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Thiosalicylic acid as a devulcanizing agent for mechano-chemical devulcanization

Pipat Thaicharoen*, Patchanita Thamyongkit,****, and Sirilux Poompradub***,****,†**

*Program of Petrochemistry and Polymer Science, Faculty of Science,
**Department of Chemistry, Faculty of Science,
***Department of Chemical Technology, Faculty of Science,
****Center of Excellence for Petroleum, Petrochemical, and Advanced Materials,
Chulalongkorn University, Bangkok 10330, Thailand
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Abstract−A mechano-chemical devulcanization process for vulcanized natural rubber (NV) was investigated. Thiosalicylic acid was used as a test devulcanizing agent in comparison to diphenyl disulfide as the reference. The optimum condition for devulcanization of NR vulcanizates (NVs) was found to be grinding of the NV and subsequent mixing with a selected devulcanizing agent at 140 °C for 30 min. The degree of devulcanization was indicated by using sol-gel fractions of the devulcanized rubber (DVR). Revulcanized rubber was made by using virgin natural rubber (NR) containing DVR at different ratios. The tensile strength of the DVR/NV composite, after revulcanization, decreased by 5-10%, while the elongation at brake was improved by 5-10% at a DVR content of 5-15%. Devulcanization of industrial truck tires, as a typical sample of industrial products, was also demonstrated for the practical application of this technique.

Key words: Devulcanized Rubber, Devulcanization, Devulcanizing Agent, Vulcanization, Mechano-chemical Devulcanization

INTRODUCTION

The process of rubber vulcanization by sulfur is essential for the manufacture of suitable tires for automobiles and aircrafts, which are indispensable to our modern society. Sulfur bridge formation, generated between rubber molecules by this process, affords an elastic three-dimensional network. However, the sulfur bridges cannot be naturally removed or decomposed upon disposal of the rubber waste. Attempts to destroy the three-dimensional structure have proved difficult, expensive and energy-consuming. Currently, the amount of global rubber waste has drastically increased, leading to global environmental problems. The devulcanization of rubber waste, especially automobile tires, is the most important issue for the rubber industry, and potential methods of recycling waste tire rubber are of great value and demand. Devulcanization involves the cleavage of the intermolecular carbon-sulfur and/or sulfur-sulfur bonds, leading to shorter polymer chain lengths compared to that of the original, and so resulting in poorer rubber properties [1]. In general, the devulcanization of rubber waste is carried out through physical, chemical or biotechnological processes. In the physical process, rubber wastes are devulcanized with the help of external energy, for example, mechanical [2], cryomechanical [3,4], microwave [5,6], or ultrasonic [7-11]. However, these processes lead to a random cleavage of cross-linked and polymeric main chain linkage bonds, leading to a lower quality rubber after revulcanization.

In the chemical process, several chemical reclaiming agents have been used for the manufacture of devulcanized rubber, and typical examples include organic disulfides or mercaptans. Apart from these, the use of inorganic compounds has also been reported [12]. In this process, the rubber to be treated is initially swollen in common organic solvents, e.g., toluene, benzene and cyclohexane. However, such solvents are difficult to remove after the reaction. Recently, Kojima et al. reported the effectiveness of supercritical CO_2 as a swelling solvent for the chemical reclaiming process of unfilled and carbon black-filled polyisoprene rubber vulcanizates [13-16]. Residual CO_2 in the rubbery matrix was easily and rapidly removed by releasing pressure. Moreover, applications of supercritical *n*-butanol and toluene have been reported for the decomposition of used rubber into fuel oil [17]. However, a major drawback of the chemical devulcanization process is its high economic and environmental costs.

In the biotechnological process, biodegradation of the rubber powder has been performed by oxidation-reduction reactions driven by different species of *Thiobacillus*, *Acidithiobacillus*, *Nacardia*, and so on [18-22]. The efficiency and rate of devulcanization were found to be a function of particle sizes. Therefore, this process was exclusively and primarily limited to the surface layers of the elastomers [18].

The mechano-chemical process, which applies a mechanical force to the rubber and the addition of the devulcanizing agent, has been shown to be a more efficient approach, in terms of mechanical properties, in several studies [23-25]. In this content, here the mechano-chemical method, starting from grinding of the natural rubber (NR) vulcanizates, is followed by treating the resulting rubber sample by thiosalicylic acid.

The potential advantage of thiosalicylic acid as a devulcanization agent is that structurally, thiosalicylic acid combines thiol and carboxyl functional groups consistent with thiophenol and benzoic acid,

†To whom correspondence should be addressed.
E-mail: sirilux.p@chula.ac.th

respectively. Thiophenol is usually used as a devulcanizing agent to break the sulfur crosslinked bonds, while benzoic acid is well known as a retardant to prevent a fast curing in the vulcanization process. Therefore, thiosalicylic acid is expected to act as a bifunctional devulcanizing agent. Moreover, to our knowledge, the use of thiosalicylic acid has not yet been reported in the devulcanization of rubber, including in a mechano-chemical process. The aim of this study was hence to investigate the efficiency of thiosalicylic acid in the mechano-chemical rubber devulcanization process. The effects of the devulcanization time, and the amount of thiosalicylic acid, on the mechanical properties of the resulting rubber were studied in order to find the optimum condition for devulcanization. The mechanical properties of the revulcanized rubber were examined in comparison with that obtained when using the well-known diphenyl disulfide as the devulcanizing agent. The devulcanization of truck tires by the developed mechano-chemical process was also demonstrated so as to exemplify a potential practical industrial use.

EXPERIMENTAL

1. Materials

STR-5L from Thai Hua Chumphon Rubber Ltd. was used as NR. Active zinc oxide (ZnO), stearic acid, sulfur and *n*-cyclohexyl-2-benzothiazyl sulfenamide (CBS), used as curing reagents, were purchased from PI Industry Limited. Truck tire rubber powder (40 mesh size) was obtained from Union Pattanakit Ltd. Thiosalicylic acid and diphenyl disulfide were purchased from Merck and Fluka, respectively. Process oil was purchased from Pan Innovation Ltd. Toluene was purchased from Fisher and used as received.

2. Preparation of NR Vulcanizate (NV)

A sample, comprised of NR (100 part by weight per hundred part of rubber: phr), active ZnO (5 phr), stearic acid (2 phr), CBS (1 phr), and sulfur (3 phr) was mixed in a two-roll mill. The resulting mixture was then cured at 150 °C for 15 min by compressing molding. The sample NR vulcanizate is referred to hereafter as NV.

3. Devulcanization Process

The NV samples were each ground by a two-roll mill at room temperature for 10 min, and each resulting NV rubber powder (20 mesh size) was then mixed with the devulcanizing agent and process oil, and oven heated at 140 °C for 30 min to give devulcanized rubber (at various pretreatment times DVR). The formulation of the DVR so obtained is shown in Table 1.

4. Revulcanization Process

The virgin NR was mixed with the DVR at various (w/w) ratios (95/5, 90/10, 85/15, 80/20, 60/40 and 20/80) by two-roll mill, and then a sample (100 phr) was mixed with active ZnO (5 phr), stearic acid (2 phr), CBS (1 phr) and sulfur (3 phr), and cured as outlined in section 2.

5. Characterization

The two curing properties, scorch and optimum cure time, of the rubber compounding were measured by a rheometer MDR2000 (Monsanto, USA) at 150 °C.

The tensile test was performed with a universal testing machine LLOYD LR 5 K, (Fareham, UK), in accordance with ASTM-D412 at room temperature and a crosshead speed of 500 mm/min. The average value of five measurements is reported.

Sol and gel fractions were taken as the indication of degree of devulcanization and calculated by using Eqs. (1) and (2), respectively.

$$\text{Sol fraction } (\%) = \frac{(W_0 - W_1)}{W_0} \times 100 \quad (1)$$

$$\text{Gel fraction } (\%) = 100 - \text{Sol fraction } (\%) \quad (2)$$

where W_0 and W_1 is the dry sample mass before and after swelling, respectively.

The crosslink density of each sample was determined in triplicate by immersing the sample in toluene at room temperature for 72 h to attain equilibrium swelling. Each sample was then taken out from toluene and the solvent was blotted from the sample surface. The resulting sample was weighted immediately and then it was oven dried at 80 °C to constant weight. The crosslink density was calculated by the Flory-Rehner Eq. (3) [26].

$$\nu = \frac{-[\ln(1-\nu_r) + \nu_r + \chi\nu_r^2]}{V_0[\nu_r^{1/3} - (\nu_r/2)]} \quad (3)$$

where ν is the crosslink density, ν_r is a volume fraction of the rubber network in the swollen gel, V_0 is a molar volume of the swelling

Table 1. Formulations and properties of NR vulcanizate (NV) and devulcanized rubber (DVR), derived from either thiosalicyclic acid (DVRD1-6) or diphenyl disulfide (DVRD7)

Sample code	NV	DVRD1	DVRD2	DVRD3	DVRD4	DVRD5	DVRD6	DVRD7
NR vulcanizate (phr[a])	100	100	100	100	100	100	100	100
Thiosalicylic acid (phr)	-	1	1	1	1	0.5	2	-
Diphenyl disulfide (phr)	-	-	-	-	-	-	-	1
Process oil (phr)	-	8	8	8	8	8	8	8
Pretreatment time (h)	-	0	24	48	72	48	48	48
Devulcanizing temperature[b] (°C)	-	140	140	140	140	140	140	140
Sol fraction (%)[c]	1	26	29	33	33	29	35	33
Gel fraction (%)	99	74	71	67	67	71	65	67
Crosslink density $\times 10^4$ (mol/cm^3)	1.47	0.54	0.47	0.39	0.39	0.50	0.37	0.41

[a]Part by weight per hundred part of rubber
[b]at room temperature
[c]for 30 min

solvent and χ is equal to 0.393 and used as the rubber-solvent interaction parameter [27].

The morphology of revulcanized rubber was examined in cross-section after coating with gold using scanning electron microscopy (JSM-6480LV; JEOL, Japan) at an operating voltage of 15 kV.

RESULTS AND DISCUSSION

1. Properties of DVR Using the Mechano-chemical Process

Table 1 shows the properties of the untreated NV and the DVR obtained by the mechano-chemical devulcanization using thiosalicylic acid (DVRD1-DVRD6) or diphenyl disulphide (DVRD7) as the devulcanizing agent. The amount of thiosalicylic acid and process oil was kept constant at 1 and 8 phr, respectively, and the devulcanizing temperature was fixed at 140 °C. The use of this relatively low temperature, at below 150 °C, is to prevent the revulcanization reaction of NV and DVR during the devulcanization process. Attempts to perform this stage at an even lower temperature of 120 °C, however, resulted in inferior rubber (data not shown). In this study, the degree of devulcanization, which implies the cleavage of intermolecular bonds of the three-dimensional network, was determined by the sol-gel fraction of the DVR. A high sol fraction indicates that the three-dimensional network in the NV was efficiently destroyed, resulting in a decreased level of crosslink density. According to the derived sol and gel fractions of samples DVRD1-DVRD4, the sol fraction of the DVR gradually increased with increasing pretreatment time up to 48 hours inclusive, while the gel fraction correspondingly decreased, because of the higher degree of devulcanization with time. However, the sol-gel fraction seemed to be constant at 48 hours onwards with no further changes with longer pretreatment times. Accordingly, the shortest time that still provides the highest sol fraction, and thus the lowest crosslink density, of DVR was 48 hr.

Comparison of DVR samples DVRD3, DVRD5 and DVRD6 allows a rough investigation of the effect of the amount of thiosalicylic acid (in the 0.5 to 2.0 phr range) on the properties of the obtained DVR, while the amount of process oil (8 phr), pretreatment time (48 h at RT) and devulcanizing temperature (140 °C for 30 min) were all kept constant. While the sol fraction (%) of the DVR increased (29-35%) with increasing amounts of thiosalicylic acid (0.5 to 2.0 phr), the crosslink density of the DVR samples was significantly decreased compared with that of NV at 0.5 phr thiosalicylic acid, and even more so at 1 and 2 phr, although the sol fraction and crosslink density of these two thiosalicylic acid concentrates (DVRD3 and DVRD6) were almost comparable. The mechanical properties (tensile strength and % elongation at break) of the revulcanized rubber obtained by mixing the virgin NR with the DVR (DVRD3, DVRD5 and DVRD6) at three different (w/w) ratios are illustrated in Fig. 1. It is clearly observed that the high tensile strength of the revulcanized rubber was obtained when DVRD3 was mixed with the virgin NR. In contrast, at 2 phr thiosalicylic acid (DVRD6) a lower tensile strength was obtained in all three of the DVRD6/NR composites, but this was DVRD6 : NR ratio dependent. Almost all the samples showed an improvement of elongation at break. At a DVRD3 content of 5-15 phr, the tensile strength decreased by 5-10%, while the elongation at break was improved by 5-10%. Thus, the optimal condition for the mechano-chemical devulcanization process of NV in this study was found to be the use of 1 phr thiosalicyclic acid at

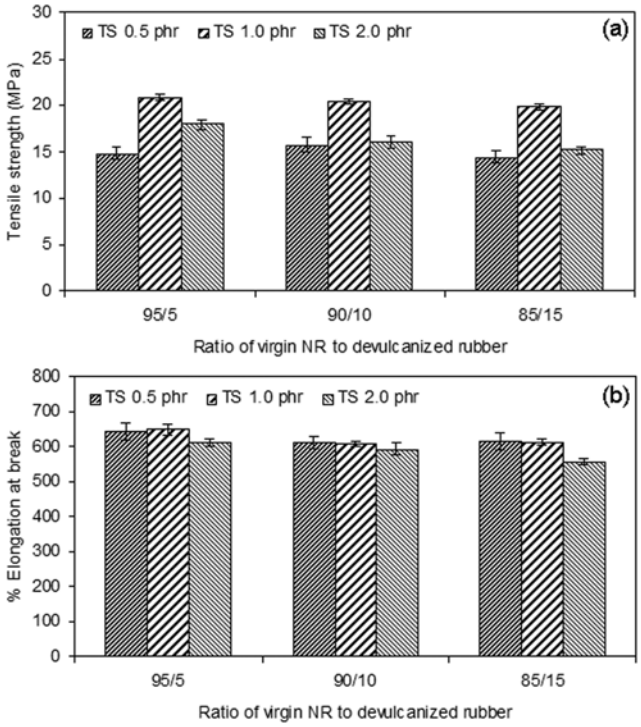

Fig. 1. The (a) tensile strength and (b) percentage of elongation at break of the revulcanized rubber under different amounts (0.5, 1.0 and 2.0 phr) of thiosalicyclic acid (TS) as the devulcanizing agent. Virgin NR (95, 90, 85 phr) was mixed with the devulcanized rubber (5, 10, and 15 phr) by two-roll mill, and then vulcanized again with the addition of active zinc oxide (5 phr), stearic acid (2 phr), CBS (1 phr) and sulfur (3 phr) at 150 °C up to the optimum cure time.

140 °C for 48 h.

For comparison purposes, the degree of devulcanization of NV using diphenyl disulfide as the standard devulcanizing agent was examined. Jana et al. reported that the use of disulfide families as a devulcanizing agent for NVs gave a good efficiency of sulfide bond cleavage [23,24]. The sol-gel fraction and crosslink density of DVR obtained using diphenyl disulfide (DVRD7) under otherwise the same conditions showed similar values to those obtained by devulcanization using 1 phr thiosalicylic acid (DVRD3) (Table 1). These results demonstrate that thiosalicylic acid can be used as the devulcanizing agent for NV with comparable devulcanization efficiency to that of the widely used diphenyl disulfide.

In general, sulfur is combined in the vulcanization network in several manners: that is mono-, di-, or poly-sulfide crosslinks; pendent sulfide groups; or cyclic mono- or di-sulfide unit. The number of sulfur atoms for each crosslink formed has been estimated by the accelerator to sulfur ratio [28]. The curing system used in this study was the conventional system since the level of sulfur is in the range of 2.0-3.5 phr and the level of accelerator is in the range of 1.0-0.4 phr. An accelerator to sulfur ratio of no more than 0.5 gives an NV which contains, at optimum cure conditions, a preponderance of polysulfidic crosslinks [29]. After the NV was subjected to the mechano-chemical devulcanization in the presence of 1 phr thiosalicylic acid at 140 °C, the sol and gel fractions of the revulcanized rubber were about 33 and 67%, respectively (Table 1). These results

Table 2. Cure characteristic and mechanical properties of revulcanized DVR/virgin natural rubber (NR) composite rubber

Sample code	NV	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10
Virgin NR (phr)	100	95	90	85	80	60	40	20	95	90	85
DVRD3 (see table 1) (phr)	-	5	10	15	20	40	60	80	-	-	-
DVRD7 (see table 1) (phr)	-	-	-	-	-	-	-	-	5	10	15
Scorch time (min)	6.0	3.3	2.4	2.0	2.0	1.3	1.1	1.1	3.4	2.4	2.0
Optimum cure time (min)	10.2	7.2	6.1	5.3	5.2	4.6	5.0	5.1	7.2	6.1	5.4
Max torque (dNm)	8.61	8.86	8.80	8.74	8.50	8.47	8.32	7.69	8.23	8.09	7.85
M_{100}[a] (MPa)	1.1±0.05	0.9±0.02	1.0±0.02	1.0±0.03	1.3±0.02	1.3±0.02	1.5±0.04	1.7±0.06	1.0±0.02	1.1±0.02	1.1±0.03
M_{200}[a] (MPa)	2.0±0.04	1.5±0.02	1.7±0.05	1.8±0.06	2.3±0.07	2.6±0.04	2.9±0.04	-	1.7±0.03	1.9±0.03	2.0±0.07
T_B[b] (MPa)	22.5±0.63	20.9±0.36	20.4±0.49	19.8±0.33	14.6±0.7	6.7±1.34	3.3±0.48	2.7±0.33	19.9±0.38	20.2±0.08	19.0±0.17
E_B[c] (%)	577±15	651±16	607±7	613±11	486±5	343±27	215±25	159±17	610±5	562±4	582±19
Crosslink density×10^4 (mol/cm^3)	1.47	1.93	2.00	2.13	2.20	2.80	3.52	4.02	1.88	1.88	1.98

Other components common to all formulations were: active zinc oxide (5 phr), stearic acid (2 phr), sulfur (3 phr) and *N*-cyclohexyl-2-benzothiazyl sulfenamide (CBS; 1 phr)

[a]M_{100} and M_{200} mean the stress at 100% and 200% elongation, respectively

[b]Tensile strength

[c]Elongation at break

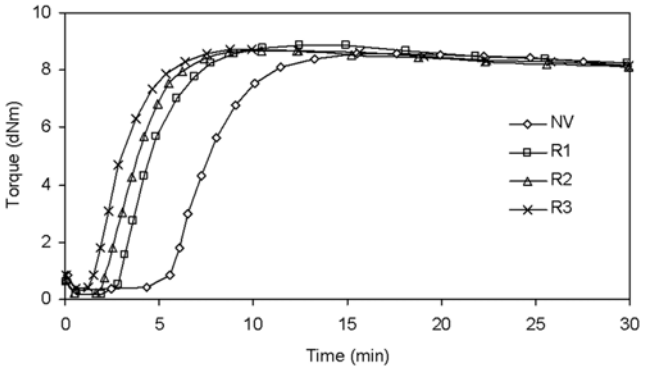

Fig. 2. Cure behaviors of NR vulcanizate (NV) and revulcanized rubber.

implied that the polysulfidic crosslinks were broken up, while the disulfidic or monosulfidic bonds may remain in the network.

2. Properties of the DVR Obtained by Devulcanization of NV Using Thiosalicylic Acid

2-1. Effect of DVR Content on the Curing Properties

The DVR obtained from section 1 was revulcanized with virgin NR at various proportions together with the curing reagents, as listed in Table 2. The cure behaviors of the resulting revulcanized rubber composites are presented in Fig. 2. Three regions of cure behaviors are clearly observed for all samples. The first region was the scorch time or the induction period, where the torque initially dropped due to the increase in the temperature, and remained low before the crosslink formation. The scorch time was found to slightly decrease with increasing DVR contents. This may be because the DVR still contained active crosslinks. In the second region, the torque rapidly increased due to the curing reaction, during which time the network structure was developed. The cure rate, obtained from the slope of the cure curve, also increased with increasing levels of DVR in the rubber composite. In the last region, when the equilibrium was reached, all samples attained a similar equilibrium torque and showed a similar slow rate of decrease thereafter with time, which was assumed to be attributed to the slow degradation of polysulfide bonds or so-called "reversion" [1]. Note that the equilibrium torques of the three revulcanized samples were comparable to that of the NV sample. Thus, the mechanical properties for three revulcanized DVR/NV composite samples should be comparable to those of the NV, which is discussed further in the next section.

2-2. Effect of DVR Content on the Mechanical Properties

The mechanical properties of the NR, and the DVRD3/NR and DVRD7/NR composite, after revulcanization, are summarized in Table 2, where the stress at 100% and 200% elongation (M_{100} and M_{200}, respectively) for all revulcanized samples was gradually increased with increasing devulcanized DVR rubber contents (R1-R7 samples) and were all higher than those of the NV when the proportion of DVRD3 in the DVRD3/NR composite was 20 phr or higher. The higher modulus observed with increasing DVR content in the vulcanized DVR/NR composite rubbers may be due to the higher crosslink density of the revulcanized rubber, but regardless is consistent with that previously described by Cavalieri et al. [30]. The tensile strength (T_B) and percentage of elongation at breaks (E_B) of the revulcanized rubber composites decreased with increasing levels of DVR in the composites. A significant change in the T_B and E_B values was observed when the DVRD3 content in the DVR/NR composite was 20 phr. However, the T_B values of the revulcanized rubber DVR/NR composites with a DVR composition of 5, 10 and 15 phr were slightly smaller than those of the NV rubber. This decrease in the T_B value is likely to be mainly caused by the low-molecular-weight component and by the polymer structural changes that occurred during the devulcanization process [16,31,32]. It is noteworthy that, in contrast, a 5-10% improvement in the E_B was obtained at a DVR content of 5-15 phr. The mechanical properties of the rubber obtained by the revulcanization using diphenyl disulfide (R8-R10) showed a similar, and dose-dependent, tendency as those using thiosalicylic acids (R1-R3).

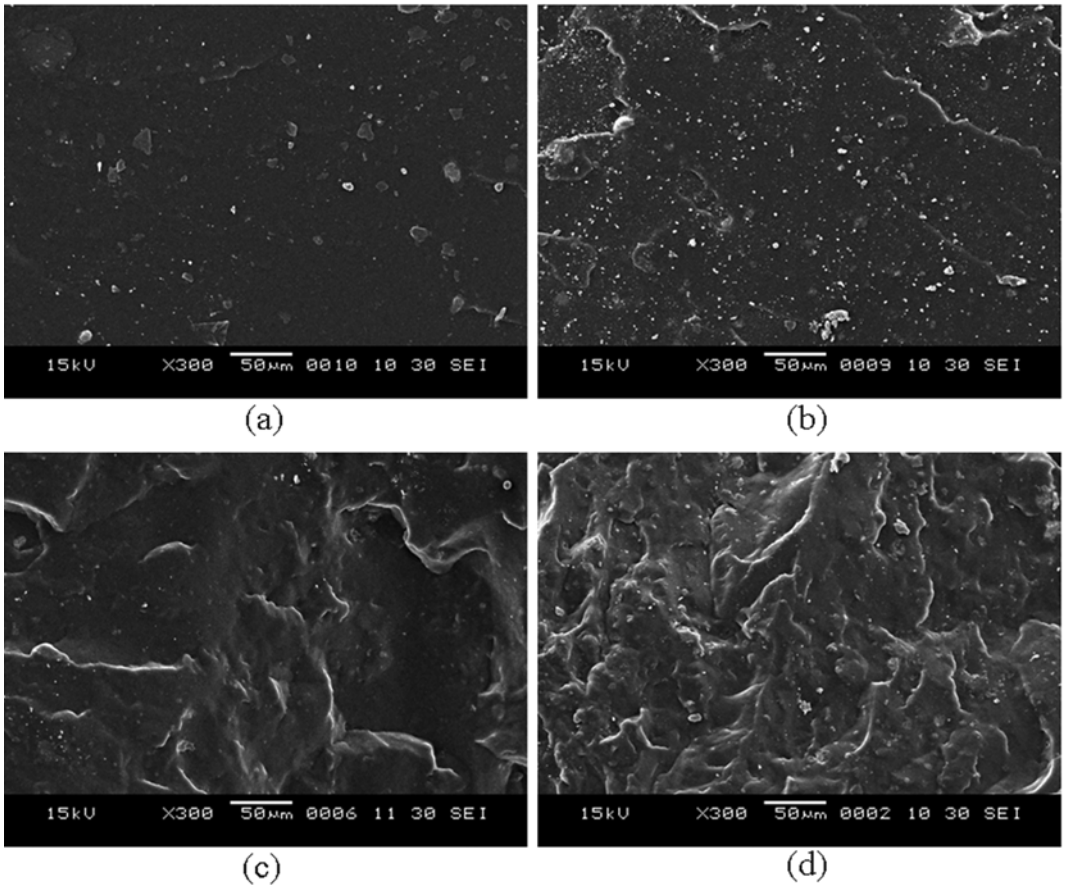

Fig. 3. Representative SEM photographs of the (a) NR vulcanizate (NV), (b) R1 (DVRD3/NR=5/95), (c) R3 (DVRD3/NR=15/85) and (d) R5 (DVRD3/NR=40/60).

The above explanation for the improvement of the mechanical properties of the revulcanized rubber was supported by the SEM images of cross-sections across the surface of the rubber, as shown in Fig. 3. The number of crack paths in different directions increased with increasing proportions of DVRD3 content in the vulcanized DVR/NR composite rubber. This result supports the notion that at the lower DVRD3 contents (5-15 phr), a higher compatibility between the virgin NR and DVR was obtained, resulting in the improvement in the mechanical properties of the resulting revulcanized composite rubber. Accordingly, the DVR prepared by using 1 phr thiosalicylic acid (DVRD3) can be practically used (recycled) at 5-15 phr with virgin NR to make revulcanized rubber that exhibits satisfactory mechanical properties when compared with those of NV rubber.

Table 3. Formulation of devulcanized truck tire rubber

Sample code	A	B	C
Truck tire rubber (phr)	100	100	100
Thiosalicylic acid (phr)	-	1	-
Diphenyl disulfide (phr)	-	-	1
Process oil (phr)	-	8	8
Pretreatment time (h)	-	48	48
Devulcanization temperature[a] (°C)	-	140	140
Sol fraction (%)[b]	9	30	29
Gel fraction (%)	91	70	71
Crosslink density $\times 10^4$ (mol/cm^3)	1.33	0.53	0.54

[a]at room temperature
[b]for 30 min

3. Devulcanization of a Truck Tire Vulcanizate

The devulcanization of a truck tire vulcanizate was evaluated as an example of a potential application of this method. The three formulations used for the devulcanization of truck tire compounds are shown in Table 3. Truck tire vulcanizates were devulcanized under the same conditions employed in the study on the devulcanization of NVs. The sol fraction and crosslink density of the truck tire DVR using 1 phr thiosalicylic acid (B) or diphenyl disulfide (C) were very similar to each other but significantly increased and decreased, respectively, over that seen in the truck tire rubber (A). These results indicate that thiosalicylic acid can also be used to destroy the three-dimensional network in vulcanized truck tires. Revulcanized truck tires were then prepared by mixing the virgin NR with the derived DVR from the truck tire, according to the formulations shown in Table 4.

The derived M_{200} values of the revulcanized composite rubber truck tires obtained from mixing NR with either truck tire rubber (A), or with DVR derived from thiosalicylic acid (B) or diphenyl disulphide (C), are summarized in Fig. 4. The results demonstrate that at the same virgin NR to truck tire DVR (w/w) ratio, the M_{200} values are in the range (ΔM_{200}=0.2 MPa) regardless of the pres-

Table 4. Formulation of revulcanized truck tire rubber

Sample code	A1	A2	A3	B1	B2	B3	C1	C2	C3
Virgin NR (phr)	95	90	85	95	90	85	95	90	85
A (truck tire rubber) (phr)	5	10	15	-	-	-	-	-	-
B (DVR-thiosalicylic acid) (phr)	-	-	-	5	10	15	-	-	-
C (DVR-diphenyl disulfide) (phr)	-	-	-	-	-	-	5	10	15
Crosslink density $\times 10^4$ (mol/cm^3)	1.91	1.97	2.09	2.02	2.14	2.19	2.00	2.11	2.18

Other components common to all formulations were: active zinc oxide (5 phr), stearic acid (2 phr), sulfur (3 phr) and *N*-cyclohexyl-2-benzothiazyl sulfenamide (CBS; 1 phr); whilst all samples were cured at 150 °C for 15 minutes

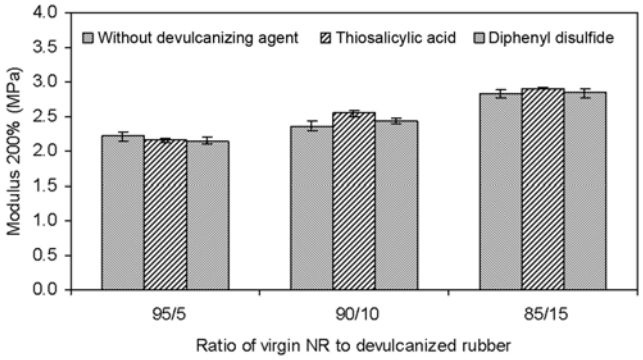

Fig. 4. The modulus 200% of revulcanized truck tire rubbers as a function of the different types of devulcanizing agent.

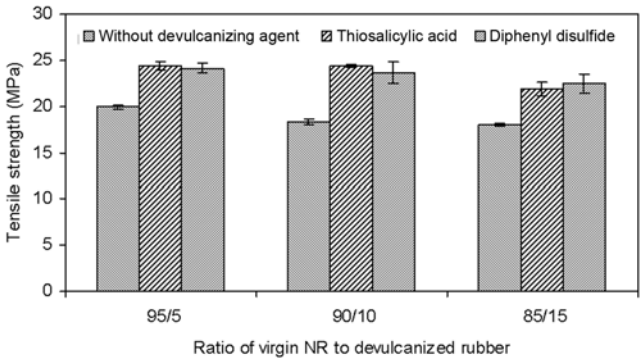

Fig. 5. The tensile strength of revulcanized truck tire rubbers as a function of the different types of devulcanizing agent.

ence or the absence of the devulcanizing agent, and that M_{200} tended to increase as the virgin NR to DVR (w/w) ratio decreased. The T_B and E_B values derived for the same revulcanized truck tire composite rubbers are illustrated in Fig. 5 and Fig. 6, respectively. The T_B value of the revulcanized truck tire rubber composites at the same virgin NR to DVR ratio was improved by 20-30% when the devulcanized rubber was treated with thiosalicylic acid (B1) or diphenyl disulfide (C1). However, the T_B value for the two DVR/NR composite rubbers at a DVR (w/w) ratio of 15% was lower than that at 5 and 10%. In a similar manner, the E_B value was increased by 5-10%. Thus, the implication is that the DVR acts as a reinforcing filler improving the mechanical properties of the revulcanized rubber [33]. These phenomena are in agreement with the observed trend in the case of E_B at all three virgin NR to DVR (w/w) ratios. It is interesting to note that the crosslink density for all revulcanized truck

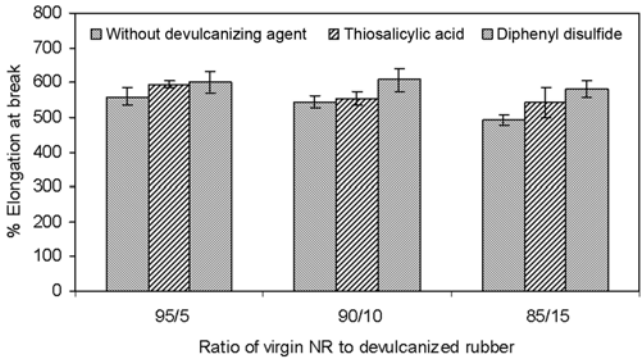

Fig. 6. The percentage of elongation at break of revulcanized truck tire rubbers as a function of the different types of devulcanizing agent.

tires was almost comparable (Table 4), and no significant effect of crosslink formation on the mechanical properties was observed. The improved mechanical properties were obtained from the inclusion of the DVR, formed from either devulcanizing agent, to the NR composite. Therefore, it is most likely that DVR derived from 1 phr thiosalicylic acid has a practical application for partial recycling of old NV when combined with virgin NR.

CONCLUSIONS

In this study, an NV was subjected to devulcanization by a mechano-chemical process in the presence of thiosalicylic acid as a devulcanizing agent. The optimum condition for the mechano-chemical process of the NR vulcanizate was the use of 1 phr thiosalicylic acid at 140 °C for 30 min. The sol fraction of DVR so obtained was significantly decreased compared with that of the NR vulcanizate. The efficiency of devulcanization by thiosalicylic acid under these conditions was comparable to that of the widely used diphenyl disulfide. The revulcanized rubber obtained from this process showed satisfactory mechanical properties. With the incorporation of thiosalicylic acid, an improvement in the E_B was obtained by the inclusion of DVR to 5-15% (w/w), due to the high compatibility between virgin NR and DVR, a notion which was supported by SEM cross-sectional images. The mechano-chemical process was also applied to devulcanize truck tire rubber, where this devulcanization condition affords good processability and can serve a practical use for the rubber industry towards partial recycling (environmentally and economically sound disposal) of used waste tires and to slightly reduce the requirement for new NR.

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