-vulcanized rubber (Fig. 6B) is lower than the original tyre. This indicates that the heat build up properties of the re-vulcanized rubber are better than original tyre. The temperature variation of  $\text{Tan}\delta$  curves is shown in Fig. 7. Only one relaxation peak was observed over the whole temperature studied. The maximum loss tangent is higher for re-vulcanized rubber KR than the GR and HR and the height of the peak also reduced in the same order. It is accepted that the higher the  $\text{Tan}\delta_{\text{max}}$  the greater is the mechanical losses; these losses are related to high-energy input required for the motion of the molecular chain of the polymer as the transition is being approached<sup>24</sup>. This is due to the improved cross-linking of the disulfide containing re-vulcanized rubber, which resists the flexibility of the vulcanizate.

## CONCLUSIONS

De-vulcanization of Scrap tyre (natural rubber based) through mechanochemical process is possible. The mechanical property of the re-vulcanized rubber markedly depends on the disulfide concentration used in de-vulcanization step and it improves with increasing the concentration of disulfide. With incorporation of 1 phr of diaryl disulfide it is possible to recover more than 80% of the original tyre properties. So even though it is difficult to obtain 100% re-vulcanized rubber, the properties (mechanical and thermal) of the re-vulcanized rubber can still prove that mechanochemical de-vulcanization process can be an effective process for recycling of scrap tyre.



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**TABLE I.** Composition of de-vulcanizing agent for Automobile Tyre

Ingredients (phr)	D <sub>1</sub>	D <sub>2</sub>	D <sub>3</sub>
Automobile Tire (NR)	100	100	100
Process oil	6	6	6
Diaryl disulfide	-	0.7	1.0

**TABLE II.** Compound formulations for re-vulcanization study

Ingredients (phr)	KR	GR	HR
D <sub>1</sub>	100	-	-
D <sub>2</sub>	-	100	-
D <sub>3</sub>	-	-	100
Sulfur	2.5	2.5	2.5
CBS	0.8	0.8	0.8
Retarder	1	1	1

**TABLE III.** Cure characteristics of the vulcanizate



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Sample Code	Scorch Time ( $t_{s2}$ ) (min)	Optimum Cure Time ( $t_{90}$ ) (min)	Minimum Torque (dN.m)	Maximum Torque (dN.m)	State of Cure (dN.m)
KR	3	14	22.5	63	40.5
GR	9	20	18	61.5	43.5
HR	11	23	12	67.5	55.5

**TABLE IV.** Mechanical properties of the vulcanizate

Sample Code	200% Modulus (MPa)	300% Modulus (MPa)	Tensile Strength (MPa)	% of elongation at break	Tear Strength (N/mm)	Hardness (Shore A)	Cross-link Density (moles/gm) X 10 <sup>4</sup>	% Of Gel
OR	6.9	10.4	24.3	596	42.2	68	1.712	93.8
KR	4.0	6.8	8.5	352	29.6	62	0.761	82.5
GR	3.7	6.1	16.4	625	41.1	63	1.421	87.7
HR	4.2	7.1	19.6	631	47.5	65	1.655	91.8

**TABLE V.** TGA parameters

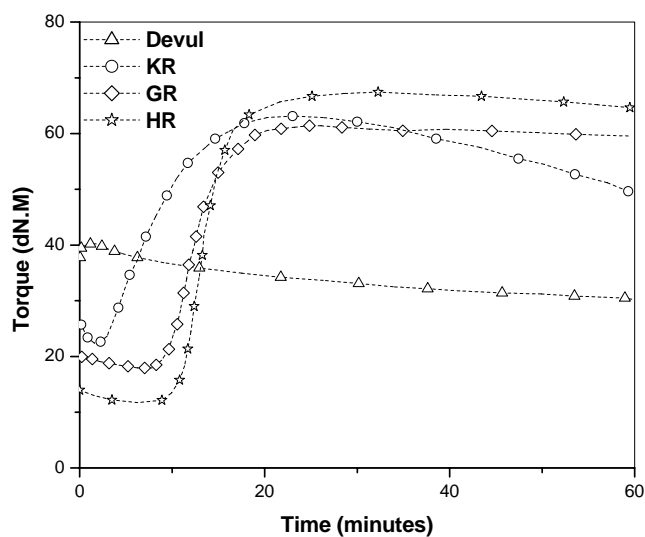
Sample code	T <sub>1</sub> ( <sup>0</sup> C)	T <sub>50</sub> ( <sup>0</sup> C)	T <sub>90</sub> ( <sup>0</sup> C)	T <sub>max</sub> ( <sup>0</sup> C)	d(m/m <sub>0</sub> )/dt(mg/min)
OR	310	366	700	385	1.313
KR	272	343	388	371	0.391
GR	296	372	-	387	0.824
HR	305	376	-	386	0.913

**TABLE VI.** Viscoelastic properties of the vulcanizates



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Sample Code	OR	KR	GR	HR
$E''_{max}$ (MPa)	690	719	677	846
$T_{g, taken at E''_{max}}$ ( $^{\circ}C$ )	-61.0	-48.6	-49.1	-51.2
$Tan\delta_{max}$	0.977	0.975	0.937	0.961
$T_{g, taken at Tan\delta_{max}}$ ( $^{\circ}C$ )	-50.5	-33.2	-35.8	-36.1



**Figure 1.** Rheometric plots of the rubber vulcanizate

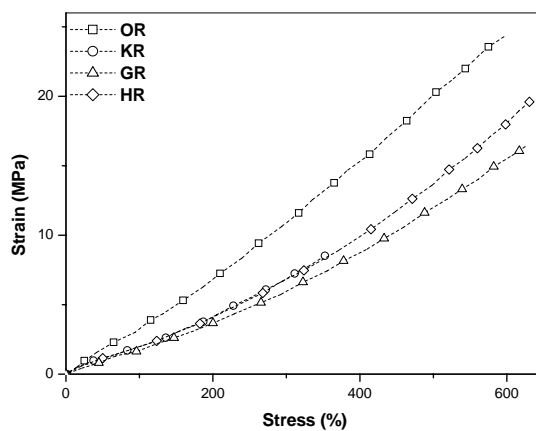




Figure 2. Stress-Strain curves of the rubber vulcanizate

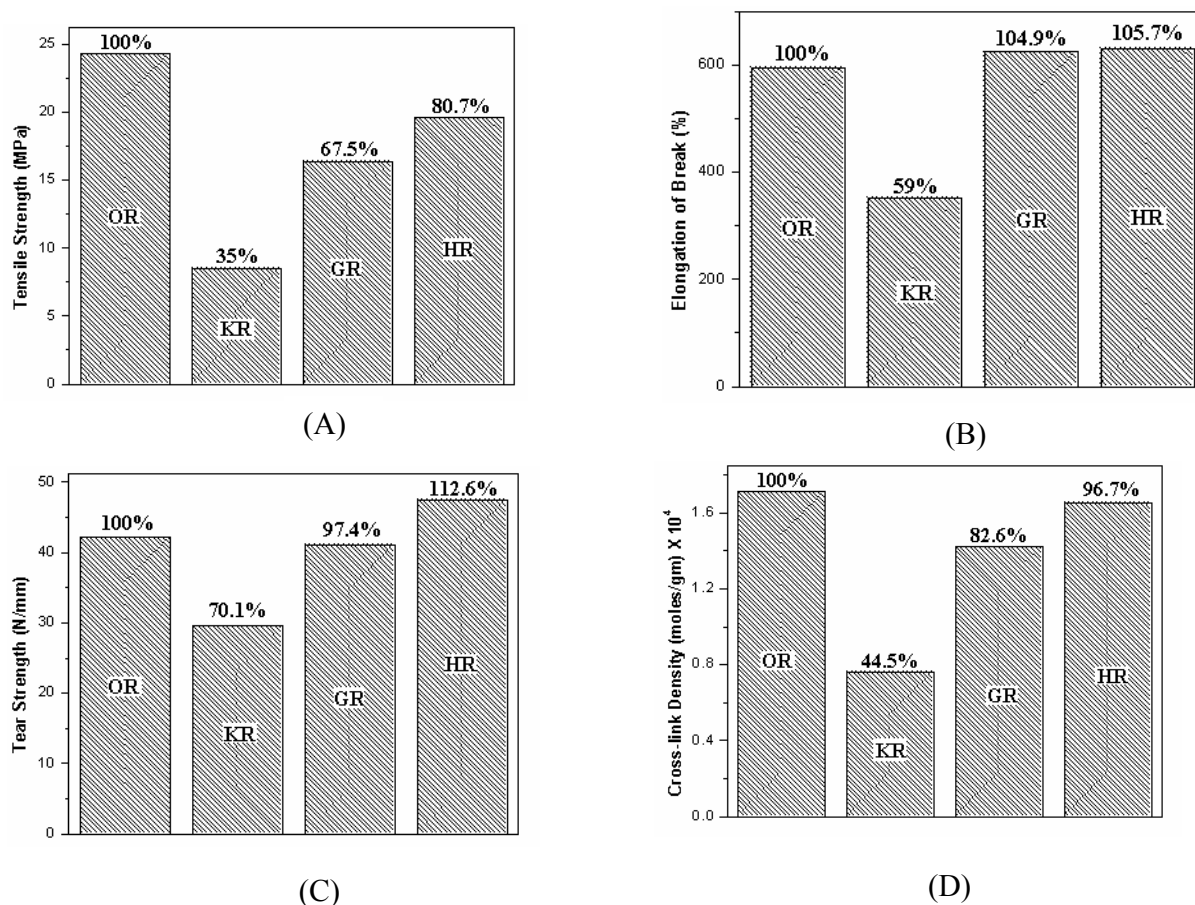
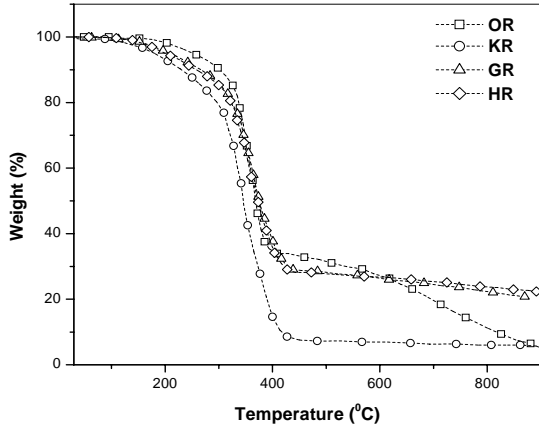
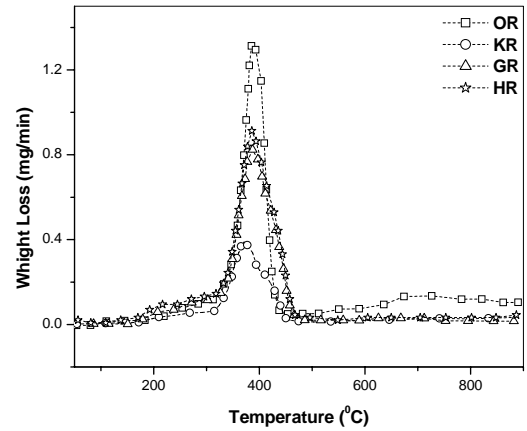


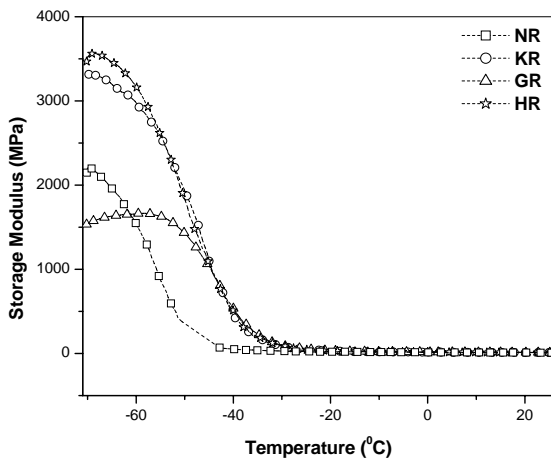
Figure 3. (A). Variation of tensile strength as a function of disulfide concentration, (B) Variation of Elongation at break as a function of disulfide concentration, (C) Variation of tear strength as a function of disulfide concentration, (D) Variation of cross-link density as a function of disulfide concentration.



(A)

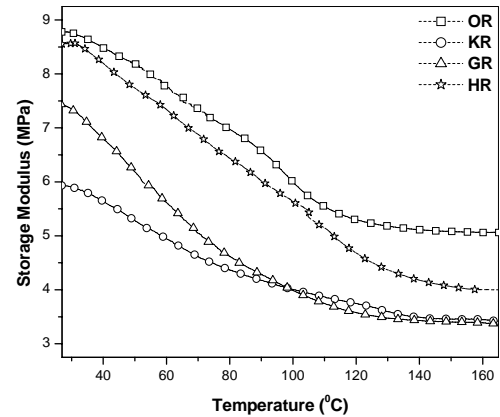


(B)



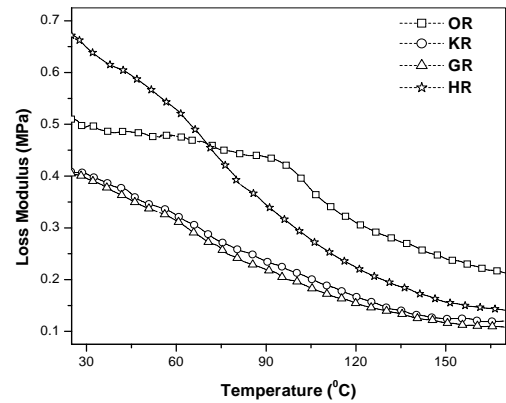
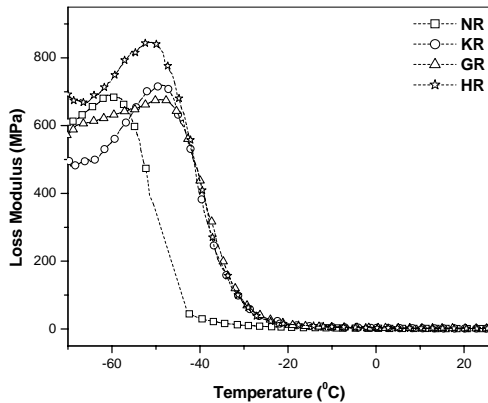
(A)

al tyre



(B)

**Figure 5.** Storage modulus of the rubber as a function of temperature, (A) below room temperature (B) above room temperature.

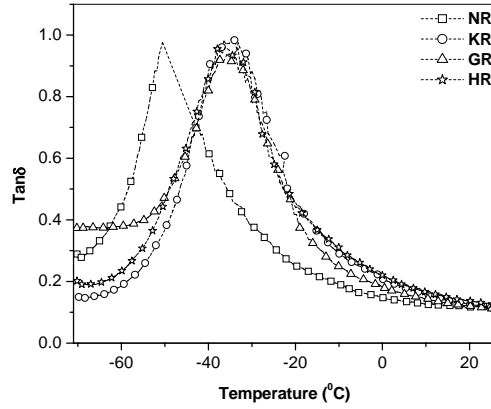


(B)



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**Figure 6.** Loss modulus of the rubber as a function of temperature, (A) below room temperature (B) above room temperature.



Fig

perature