

Comparison of Physical and Mechanical Properties of NR/Carbon Black/Reclaimed Rubber Blends Vulcanized by Conventional Thermal and Microwave Irradiation Methods

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ABSTRACT: This work experimentally monitored the physical and mechanical properties of natural rubber/carbon black/reclaimed rubber blends vulcanized by microwave (MW) irradiation and conventional thermal (CT) systems, and the curing mechanisms are addressed in conjunction with the vulcanizate properties. The microwave oven for rubber blend vulcanization was specially designed and constructed in this work. The results suggested that the tensile modulus and the hardness of the vulcanizates could be enhanced by increasing the reclaimed rubber (RR) content, whereas the tensile stress, elongation at break, and tear strength decreased with the RR loading. The mechanical properties of the vulcanizates from the CT method were higher than those from the MW method. The vulcanizates

from the MW method had higher resistance to toluene penetration than those from the CT method. The differences in the properties of the vulcanizates from the CT and MW methods became smaller at higher RR contents. The sensitivity in property changes of the vulcanizates due to addition of the RR content was greater for the CT method than for the MW method. The differences in the results between these two curing systems could be explained in terms of the density and the types of crosslinks present in the vulcanizates. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 5039–5048, 2006

Key words: natural rubber; reclaimed rubber; mechanical properties; swelling; microwave

INTRODUCTION

The use of rubbers in various applications has resulted in a growing volume of rubber wastes, especially in the automobile industry. Increasing environmental concerns have resulted in significant pressure to reduce and recycle automotive tires. Conversion of automotive tire wastes into reclaimed rubbers (RR) has possibilities in developing new products, and has now become the focus of research studies among rubber technologists. The recycling of the rubber wastes mainly involves reclamation and devulcanization processes: the reclamation process causing fracture of long polymer molecules to produce rubber with lower molecular weight (RR) and the devulcanization process causing fracture of chemical crosslinks in the vulcanized rubber.¹ Recent technology allowing the use of RR in virgin polymer materials has attracted

many rubber industries. Most works in this field have investigated the cure characteristics and product properties obtained by the incorporation of the RR, in various forms, into different virgin materials (mainly natural rubber (NR)). Sombatsompop and Kumnuantip^{2,3} introduced tire-tread RR into two grades of NR and investigated various properties of the blends. They found that the Mooney number, shear viscosity and cure rate increased with RR content, while the cure time was independent of it. The property changes of the reclaimed/NR blends were mainly associated with the crosslink precursor or untreated curatives, and the amount of carbon black (CB) present in the RR. Sreeja and Kutty⁴ studied the cure characteristics and mechanical properties of NR/ RR blends, using the EV system. They observed that the scorch time and tensile properties of the blends reduced with the RR loading. Nevatia et al.⁵ blended RR with recycled PE and studied the physical properties, dynamic mechanical properties, and rheological behaviors of the blend. They found that a 50:50 rubber/PE ratio gave optimum processibility, ultimate elongation, and set properties. The use of a sulfur-accelerator cured system was found to give better product properties than a peroxide-cured system.

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It is widely known that the properties of vulcanizates are very much dependent on the conditions under which the green rubbers are cured, these including cure time and temperature, design of vulcanizing recipes, as well as the type of curing system. The last factor is usually referred to as the method used for curing the rubber compounds. The simplest curing system used in the rubber industry is direct heating (conventional thermal curing). Many published works have suggested that the duration and mold temperature are the main parameters that control the degree and type of crosslinks to be formed, and these factors directly affect the properties of the end products. Microwave curing has increasingly become one of the more interesting curing systems in many applications, such as food industries, dried-wood systems, concrete curing, and shape-setting of thermosets and rubbers.⁶ The main advantages of the microwave cure include faster curing times, improved efficiency of curing, and enhanced mechanical properties of the products.⁷ Zhou et al.⁸ studied the effects of curing systems on the properties of epoxy resin, with different proportions of maleic anhydrides and curing agents, using microwave and thermal cures. The results showed that the microwave curing could increase the mechanical properties of the resin, with reduction in cure time and curing agent. Zhou and Hawley⁹ used a cylindrical single-mode applicator for microwave adhesive bonding. An epoxy-based adhesive was used to bond the two polymeric materials, and the results were compared with those cured by a thermal process. They found that microwave curing reduced the bonding time dramatically, which resulted from faster curing of the adhesive.

To date, subjects on the effects of microwave curing on the changes in vulcanized rubber properties in comparison with those obtained by conventional thermal methods are still open for discussion, since this information is rare in literature, especially when some secondary materials such as RR and CB are incorporated. In this work, NR was blended with various contents of tire-tread RR (RR). A fixed amount of 60 phr CB was loaded into the NR-RR blends as reinforcement filler. The rubber blends were vulcanized by two different methods: the microwave irradiation method (MW method) and the conventional thermal method (CT method). The physical and mechanical properties of the blended vulcanizates obtained from the MW and CT methods were compared and discussed with respect to the RR contents.

EXPERIMENTAL

Raw materials

The NR used in this work was STR20CV, supplied by Tech Bee Hung Co., Ltd. (Bangkok, Thailand), and the

tire-trade RR was UCD-103 grade, supplied by Union Commercial Development Co., Ltd (Samutsakorn, Thailand). The initial Mooney Plasticity numbers [ML (1 + 4) 100°C] of the STR20CV and UCD-103 rubbers were 63 and 69, respectively. The average molecular weights of STR20CV and UCD-103 were 9.5×10^4 and 7.0×10^4 g/mol, respectively. The RR is composed of 24% CB, 15% acetone extract, and 6% ash. The RR contents for the study ranged from 0 to 60% by weight. The NR and the RR were blended together in various NR:RR ratios of 100:0, 85:15, 70:30, 64:45, and 40:60. For effective microwave adsorption, the rubber blends were also loaded with 60 phr CB (N330), which was supplied by Thai Carbon Black Co., Ltd. (Bangkok, Thailand). It should also be noted that CB is necessary when curing by microwave, since the NR is itself a low-dielectric loss material that does not react with microwave. Therefore, to achieve effective curing, CB needs to be added because CB has a relatively high dielectric loss,¹⁰ and this will increase the dielectric loss property of the blend and thus be induced by microwave irradiation.

Rubber compound preparation

A conventional vulcanization (CV) formulation was used in this work. The formulation of the rubber compounds is detailed as follows: 100 phr blended rubber (NR:RR; 100:0, 85:15, 70:30, 64:45, and 40:60 by weight), 60 phr CB, 5.0 phr zinc oxide (ZnO), 2.0 phr stearic acid, 0.5 phr mercaptobenzthiazole (MBT), 0.2 phr diphenylguanidine (DPG), and 3.0 phr sulfur. In the mastication step, the NR was masticated on a laboratory two-roll mill (Yong Fong Machinery Co., Ltd., Samutsakon, Thailand) for 5 min and was then blended with the tire-trade RR for another 5 min. In the compounding step, the rubber and filler were compounded with prepared vulcanization chemicals on the two-roll mill for another 10 min, and the compounds were then kept at 25°C at 50% humidity before further use.

Design and construction of microwave vulcanization oven

In this present work, a microwave vulcanization oven was specially designed and manufactured for curing all the blends of NR/CB/RR. The schematic diagram of the microwave vulcanization oven is shown in Figure 1. The microwave vulcanization oven consisted of a microwave generator (magnetrons), mold, waveguides, stirrer blades, magnetron blowers, oven control connector, and an oven control unit. The microwave oven body was made from stainless steel. The oven cavity size was 30 cm high, 40 cm wide, and 50 mm long. The mold used for the production of rubber sheet of 2 mm thick was made of Teflon. Four

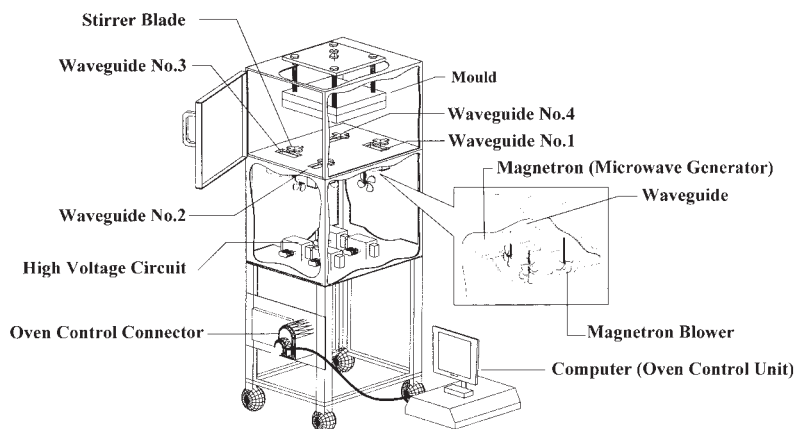


Figure 1 Design and construction of microwave oven for rubber vulcanization.

magnetrons were used for effective and uniform heating during the vulcanization, and they were located at the base of the oven for good microwave distribution. Each magnetron gave a microwave irradiation power of 1000 W, and the irradiation powers of the magnetrons were adjusted using an oven control unit. The microwaves were transferred from the magnetrons to the oven cavity by passing through four waveguides, which were rectangular in shape (900 mm wide, 80 mm deep, and 40 mm high). The stirrer blades, located above the waveguide, were utilized to improve the distribution of the microwave around the oven for uniform heating.

Vulcanization methods

Two vulcanization methods were used in this work for curing the rubber blends, and these were the microwave irradiation (MW) method, and the conventional thermal (CT) method.

- For the MW method, the resultant rubber compounds were vulcanized in a microwave vulcanization oven. The resultant rubber compounds were placed in the microwave apparatus. The temperature of the vulcanizates was determined using an IR probe immediately after stopping the microwave irradiation. The cure temperatures used for this method and for the CT method were the same for any given RR content (145°C for the NR–CB compounds without RR, and 150°C for those with RR). The trial and error experiments determined the cure time for the MW method at 3 min; a longer cure time caused over-curing of the rubber compounds.
- For the CT method, the resultant rubber compound was compression-molded using a hydraulic press (LAB TECH Co., Ltd., Bangkok, Thailand) at 170 kg/cm². For comparison purposes,

the cure time was fixed at 3 min for all rubber compounds. The vulcanized temperatures were 145°C (for nonreclaimed compounds) and 150°C (for NR and RR blended compounds).

Vulcanizate characterizations

Physical properties

Crosslink density. The crosslink density of the vulcanized rubbers from both methods was determined using a swelling method.² The vulcanized rubber samples were cut to small pieces and then weighed before being immersed in 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 dried 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).²

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

where V_r is the volume fraction of rubber in swollen gel, V_s is the molar volume of the toluene (in this work it is 106.2 cm³/mol), χ is the rubber–solvent interaction parameter (in this case is 0.3795), η_{swell} is the crosslink density of the rubber (mole/cm³), f is the functionality of the crosslinks (4 for sulfur curing system).

Vulcanizate swelling behavior. A sorption–desorption method was used to determine the swelling behavior of the vulcanizates, and this was demonstrated as the mol % uptake of toluene by 100 g of rubber at 25°C. The rubber sample was cut to square shape, which was 20 mm in length, 20 mm in width, and 2 mm

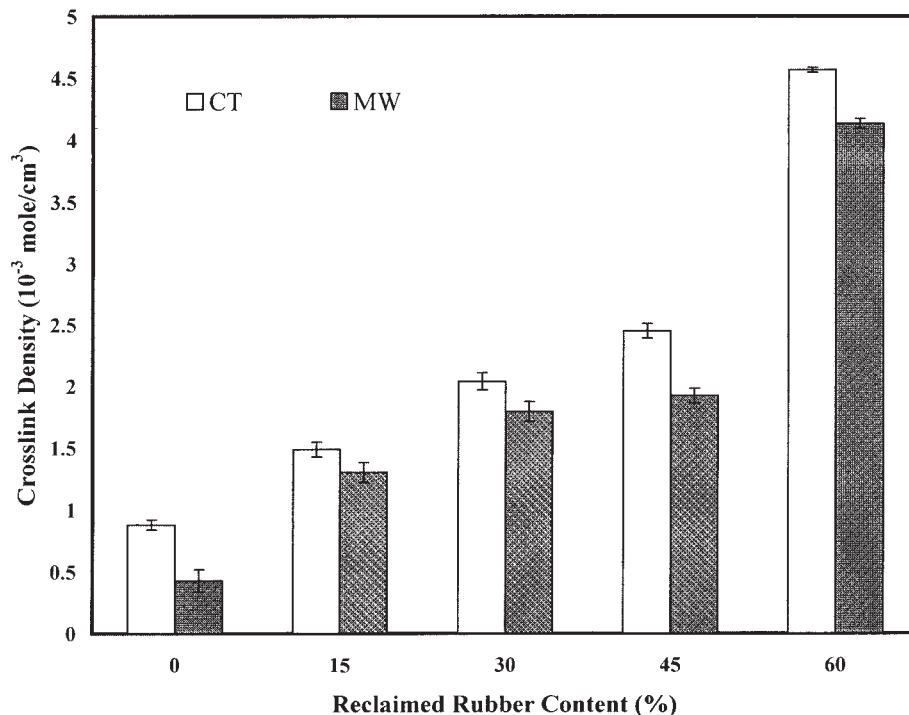


Figure 2 Effects of RR content on crosslink density of the vulcanizates cured by the CT and MW methods.

thickness. The samples were weighed before soaking in toluene solvent. The samples were then immersed in toluene. The swollen samples were taken out periodically and any excess liquid on the specimen surface was removed by light contact pressure with filter paper. The swollen weight was immediately measured with utmost care. After an equilibrium period, the samples were taken out of the toluene and weighed until their weights remained unchanged. The mole % uptake of solvent at time t (Q_t) was calculated from eq. (2).^{3,11}

$$Q_t = \frac{(W_t - W_0)W_w}{M_w} 100 \quad (2)$$

where W_0 and W_t are the weights of dry and swollen samples, respectively and M_w is the molar mass of toluene (92.14 g mol⁻¹).

Mechanical properties

Various mechanical property evaluations were performed. The tensile properties (modulus at 100% elongation, ultimate tensile stress, and % elongation at break) of the vulcanizates were tested according to ASTM D 412-92 (1998); the tests being carried out with a universal testing machine (Autograph AG-I, Shimadzu, Tokyo, Japan). Tear strength was determined as specified by ASTM D 624-00(2000) using angle-shaped samples and a Shimadzu tear-strength testing

machine. The testing speed for both the tensile and tear properties was 500 mm/min. Vulcanizate hardness was carried out using a Shore A Instrument and M.F.G., model PN71500 by ASTM D 2240-97 (1997).

RESULTS AND DISCUSSION

Physical properties

Figure 2 shows the effect of RR content on the crosslink density of NR/CB vulcanizates cured by conventional thermal (CT) and microwave (MW) methods. It can be observed that for both curing systems, the crosslink density greatly increased with the RR content, especially at 60 wt % RR content. This was because the loading of RR into the NR/CB compounds automatically increased the CB contents in the rubbers.² It has been evidenced¹² that CB can perform as a physical crosslinker in the rubber matrix, as a result of the rubber molecules being absorbed into the black surface during compounding. The details of the NR-CB molecular interaction can be found elsewhere.¹²⁻¹⁴ Considering the effect of the curing method, it was found that the MW method gave a slightly lower crosslink density than the CT method for any given RR content. The effect of RR content on the change in crosslink density in the MW method was similar to that in the CT method, indicating that the crosslinking reactions as a result of RR loading, which had occurred in both curing methods, were the same.

Since the cure times of the MW and CT methods were the same, any differences in the properties of the vulcanizates would have resulted from the mechanism of the heat generation and distribution throughout the vulcanizates during curing. Although the crosslinking reactions of the rubbers from these two curing methods were thought to be the same as stated in the explanation given for Figure 1, their mechanisms of heat generation and its transfer to the rubber were probably different. In the CT method, the heat was generated by an external heating system and was transferred as a result of the thermal conduction from the mold to the rubber compound where the crosslinking process took place. It was predicted that during compression molding, temperatures across the thickness of the vulcanized sheet were not uniform, and this would lead to differences in cure times across the sheet thickness, thus resulting in nonuniform vulcanizate properties across the sample thickness. For the MW method, the temperatures across the sheet thickness were thought to be the same, since the heat was generated simultaneously throughout the rubber sheet during curing, and therefore, leading to a more uniform crosslinking and thus uniform vulcanizate properties across the sample thickness. The mechanism of rubber curing by microwave involves changes in polarization of the materials caused by microwave absorption. When the microwave passes into the rubber compounds (now regarded as an absorber material), an electric field occurs inside the compounds as a result of microwave absorption.¹⁰ This electric field activates the changes in motions (rotations and vibrations) and polarizations of the rubber molecules, which then causes molecular frictions and heat in the rubber compound and initiates the curing.^{10,15}

Figure 3 illustrates the sorption-desorption behavior of the NR/CB vulcanizates in toluene as a function of RR content for the CT (dashed lines) and MW (solid lines) curing methods. It was found that the higher the RR content, the lower the toluene uptake. This was expected because the decrease in toluene uptake was caused by the increase in crosslink density as a result of the CB in the RR. The increased crosslinks restricted the mobility of rubber molecules, which prevented the penetration of the solvent molecules into the rubber.^{3,16} When comparing the sorption behavior of the vulcanizates from the two different curing systems, it was found that for any given RR content, the rate and the mole% uptake (up to 600 min) and mol % release (after 600 min) of toluene in vulcanizates were greater for the CT method. It is interesting to note that although the crosslink densities of the vulcanizates in the CT method were slightly higher, the swelling resistance to solvent penetration was less than the vulcanizates in the MW method. Previous works^{17,18} have suggested a direct relationship between the swelling level in a solvent and the type of crosslinks in an NR

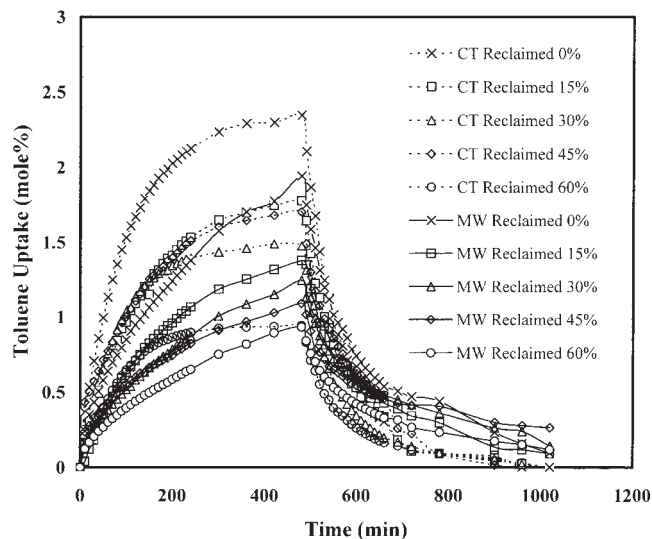


Figure 3 Toluene uptake in the vulcanizates with various RR contents by CT and MW methods.

vulcanizate. That is, a vulcanizate with a high proportion of mono- and disulfidic crosslinks usually has a higher resistance to swelling than one with polysulfidic crosslinks. Therefore, the reason for the lower swelling of the vulcanizates in the CT method is probably related to the type of crosslinks formed in the vulcanizates. In this preliminary study, since the heating mechanisms between the CT and MW methods were different, the types of crosslinks formed in the rubber compounds would probably be different. The CT method gave a majority of polysulfidic crosslinks in the vulcanizates, while the MW method gave a majority of mono- and dipolysulfidic crosslinks. This postulation was drawn based on the likelihood that the MW curing was more instantaneous and rapid than the CT curing for any given crosslink density, and this is in analogy with the crosslinking process having a greater ratio of accelerator-sulfur in the rubber compound. Previous works^{17,18} have shown that faster curing in a rubber vulcanization system tended to produce more mono- and disulfidic crosslinks than polysulfidic ones. If this postulation is true, one would be able to explain all the differences in toluene uptake/release, as already mentioned in Figure 3. That is, the monosulfidic and disulfidic linkages had less free volume and flexibility of the crosslinked rubber chain as compared to the polysulfidic linkages. Therefore, the ability of toluene to penetrate the vulcanizates cured by the MW method, which contained more mono- and disulfidic crosslinks, would be less.

Another indirect experiment was performed to substantiate the reason for crosslink-type differences in the vulcanizates obtained by the CT and MW methods. This was carried out by calculating a Mooney-Rivlin constant (C_2) from a stress-strain measure-

ment,¹² which is referred to as the type of crosslink in a rubber vulcanizate. Works by Sombatsompop^{16–18} established a relationship between the C_2 constant and the type of crosslinks in the sulfur-cured NR vulcanizates. It was suggested that the C_2 constant appeared to be associated with intermolecular forces between crosslinks and rubber molecules.¹⁶ That is, for any given crosslink density, vulcanizates containing more mono- and disulfidic crosslinks tended to give higher C_2 values than those containing more polysulfidic crosslinks. In relation to this work, one would expect to obtain higher C_2 values in the vulcanizates cured by the MW method, as postulated earlier. This was found to be the case according to the results in Figure 4 and Table I, illustrating the Mooney-Rilvin stress-strain curves and the calculated C_2 values, respectively, of the vulcanizates cured by the CT and MW methods. The results in Figure 4 and Table I were obtained from the stress-strain relationship shown in eqs. (3) and (4).^{12,16} The C_2 values were obtained from the slopes of the plot of $\sigma/(\lambda - \lambda^{-2})$ and $1/\lambda$.¹¹ To obtain a straight line for assessing the C_2 value (slope), a tensile elongation range of 5–25% ($1/\lambda \approx 0.8–0.95$) was used.¹² It should be noted that details of the experimental procedures and conditions for the production of Mooney-Rilvin stress-strain curves and the C_2 values can be obtained from previous work.¹⁶

$$F = 2A(\lambda - \lambda^{-2})(C_1 + C_2\lambda^{-1}) \quad (3)$$

$$\sigma/(\lambda - \lambda^{-2}) = 2C_1 + 2C_2/\lambda \quad (4)$$

where F is the force required for stretching the specimen, A is the cross section area of the unstretched rubber specimen, λ is the extension ratio (which is $1 +$

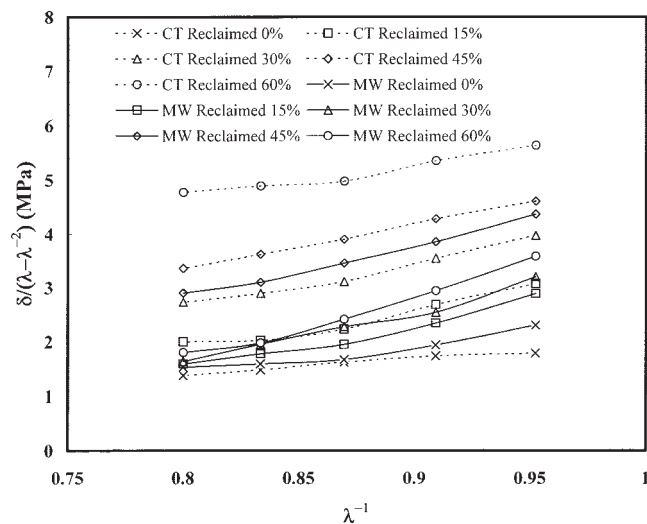


Figure 4 Plot of $\sigma/(\lambda - \lambda^{-2})$ and $1/\lambda$ for the vulcanizates with different RR contents from the MW and CT methods.

TABLE I
Mooney-Rivlin Constants (C_2 Values) for the
Vulcanizates with Different RR Contents from
the MW and CT Methods

Curing methods	RR contents (%)	Mooney-Rivlin constant, C_2 value (MPa)
CT	0	1.40
	15	3.74
	30	4.15
	45	4.15
	60	4.09
MW	0	2.54
	15	4.20
	30	4.91
	45	4.87
	60	6.01

ε), ε is the strain, and values of C_1 and C_2 are Mooney-Rilvin constants.

Mechanical properties

Figures 5, 6, and 7 show the effects of RR content on tensile modulus at 100% elongation, ultimate tensile stress (UTS), and elongation at break of the NR/CB compounds cured by the CT and MV methods, respectively. It can be seen that the tensile modulus progressively increased with RR content. For a given RR content, the tensile modulus of the vulcanizates for the MW curing method was lower. These could be explained in terms of the differences in the densities and types of crosslinks, as discussed earlier. It was interesting to note that the differences in the tensile properties became smaller at higher RR contents. This may be associated with the amount of CB in the rubber compounds (carbon content varying with RR content), in order for the rubber blends to be induced by microwave irradiation.¹⁰ The values of UTS and elongation at break were also found to decrease with RR content, and this involves the heterogeneity and defects present in the NR phase caused by the addition of RR (the detailed explanation for this can be found in a previous work²). However, it is of interest to mention that the magnitude of the reductions of UTS and elongation at break with changing RR content (i.e., property sensitivity to RR content) was greater with the CT method. This was probably associated with the uniformity of the vulcanizate properties across the sample thickness between these two curing methods, as discussed earlier: less uniform vulcanizate properties across the sample thickness occurring in the CT method.

Figure 8 shows a plot for the tear strength of NR/CB containing different RR contents. It was found that the tear strength of microwave-cured vulcanizate was

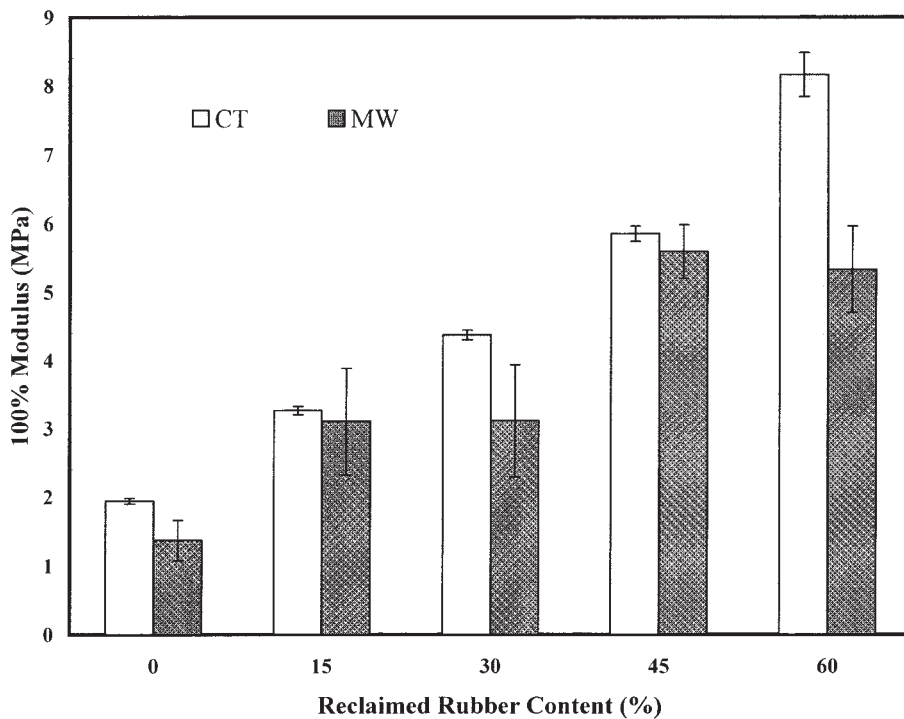


Figure 5 Tensile modulus at 100% elongation of the vulcanizates for different RR contents by the CT and MW methods.

lower than that of the conventional thermal-cured vulcanizate because of the differences in densities and types of crosslinks formed in the vulcanizates. Again, the magnitude of the reduction of the tear strength in

the vulcanizates with increasing RR content was greater when using the CT method.

The changes in the hardness results for the vulcanizates cured by the CT and MW methods are shown in

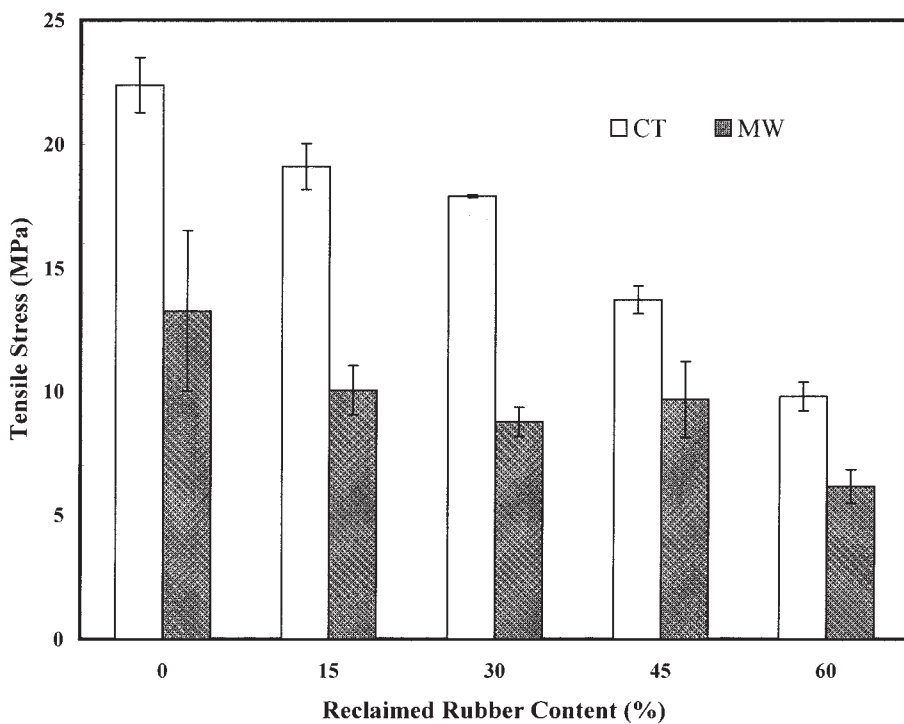


Figure 6 Ultimate tensile stress of the vulcanizates for various RR contents by the CT and MW methods.

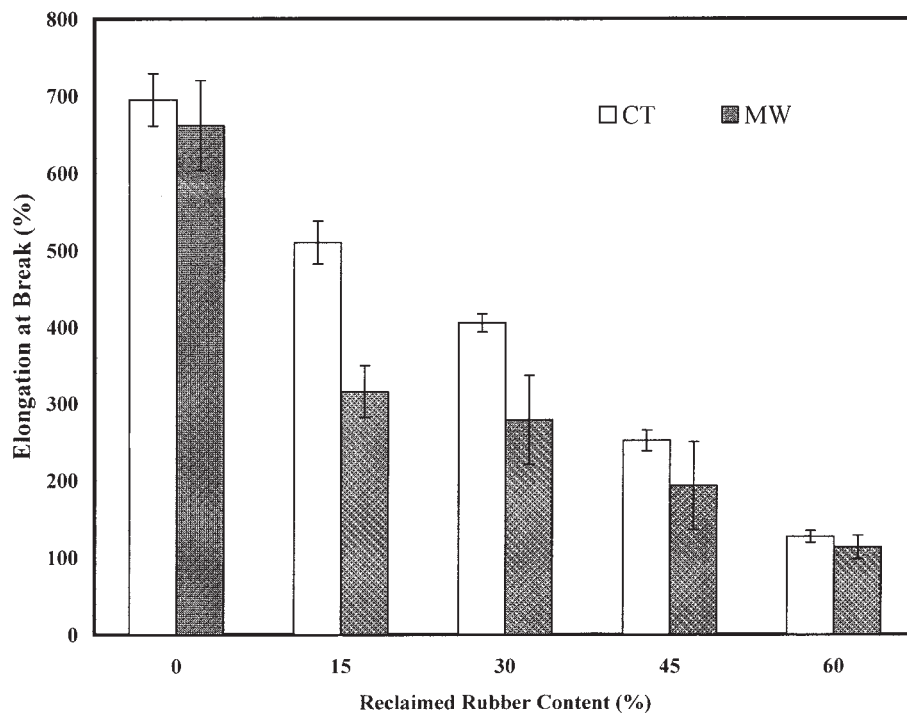


Figure 7 Elongation at break of the vulcanizates for various RR contents by the CT and MW methods.

Figure 9. It was found that the changing trend was similar to the tensile modulus results.

Overall, under the experimental conditions used in this work, it could be concluded that the mechanical

properties of the NR/CB/RR vulcanizates cured by the CT method were higher than those by the MW method, except for the resistance to toluene penetration. The discrepancies in the mechanical properties of

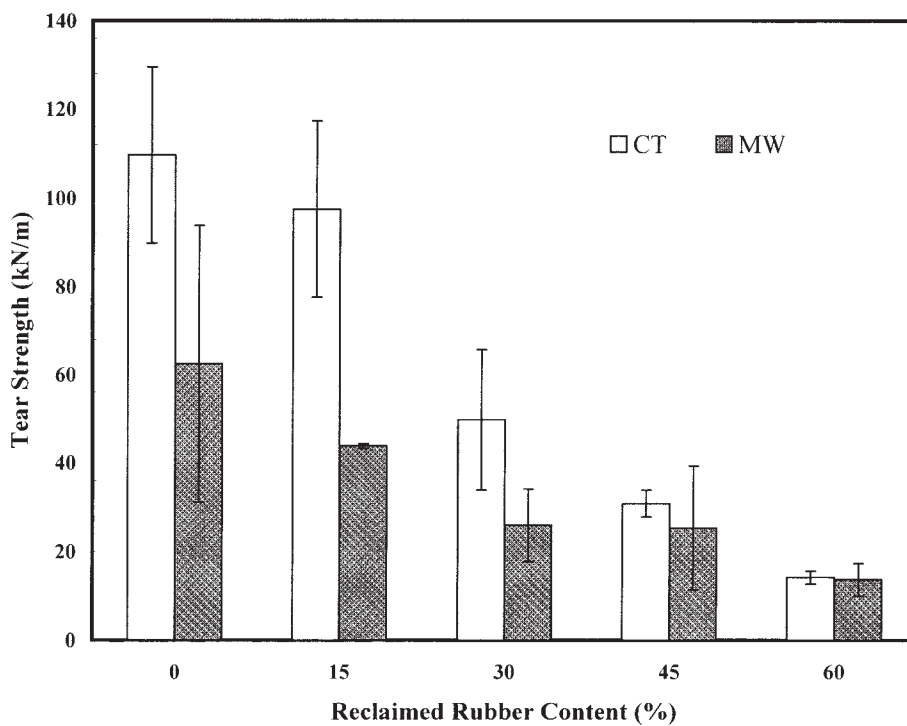


Figure 8 Tear strength of the vulcanizates for different RR contents by the CT and MW methods.

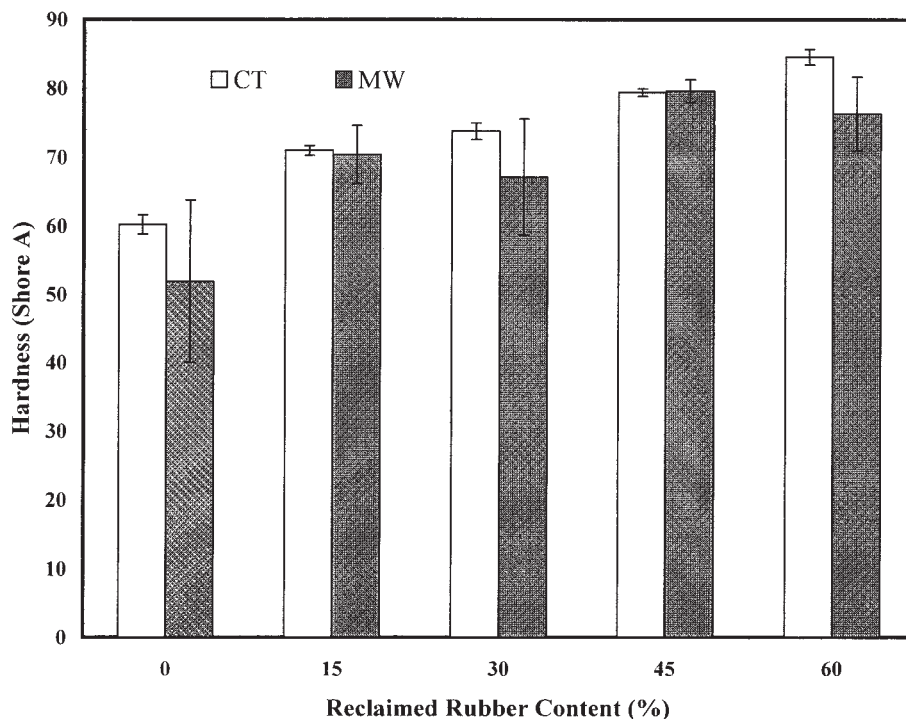


Figure 9 Hardness of the vulcanizates for various RR contents by the CT and MW methods.

the vulcanizates by the two different methods were caused by differences in densities and types of crosslinks formed in the vulcanizates, although the property differences were minimized as the RR content was increased. From the experimental results, the CT-cured vulcanizates contained relatively high proportion of polysulfidic crosslinks, whereas the MW-cured vulcanizates produced more monosulfidic and disulfidic crosslinks. It is widely accepted¹² that the vulcanizates with high proportion of polysulfidic crosslinks tend to show greater mechanical properties. This conclusion suggested that to improve the mechanical properties of the vulcanizates cured by the MW method, high polysulfidic crosslinks in the vulcanizates are required. Previous work by Sombatsompop¹⁸ indicated that different crosslink types in the rubber vulcanizates could be altered by varying the sulfur-to-accelerator ratio during the compounding stage. As a result, further work, beyond the scope of this work, would be to quantitatively verify the crosslink types in the vulcanizates cured by CT and MW methods, and to improve the mechanical properties of the rubber vulcanizates cured by MW method by altering the sulfur-to-accelerator ratio in the rubber compounds.

CONCLUSIONS

The effect of RR content on the crosslink density, swelling behavior, and mechanical properties of NR

added with 60 phr CB was examined through MW and CT curing methods. The experimental results showed that, for both curing methods, the tensile modulus and hardness of the vulcanizates increased with RR content. The tensile stress, elongation at break, and tear strength were found to reduce with RR content. The mechanical properties of the vulcanizates from the CT method were higher than those from the MW method. An opposite effect was observed for the resistance to swell in toluene. The sensitivity in property changes of the vulcanizates due to addition of the RR content were greater for the CT method than the MW method, and this was related to the uniformity of the properties across the sample thickness. The differences in the properties of the vulcanizates from the CT and MW methods became smaller at higher RR contents. The discrepancies in the vulcanizate properties obtained by the CT and MW systems were explained in terms of the density and types of crosslinks present in the vulcanizates.

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