

Rheology, Cure Characteristics, Physical and Mechanical Properties of Tire Tread Reclaimed Rubber/Natural Rubber Compounds

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ABSTRACT: This work aimed to examine the effect of addition of tire-tread reclaimed rubber on the properties of two natural rubber (NR) compounds with respect to the reclaimed rubber concentration and mastication time, the properties of interest including rheological and cure characteristics, physical and mechanical properties. The results under the test conditions suggested that Mooney Plasticity and shear viscosity increased with reclaimed rubber content, but decreased with mastication time. The greater the molar mass of the natural rubber the higher the sensitivity to the change in compound viscosity due to mastication and reclaimed rubber content. The die swell was more dependent on the reclaimed rubber than the molar mass of the rubbers. The cure rate and scorch time were found to increase and decrease with reclaimed rubber content, respectively, whereas the cure time was independent of the reclaimed

rubber content. For vulcanized rubbers, it was also observed that 100% modulus of the rubber increased with reclaimed rubber content, but this was not the case for tensile stress and elongation at break. The hardness and heat buildup properties of the vulcanizates increased with reclaimed rubber content whereas the tear strength became independent of the reclaimed rubber. The findings in this work suggested that the variations in the rheological and cure characteristics for the unvulcanized rubber were very much dependent on the molar mass of the rubber whereas the mechanical properties for the vulcanized rubber were influenced by crosslink density. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 87: 1723–1731, 2003

Key words: rubber; recycling; fillers; crosslinking; vulcanization; rheology.

INTRODUCTION

International legislation and increasing environmental concerns have resulted in significant pressure to reduce and/or recycle automotive scrap. Automobile tire recycling into new tires or other products has been the focus of a number studies, and such pressures have also had significant implications for the rubber tire industry in the use of reclaimed rubbers from the tire scrap. Reclaiming allows the conversion of vulcanized rubber into new rubber compounds that can be recompounded into virgin polymers and then re-crosslinked. Reclaiming causes the fracture of long polymer molecules and this produces rubber with lower molecular weight while devulcanization involves the fracture of the crosslinks in the vulcanized rubber.¹ Recent technology to use the reclaimed and devulcanized rubbers in the virgin polymer materials

has attracted many rubber industries, and research on the issue is still limited in the literature.

Most work in the field have been carried out to investigate the cure characteristics and the products properties obtained by the incorporation of the reclaimed rubber, in various forms and treatment methods, into different virgin materials [mainly natural rubber (NR)]. Sreeja and Kutty² studied the cure characteristics and mechanical properties of NR/reclaimed rubber blends using an efficient vulcanization system. The maximum torque of the rubber blends increased with increasing the reclaimed rubber content. However, at higher reclaim loading, the effect of the filler present in the reclaim seemed to offset the increased plasticity. The scorch time and tensile properties of the blends reduced with the reclaimed loading. The reduced scorch safety with increased cure time indicated a lower rate of cure. Similar results and the explanations were given by Zhao et al.,³ who studied the effect of the waste rubber powder on the cure characteristics of styrene-butadiene rubber (SBR).

The cure characteristics are also affected by the vulcanization system. Theodore et al.⁴ blended devulcanized tire crumb rubber (DE-Vulc) with natural rubber

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using different vulcanization systems, including conventional vulcanization (CV), semi EV (closer to conventional), and Semi EV (closer to EV system). It was found that the scorch time of the rubber blends was shorter than that of the pure NR for CV and semi-EV closer to CV systems, but was longer for Semi EV closer to EV system. The cure time of the rubber blends was longer than that of the pure NR for CV, but was shorter for both Semi-EV systems.

Attempts to improve the properties of the reclaimed/virgin rubber compounds and vulcanizates have also been of interest to many researchers. De et al.⁵ used their own made reclaimed rubber (using diallyl disulfide as reclaiming agent) and blended with natural rubber. The cure characteristics were then examined. The scorch time and cure time were observed to reduce with the reclaimed rubber content due to the presence of active crosslinking sites in the reclaimed rubber that accelerated the crosslinking reaction. The increase in the extent of curing was noted due to the presence of rubber network and crosslink precursor in the reclaimed rubber. Kim and Park⁶ used the waste tire crumb rubber with/without a chemical treatment with di-(benzanidophenyl)-disulfide and blended with natural rubber. They found that the optimum cure decreased with increasing both treated and untreated crumb rubber contents. The chemical treatment could destroy the polysulfidic bond, and the sulfur content in treated rubber compounds were less than that in the untreated. The results also indicated that the cure time was delayed by treatment of the crumb rubber, the residual sulfur in the crumb rubber participating in the vulcanization process. Kim and Lee⁷ compounded crumb rubbers with various binders and found that greater tensile elongation was obtained when using the polyurethane binder system as compared with the use of SBR binder. Isayev et al.⁸ studied the mechanical properties of revulcanized SBR obtained after ultrasonic devulcanization. They found that at certain processing condition the tensile strength of revulcanized SBR was improved compared with that of the original vulcanizates. When adding a small amount of reclaimed silicone rubber into the SBR compound, the mechanical properties of the blended vulcanizates remained intact, and in some cases, were even better than those for the virgin SBR vulcanizates due to the formation of a continuous surface layer containing 100% silicone rubber.^{9,10}

In this present article, tire-tread reclaimed rubber with different concentrations (from 0–80%) was introduced into two natural rubber compounds (STRVS60 and STR20CV) using a sulfur CV system. The rheological (flow curves, elastic die swell) and cure characteristics (Mooney Plasticity, cure rate, scorch, and cure times) were then examined for various mastication times and reclaimed rubber contents. Physical

TABLE I
Initial Average Molar Mass for NRs
and Reclaimed Rubbers

Rubber Type	Mooney Plasticity No.	Average Molar Mass (10 ⁴ g/mole)
Natural rubber—STRVS60	53	9.14
Natural rubber—STR20CV	63	9.54
Reclaimed rubber—UCD103	69	6.97

properties of the vulcanizates were detected in terms of crosslink density and weight average molar mass which used Flory–Rehner equation and gel permeation chromatography (GPC), respectively. The mechanical properties of interest covered modulus, tensile stress, and elongation at break, tear strength, hardness, and heat buildup—these results being linked with the morphological structure.

EXPERIMENTAL

Raw materials

Natural rubber with two different grades (STRVS60 and STR20CV supplied by Tech Bee Hang Co., Ltd.) were used in this work, and each grade was blended with different concentrations of the UCD-103 grade of tire-tread reclaimed rubber, supplied by Union Commercial Development Co., Ltd. The reclaimed rubber comprised of 24% carbon black, 15% acetone extract, and 6% ash. The reclaimed rubber contents for the study ranged from 0 to 100% by weight. The initial Mooney Plasticity numbers [ML (1+4) 100°C] of STRVS60, STR20CV, and UCD-103 were measured in this work and listed in Table I.

Rubber mastication and compounding

The formulation of the NR compound, in parts-by-weight, was natural rubber 100, zinc oxide (ZnO) 5, stearic acid 2, MBT 0.5, DPG 0.2, and sulfur 3. There were two steps for making the NR compounds, these being mastication and compounding processes. In mastication, the natural rubber was masticated on a laboratory two-roll mill for various times (10, 20, or 30 min) followed by blending with a specified content of the reclaimed rubber for a further 10 min. On entering the compounding, the NR and reclaim blends were added and compounded with the prepared vulcanization chemicals on the two-roll mill for a further 10 min, the compounds being then kept at 25°C with 50% humidity prior to further use. The details of the compounding procedure are also found elsewhere.¹¹

Characterizations

Rheology

The wall shear stress (τ_w) and wall shear rate ($\dot{\gamma}_w$) of NR compounds were determined using a controlled-rate capillary rheometer, which had a barrel diameter of 26 mm and a die with L/D of 40/6. The calculations of shear stress and shear rate can be obtained elsewhere.¹¹ For purposes of material comparison, no Bagley's corrections to the wall shear stress were applied in this work.¹¹ For elastic properties, percentage die swell of the compounds during extrusion (from the rheometer) was measured using a direct method, whose procedure is discussed elsewhere.¹²

Cure characteristics

Mooney Plasticity number of the rubber compounds was measured using a TECHPRO Mooney viscometer, model VisTECH, whose testing conditions being in accordance with ASTM D1646-96a (1996). The cure rate, scorch time, and cure time of the rubber compounds were detected using a Monsanto Oscillating Disk Rheometer (model MDR 2000) at a test temperature of 150°C.

Physical properties

The average molar mass of the reclaimed rubber, NRs, and their blends before and after mastication were determined using GPC, tetrahydrofuran (THF) being used as the solvent. The initial molar mass of the natural rubbers and reclaimed rubber are listed in Table I.

The determinations of crosslink density of the vulcanized rubber compounds were carried out using a swelling method.⁷ The experimental procedure was commenced when the vulcanized rubber samples were cut into small pieces and then weighed before being immersed into toluene. The samples were kept in a dark place for 7 days. Excess liquid on the surface of the specimens was removed by blotting with filter paper. The swollen samples were taken out and placed to dry at a controlled temperature of 21°C. The weight of the swollen samples was measured and the crosslink density was calculated by using the Flory-Rehner equation, the details of the calculations can be found elsewhere.⁷

$$-\ln(1 - V_r) - V_r - \chi V_r^2 = 2V_s \eta_{\text{swell}} \left(V_r^{1/3} - \frac{2V_r}{f} \right) \quad (1)$$

where V_r is volume fraction of rubber in swollen gel, V_s is molar volume of the toluene (in this work being 106.2 cm³/mole), χ is the rubber-solvent interaction parameter (in this case being 0.3795), η_{swell} is the crosslink den-

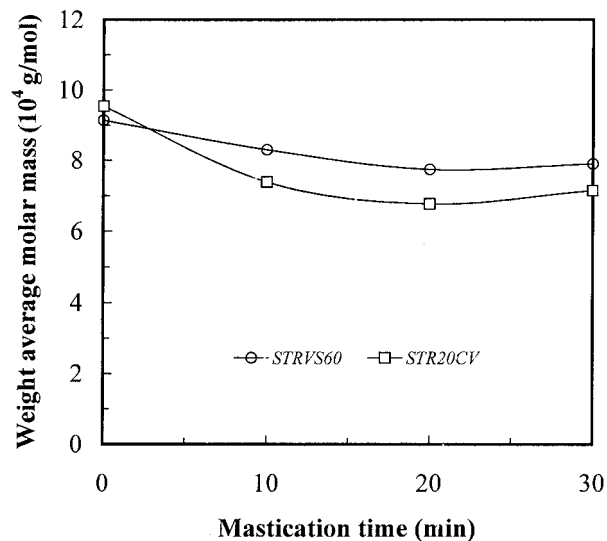


Figure 1 Variation of molar mass with mastication time.

sity of the rubber (mole/cm³), and f is functionality of the crosslinks (being 4 for the sulfur curing system).

Mechanical properties

The tensile properties of the rubber vulcanizates were tested according to ISO 37 (1994) with use of dumb-bell-shaped samples (type 1), the tests being carried out using the Instron tensile testing machine model-1011. Tear strength was determined according to ASTM D624-98 (1998) using angle-shaped samples and a LLOYD tear strength testing machine. Both tensile and tear properties used a testing speed of 500 mm/min. A durometer transducer (type A) of Shore Instrument & M.F.G., model PN71500, was used for hardness test, the test condition being in accordance with ASTM D 2240-97 (1997). A Goodrich Flexometer by Ferry Industry Inc., model 580, was utilized to measure the temperature rise (heat buildup) in the rubber specimens, which were produced in cylindrical shape, being 17.8 mm in diameter and 25 mm in length, according to ASTM D623-78 (1988).

Morphology

The failure mechanisms were investigated using a JEOL scanning electron microscope (SEM) (model JSM-6301F) at 15 kV accelerating voltage, examining fractured surfaces of rubber vulcanizates obtained after immersion in liquid nitrogen.

RESULTS AND DISCUSSION

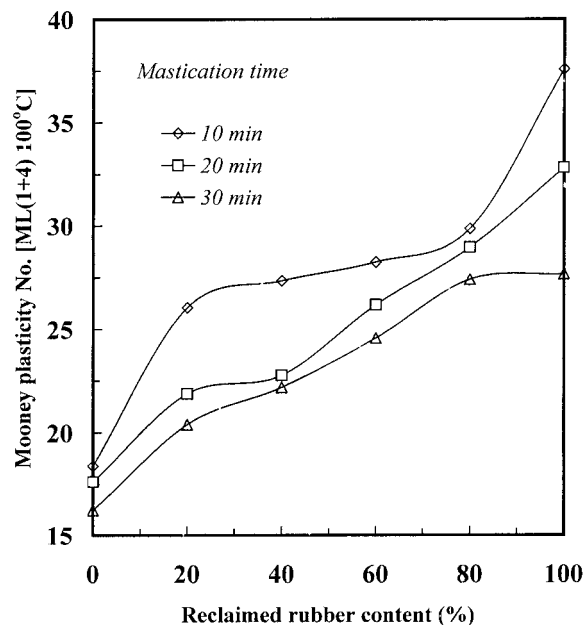
Unvulcanized rubbers

Figure 1 shows the relationship between the average molar mass of STRVS60 and STR20CV rubbers. The

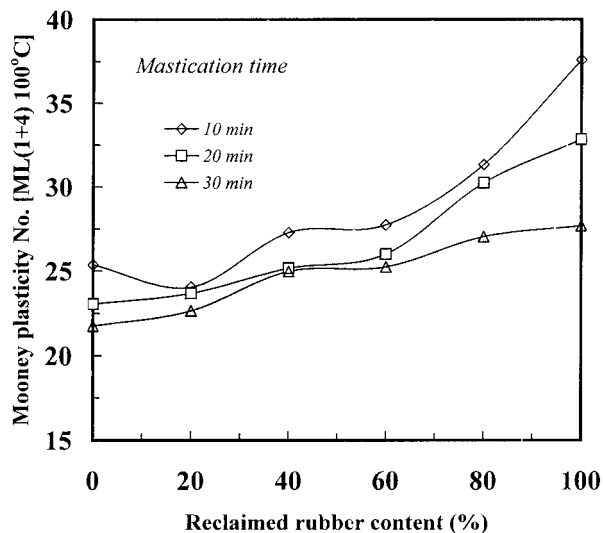
results suggested that the weight average molar mass of the rubbers decreased with mastication time up to 20 min, and then stabilized. Similar results are also found in previous work.¹¹ It was interesting to observe that the STR20CV was more sensitive to the mastication than the STRVS60, this being due to greater molar mass. After mastication, the STRVS60 had greater molar mass than STR20CV. It should therefore be noted that the STRVS60 had greater average molar mass than the STR20CV throughout this work because all the rubbers were prepared using the mastication time of 10 min.

Figures 2(a) and 2(b) show the Mooney Plasticity number of the STRVS60 and STR20CV rubbers with different reclaimed rubber contents and mastication times. As the mastication time was increased, the Mooney Plasticity reduced for both NR grades, this being due to the reduction of the molar mass of the polymers as stated above. The magnitude of the reduction in Mooney Plasticity due to the mastication was different between the two natural rubber compounds. It was also noticeable that the mastication time had more pronounced effect on the change in the Mooney Plasticity for STRVS60. Considering the effect of reclaimed rubber content, it was observed that the higher the reclaimed rubber content the higher the Mooney Plasticity. Two reasons were addressed here; First, the reclaimed rubber in this work contained some residual carbon black, which probably takes part in the increase of the compound viscosity—the more the reclaimed rubber the higher the carbon black content. Second, Table I indicates that the reclaimed rubber used in this work had a greater Mooney Plasticity number than the NR compounds. Therefore, incorporating the reclaimed rubber would result in an increase of the overall Mooney Plasticity of the compounds.

Figures 3(a) and 3(b) show the relationship between the wall shear stress and wall shear rate of the NR compounded with various reclaimed rubber contents, using 10 min mastication time). It can be seen that in all cases the compounds exhibited the pseudoplastic non-Newtonian character, the shear viscosity (note that the shear viscosity being referred as to the ratio of the shear stress to the shear rate) of the compounds decreasing with shear rate as expected.¹¹ Inclusion of the reclaimed rubber resulted in an increase of the overall shear stress (or viscosity). These results corresponded well with the Mooney viscosity, as discussed earlier. However, the magnitude of the increase in the compound viscosity was different between these two compounds. The changes in the wall shear stress (and shear viscosity) for STRVS60 seemed to be more sensitive to the reclaimed rubber content than that for STR20CV, this again being similar to the Mooney results.



(a)

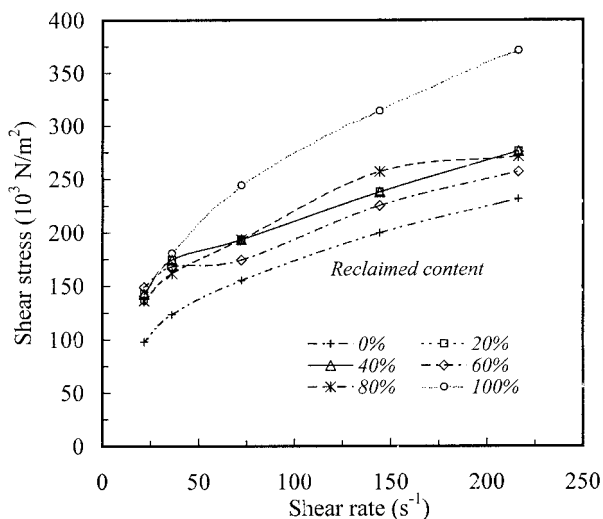


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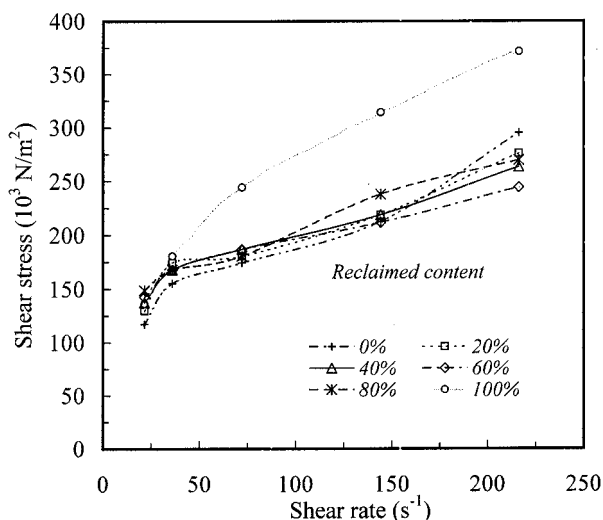
Figure 2 Effect of reclaimed rubber on Mooney Plasticity number for different mastication times. (a) STRVS60 and (b) STR20CV.

It was postulated and summarized here that the sensitivity of the change in the compound viscosity due to mastication time and reclaimed rubber content was greatly dependent on the molar mass of the polymers—the greater the molar mass the greater the sensitivity to the change of the compound viscosity.

Figure 4 shows a relationship between die swell and reclaimed rubber content. Without the reclaimed rubber, the die swell for STR20CV was higher than that for STRVS60, and the die swell sharply decreased especially around the concentrations of 20–60%. This was because of the carbon black present in the re-



(a)



(b)

Figure 3 Plots of wall shear stress and wall shear rate at 80°C using 10 min mastication time. (a) STRVS60 and (b) STRCV20.

claimed rubber. The carbon black is known as the most effective reinforcing filler for rubbers. Introducing carbon black into the rubber results in a restriction of the rubber-chain mobility and this reduces the elastic characteristics of the compound.¹³ Increasing the reclaimed rubber would automatically increase the carbon black content to the blend, and thus the decreased die swell. The sudden drop in the percentage die swell also implied that there was some molecular interactions between the rubber molecules and the carbon black in the reclaimed content around 20–40%, the molecular interactions suppressing the swelling.¹⁴ Comparing the die swell results of the two rubber compounds, one would expect to obtain a difference in the percentage die swell for any given reclaimed rubber content, because of different molar masses of

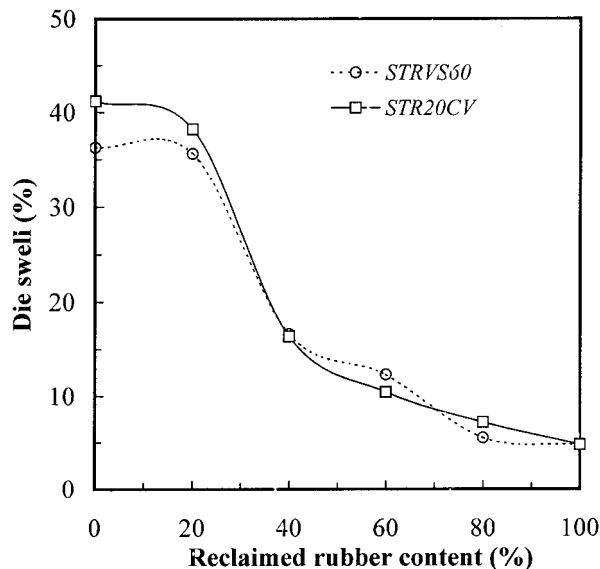


Figure 4 Percentage die swell and reclaimed rubber content for STRVS60 and STR20CV at a test temperature of 80°C, 10 min mastication, and a shear rate of 3.6 s⁻¹.

these two polymers. However, this was not the case, no considerable difference being observed. These results suggested the residual carbon black in the reclaimed rubber had greater effect on the change in die swell (or elastic property) than the molar mass of the rubbers.

Figures 5 and 6 show the results of cure rate and scorch time of the two NR grades blended with different reclaimed rubber contents. It should be noted that the cure time of the two NR compounds did not change with the addition of the reclaimed rubber (thus the results not shown). In Figure 5, it was found that the cure rate increased with increasing the reclaimed

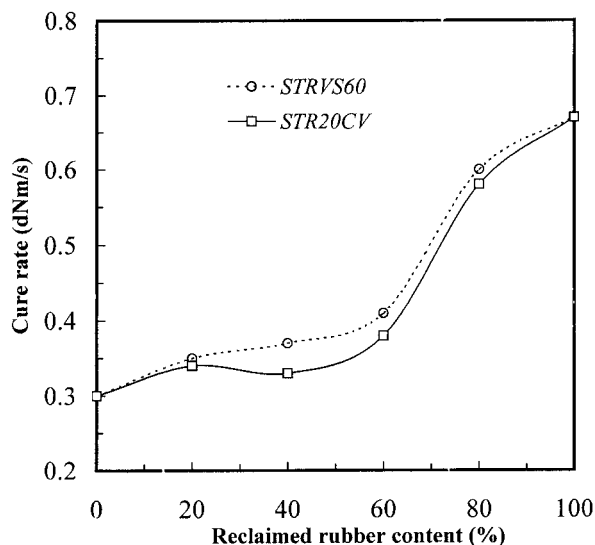


Figure 5 Relationship between cure rate and reclaimed rubber content for STRVS60 and STR20CV compounds.

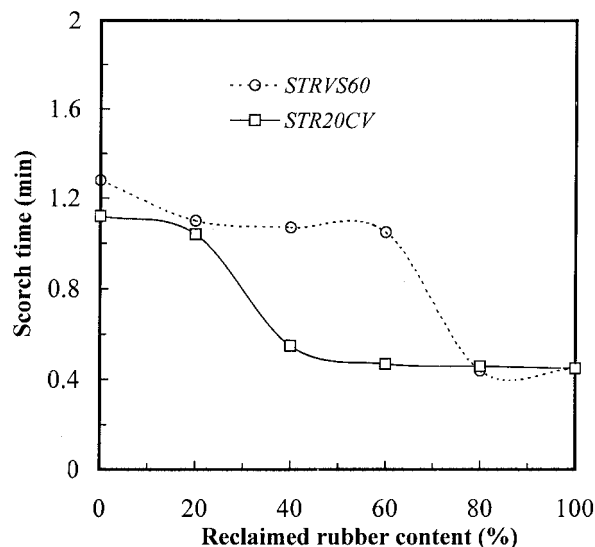


Figure 6 Relationship between scorch time and reclaimed rubber content for STRVS60 and STR20CV compounds.

rubber, especially around 60% reclaimed rubber content. The increase in the cure rate may involve the crosslink precursor or untreated curatives present in the reclaimed rubber, which would accelerate the crosslink formation rate in the compounds. Around 20–60% reclaimed rubber, a significant difference in the cure rate between STRVS60 and STR20CV compounds was observed, the cure rate of the STRVS60 being higher. This could be explained by considering the results in Figure 6, which illustrated the effect of the reclaimed rubber content on the scorch time. An obvious difference in the scorch time of these two NRs around 20–60% reclaimed rubber was markedly seen. The decrease in scorch time resulting from increasing reclaimed rubber content was due to the presence of active crosslinking sites in the reclaimed rubber.⁵ The relationship between the cure rate, scorch time, and cure time was that compounds with longer scorch time had to increase (accelerate) the rate of cure, in order to achieve the same cure time (because the cure time did not change with reclaim as stated earlier). This was why the STRVS60 had greater cure rate than the STR20CV.

Rubber vulcanizates

Figure 7 shows the modulus at 100% elongation of the NR vulcanizates with various reclaimed rubber contents. It was found that the modulus of both NR vulcanizates increased with increasing reclaimed rubber content. This was due to two possible reasons, one being an increase in crosslink density of the compounds and the other being restrictions of molecular mobility. The former can be explained using the crosslink density results, which are shown in Figure 8. It was clear that addition of reclaimed rubber in-

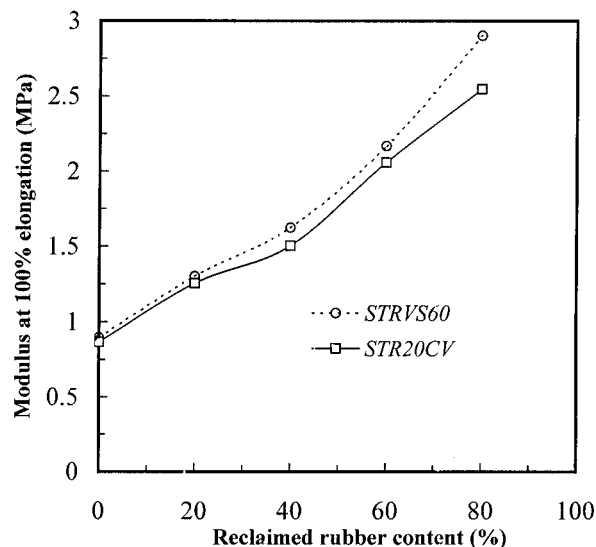


Figure 7 Variation of modulus at 100% elongation with reclaimed rubber content for STRVS60 and STR20CV vulcanizates.

creased the crosslink density of the compounds, with no differences in crosslink density between STRVS60 and STR20CV blends for any given reclaimed rubber content. For the latter it was stated earlier that addition of the reclaimed rubber to NR compounds would automatically increase the carbon black, which acted as an effective reinforcing filler, this resulting in restriction of the rubber molecular mobility under tension force, and hence increased modulus. Figure 9 shows the effect of reclaimed rubber content on the tensile stress and elongation at break of STRVS60 and STR20CV vulcanizates. Both tensile stress and elongation at break decreased progressively with reclaimed rubber content, similar results being given by other

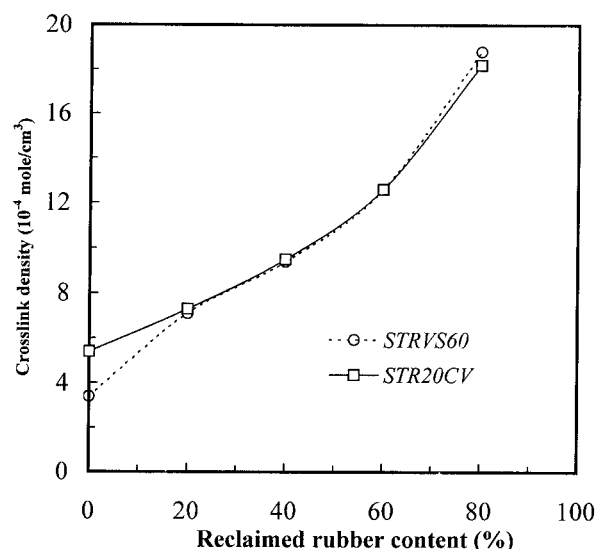


Figure 8 Effect of reclaimed rubber content on crosslink density for STRVS60 and STR20CV vulcanizates.

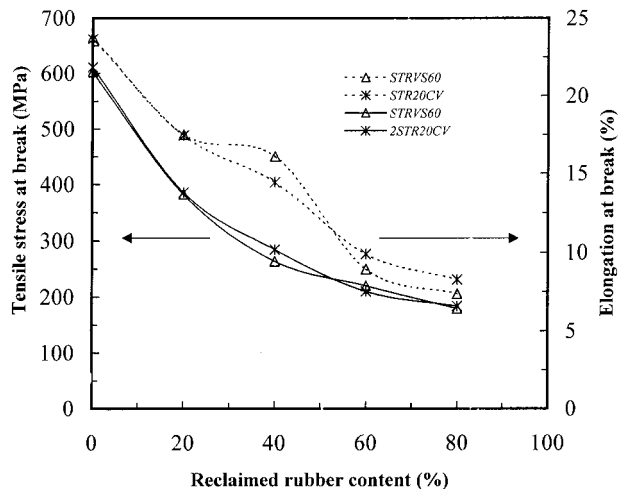


Figure 9 Variations of tensile stress and elongation at break at various reclaimed rubber contents for STRVS60 and STR20CV vulcanizates.

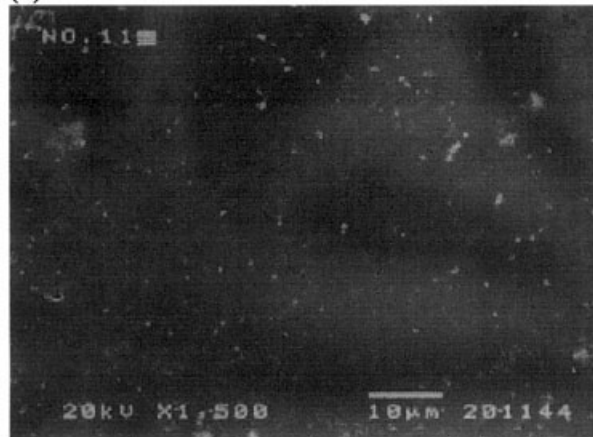
researchers.^{2,4} The decrease in tensile stress and elongation at break can be explained by two synergetic reasons as follows:

1. *Homogeneity of the Blends:* The homogeneity of the rubber vulcanizates can be considered using SEM micrographs. Figures 10(a), 10(b), and 10(c) show SEM micrographs of fractured surface for STRVS60, reclaimed, and STRVS60/reclaim blend of 80/20%, respectively. The micrographs indicated that the fractured surface of the blend had less homogeneity than that of pure NR compounds. With presence of reclaimed rubber, the phase boundary occurring may probably produce defects between the rubber molecular structure, and this then led to a reduction in the tensile stress and elongation of the vulcanizates.
2. *Restriction of Molecular Mobility:* The carbon black present in the reclaimed rubber may inhibit the molecular orientations and mobility of the rubber, thus causing the sample to fail at low elongation.²

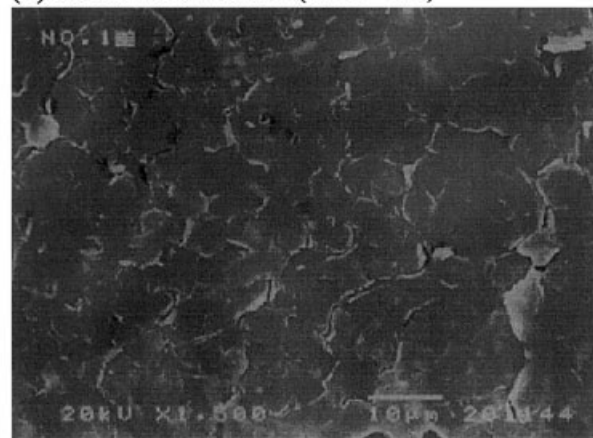
Figure 11 illustrates the tear strength of the rubber vulcanizates having various reclaimed rubber contents. Tear strength of the vulcanizates became independent of reclaimed rubber content from 0–60%, the tear strength then sharply decreasing at 80% reclaimed rubber loading. Increasing the reclaimed rubber also led to increases in hardness, whose results are shown in Figure 12. The increase in hardness of the vulcanizates were possibly caused by the presence of a small amount of SBR since the reclaimed rubber used in this work was derived from tire treads.¹ The hardness results was best explained by considering the onset of degradation temperatures of the NR compounds (STRVS60 and STR20CV) and reclaimed rub-

ber, which were carried out using Perkin-Elmer TGA-7 with a temperature range of 50–650°C and a heating rate of 20°C/min. The TGA results are shown

(a) STRVS60



(b) Reclaimed rubber (UCD-103)



(c) 20% STRVS60 with 80% Reclaimed rubber



Figure 10 SEM micrographs of rubber samples. (a) STRVS60, (b) reclaimed rubber, and (c) 20% STRVS50 with 80% reclaimed rubber.

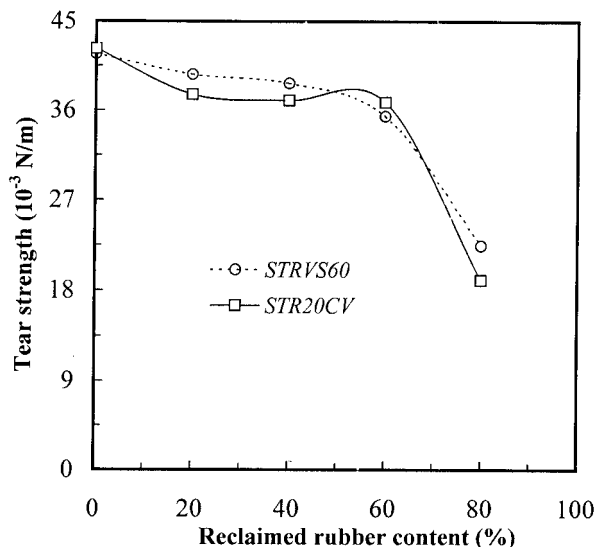


Figure 11 Tear strength of rubber vulcanizates at different reclaimed rubber contents.

in Figure 13. The STRVS60 and STR20CV had very similar values at the onset of degradation temperature, these being 370.14 and 368.41°C, respectively. For reclaimed rubber, there were two degradation temperature peaks, one being around 364.58°C and the other being 441.22°C. The former temperature was for NR compounds, as it was close to the values of STRVS60 and STR20CV, while the other arose from SBR content in the compound. Therefore, the presence of SBR content would probably involve the increase in the hardness of the blends.

Figure 14 shows the results of temperature rise (heat buildup) in the rubber vulcanizates having various reclaimed rubber contents. It was clearly seen that the heat buildup increased with increasing reclaimed rubber. As the stiffness of the vulcanizates increased, due to the carbon black in the reclaimed rubber, the molecular mobility of the rubber became more restricted. This would then generate enhanced frictions between the rubber chains, thus giving rise to an increase in heat built up in the blends, similar results being also found in literature.²

Supplemental comments

In summary, regardless of the effect of reclaimed rubber content, it was worth noting that in *unvulcanized rubber*, the rheological and cure characteristics were very much dependent on the initial weight average molar mass of the rubbers used. The molar mass became insignificant to the performance of the rubber products after the vulcanization process. All mechanical properties of the STRVS60 and STR20CV *vulcanizates* for any given reclaimed rubber contents were very similar because these two vulcanized blends had

the same crosslink density. Therefore, it can be concluded that the crosslink density was the main factor that influenced the mechanical properties of the rubber vulcanizates in the case where different natural rubbers were used. Considering a relationship between molar mass and crosslink density of the rubbers, it seemed that such relationship was complex. The overall results reported in this work have clearly suggested that the cure characteristics (cure rate and scorch time) had to be taken into account, although the same vulcanization recipe was used. However, no further attempts have been made in this present work.

CONCLUSION

Tire-tread reclaimed rubber was introduced into two grades (STRVS60 and STR20CV) of natural rubber, and the properties of unvulcanized and vulcanized rubbers were examined. The following were noted:

- General findings in this paper suggested that the variations in the rheological and cure characteristics for the unvulcanized rubber were found to be dependent on the molar mass of the rubber, whereas the mechanical properties for the vulcanized rubbers were influenced by crosslink density.
- For unvulcanized rubber compounds: It was found that Mooney Plasticity and shear viscosity increased with reclaimed rubber content, but decreased with mastication time. The greater the molar mass of the natural rubber the higher the sensitivity to the change of the compound viscosity due to mastication and reclaimed rubber content. The die swell was found to be more dependent on the reclaimed rubber than the molar mass of the rubbers. The cure rate and scorch time were

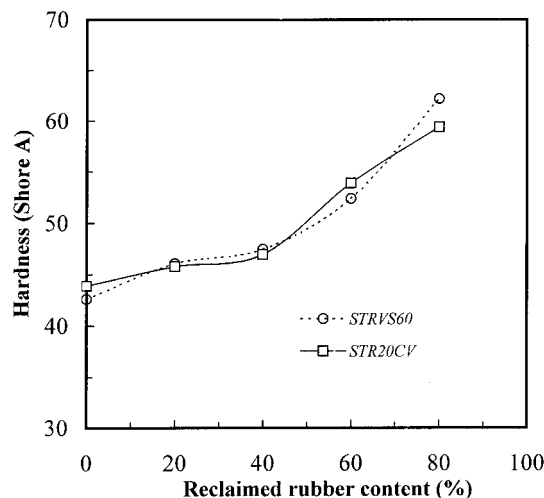


Figure 12 Hardness property of rubber vulcanizates at different reclaimed rubber contents.

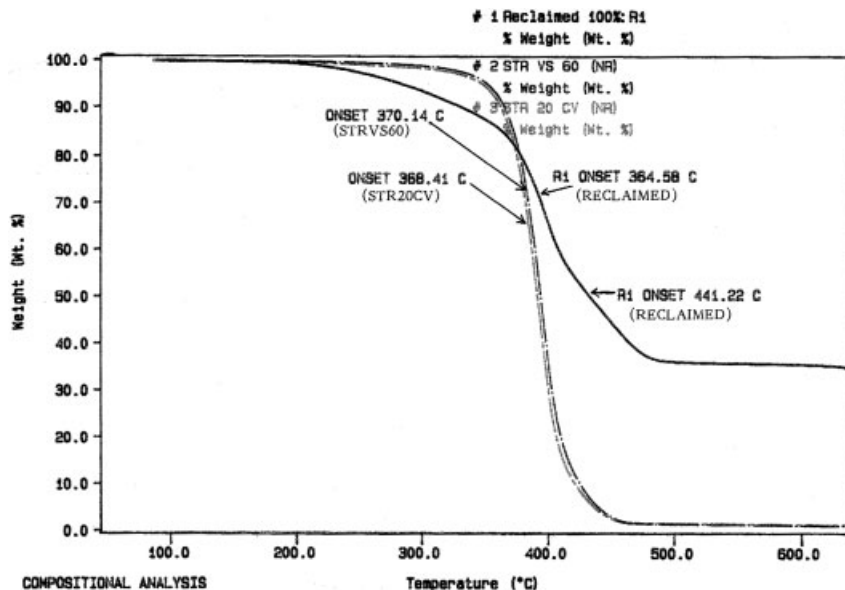


Figure 13 The onset of degradation temperature for STRVS60, STR20CV and reclaimed rubber using TGA.

found to increase and decrease with reclaimed rubber content respectively, whereas the cure time was independent of the reclaimed rubber content. The changes in the rheological and cure characteristics were mainly associated with the crosslink precursor or untreated curatives, and the amount of carbon black present in the reclaimed rubber.

- For vulcanized rubber compounds: It was observed that the modulus at 100% elongation of the rubber increased with reclaimed rubber content, but this was not the case for tensile stress and elongation at break. The hardness and heat buildup properties of the vulcanizates increased

with reclaimed rubber content whereas the tear strength was independent of the reclaimed rubber. The variations of the mechanical properties of the vulcanizates involved the changes in crosslink density, homogeneity of the blends, and the molecular mobility due to the presence of carbon black and SBR in the reclaimed rubber.

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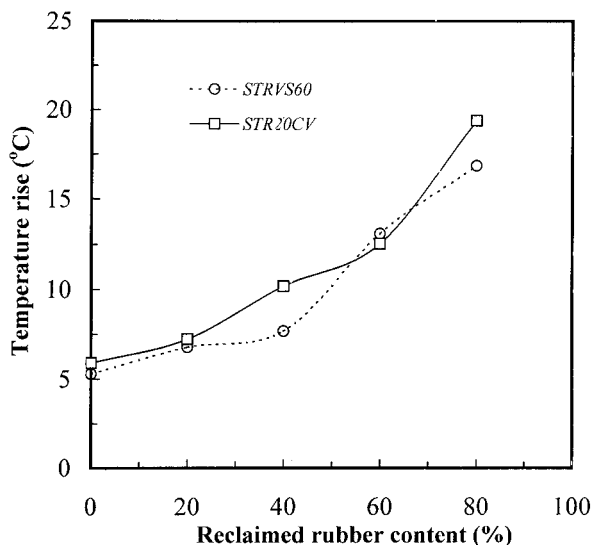


Figure 14 Effect of reclaimed rubber content on temperature rise for STRVS60 and STR20CV vulcanizates.