Highly Purified Natural Rubber by Saponificaion of Latex: Analysis of Green and Cured Properties

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ABSTRACT: Natural rubber (NR) was purified by saponification of fresh latex from *Hevea brasiliensis* and soaking process of the coagulum with aqueous NaOH solution. This treatment resulted in the decrease of nitrogen content to the same level as enzymatic deproteinized NR. The saponification of NR latex and soaking of the coagulum gave the rubber having good processability with easy to cure. The saponified rubber showed outstanding physical and dynamic mechanical properties such as high tensile

properties, high storage modulus, low tan δ , low heat build-up and low dynamic compression set. The saponified rubber blend with styrene butadiene rubber (SBR) according to the recipe of rubber tire showed also a good performance. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 3524–3531, 2010

Key words: purification; natural rubber; saponification; physical properties; dynamic mechanical properties

INTRODUCTION

It is widely recognized that natural rubber (NR) from Hevea brasiliensis, shows several superior properties than synthetic rubbers such as high green strength^{1,2} and tack¹ in the un-vulcanized state and high tensile strength,³ high crack growth resistance^{4,5} and minimal heat build up in the vulcanized state. These properties were postulated to be due to the nonrubber components amounting 6% (w/w) and structural characteristic of NR, different from synthetic cis-1,4 polyisoprene (IR). As reported previously,⁶ a linear rubber chain in NR is proposed to consist of the initiating terminal, i.e., ω -terminal, two *trans*-1,4 isoprene units, about 1000 - 3000 cis-1,4 isoprene units, and the chain end group, i.e., *a*-terminal. The *a*-terminal is composed of mono- or di-phosphate group linked with phospholipids,^{7,8} which plays a role in branching formation of rubber molecules.⁹ On the other hand, the ω -terminal of rubber molecule in NR was postulated to be a modified dimethylallyl group with a functional group,¹⁰ which can link with proteins¹¹ and phospholipids to form the branch-point through intermolecular hydrogen bond. The proteins in NR are believed to be an important component to provide outstanding properties for NR, although there is no clear evidence supporting this hypothesis. On the other hand, some proteins existing in NR cause the acute allergic reaction termed as Type I allergy to some peoples coming in contact with NR products such as gloves, condoms, and rubber dam for dental supply.¹² The proteins in NR latex can be removed by enzymatic deproteinization.¹³ Repeated centrifugation in the presence of a surfactant is reported to be a effective method to reduce the nitrogen content, which is widely used as an index of protein content in NR, to 0.02% (w/w) or lower level.¹¹ However, it has been reported that gloves of DPNR latex also positive for Type I allergic reaction, although these are much safer than ordinary NR gloves.¹⁴ In the preceding article we reported that saponification of NR latex in the presence of a surfactant can provide purified NR latex free from allergic proteins by selecting the concentration of NaOH, reaction time and reaction temperature.¹⁵ This finding suggest the preparation of allergy free solid NR by saponification of NR latex.

The procedure involving the centrifugation process to removed decomposed proteins is not practical for the commercial production of solid rubber due to the low productivity and high cost of centrifugation on a large scale. Furthermore, the use of surfactant for stabilizing the latex during reaction usually brings about the difficulty for the coagulation of latex for the preparation of solid rubber. To produce

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Composition	Part per hundred rubber (phr)
Rubber	100
Sulfur	2
Stearic acid	3
ZnO	5
MBT ^a	1
Antioxidant (6PPD ^b)	2

^a 2-Mercaptobenzothiazole.

^b *N*-(1,3-dimethylbutyl)-*N*-phenyl-*p*-phenylenediamine.

highly purified solid NR, we found these problems can be solved by the selection of the saponification conditions and application of a new coagulation process to coagulate the reacted latex instantaneously, which makes it possible to wash effectively the resulting coagulum. The most striking aspect of the resulting saponified NR is that the green and cured rubber properties are clearly different from DPNR as well as ordinary commercially available NR, showing characteristics such as no storage hardening phenomenon, good processability, low dynamic heat buildup, and high abrasion resistance. The present work is the preliminary results of the physical and mechanical properties of the saponified rubber in un-vulcanized and vulcanized states compared with the ordinary NR as well as DPNR. The effect of proteins and lipids in the rubber samples on physical and mechanical properties was also investigated. Saponified NR was also blended with styrene butadiene rubber (SBR), according to the recipe of tire for carcass and analyzed their properties.

EXPERIMENTAL

Sample preparation

Fresh natural rubber latex (FL) from *H. Brasiliensis* was used in this work. Fresh NR (FNR) was prepared by coagulation of FL latex using the combination of a commercial cationic flocculant, Floerger (provided by Kao, Thailand), and 5% (w/v) formic acid. The resulting fine coagulum formed instantaneously was subjected to wash twice with water and dried in an oven at 50°C for 24 h.

Deproteinized NR (DPNR) was prepared by incubation of FL latex (30% w/v dry rubber content) with 0.04% (w/v) proteolytic enzyme (KAO KP-3939) and 0.5% (w/v) sodium dodecyl sulfate (SDS) for 12 h at 37°C. The resulting latex was coagulated and dried by the same condition as mentioned earlier.

Saponified NR (SAP-NR) was prepared by the treatment of FL latex with 1.5% (w/v) NaOH in the presence of 0.2% (w/v) Triton®X-100 at 70°C for 3 h. The resulting latex was coagulated and dried by the same condition as mentioned earlier.

Saponified and soaked NR (SAP/SK-NR) was prepared by soaking the wet coagulum of SAP-NR in 2% (w/v) aqueous NaOH solution at room temperature for 24 h. The resulting rubber was washed with water and dried under the same condition as mentioned above.

Commercial product of constant viscosity NR (CV60) and SBR grade 1502 was provided by Chalong Latex Industry and Thaibamrung Chemical Industry, respectively.

Characterization

The nitrogen content of the sample was analyzed by a Leco Nitrogen Analyzer (model FP 528) with the sensitivity of 0.001%. The quantity of long-chain fatty acid ester group was determined by FTIR measurement based upon calibration by a mixture of methyl stearate and synthetic *cis*-1,4-polyisoprene, Kuraprene IR10. The content of fatty acid ester group per weight of rubber was determined by the intensity ratio of peaks at 1739 cm⁻¹ (C=O) to 1664 cm⁻¹ (C=C).

The gel content in rubber sample was determined by dissolving accurately weighed rubber in 0.1%(w/v) dried toluene and kept it in the dark without stirring for a week at room temperature. The insoluble fraction was separated from sol fraction by centrifugation at 10,000 rpm (14,900 g) for 30 min. The gel fraction was coagulated with methanol and dried under vacuum at 40°C. The gel content was determined as the weight percentage of the gel fraction against the total sample weight.

Mixing process and vulcanization

The compounding formulation of vulcanized rubbers and vulcanized rubber blends are shown in Tables I and II, respectively. The mixing process was carried out in an internal mixer at 40°C using the rotor

TABLE II Formulation of the Rubber Blend, According to the Recipe of Rubber Tire for Carcass

Composition	Part per hundred rubber (phr)
*	
NR	50
SBR	70
CB N660	43
Aromatic oil (Sundex 790)	8
ZnO	4
Stearic acid	1.5
TMQ ^a	1.5
MBT ^b	0.5
TMTD ^c	1
Sulfur	2.5

^a Polymerized 1,2-dihydro-2,2,4-tri-methylquinoline.

^b 2-Mercaptobenzothiazole.

^c Tetramethyl thiuram disulfide.

TABLE III

speed of 40 rpm and the fill factor 0.7. The obtained rubber compounds were passed through a two-roll mill and kept in the dark before being proceeded further for vulcanization.

The rubber compounds were vulcanized at 155°C by compression molding. Cure time of each sample was varied according to the optimum cure time of each sample measured by oscillating disk rheometer (ODR).

Physical and dynamic mechanical properties

Tensile measurement was carried out by an Instron Model 4301 with the testing crosshead speed of 500 mm/min at room temperature. The applied load cell of 100 N and 1 kN were used for un-vulcanized rubber samples and vulcanized rubber samples, respectively.

Measurement of Mooney viscosity (ML1+4) was carried out by using a TECHPRO Mooney Viscometer. The temperature of testing was $100^{\circ} \pm 1^{\circ}$ C and the large rotor size was used to measure viscosity at the strain rate of about 2 sec⁻¹. The rubber sample was preheated at 100° C for 1 min with continuous shear for 4 min followed by measuring the decay of torque when the rotor was stopped.

A Goodrich Flexometer was used to determine the storage modulus, loss modulus and tan δ of the vulcanized samples. The sample was employed the oscillating force at a constant frequency of 10 Hz. The initial and dynamic compressive strains used were 10% and 2%, respectively.

The dynamic heat buildup and dynamic compression set were also determined by Goodish Flexometer. The sample was employed the oscillating force at a constant frequency of 30 Hz and the flexing stoke of 4.45 mm for 25 minutes.

Abrasion resistance of sample was determined by DIN Abrasion Tester. The test serves to determine the volume loss of sample as a result of abrasion. The average value was taken as the abrasion loss of each sample tested at least three samples.

RESULTS AND DISCUSSION

The purification of NR latex by deproteinization and saponification resulted in the decrease of nitrogen content as shown in Table III. The nitrogen content of NR decreased from 0.36% (w/w) to about 0.11% (w/w) after deproteinization and saponification. It is clear that the nitrogen content of SAP-NR is similar to that of DPNR, indicating the similar efficiency of deproteinization and saponification to remove protein in NR latex. The nitrogen content of DPNR in the previous report was usually less than 0.02% (w/w).^{6,16} The higher nitrogen content of DPNR in

Characterization Properties of Rubber Samples						
Sample	Nitrogen content (%w/w)	Ester content (mmol/ kg-rubber)	Gel content (%w/w)	Ash content (%w/w)		
FNR DPNR SAP-NR SAP/SK-NR	0.364 0.109 0.110 0.094	28.1 28.2 24.7 20.7	9.4 1.8 1.8 3.8	0.22 0.20 0.21 0.21		

this report can be explained by the different preparation method, i.e., direct coagulation of the treated latex without centrifugation. It is known that the decomposed proteins can be removed effectively by centrifugation of DPNR latex in the presence of surfactant. The acid coagulation cannot remove all of the decomposed proteins as it can be confirmed by the residual nitrogen content. Consequently, the nitrogen content in DPNR in this work corresponds to the residual decomposed nitrogenous compounds, mostly decomposed proteins, which were not completely extracted in the course of coagulation and washing with water. A similar behavior is presumed for the high nitrogen content of SAP-NR too. It is noted that the saponified rubber after centrifugation showed the similar nitrogen content of about 0.02% (w/w).¹⁵ The soaking process of SAP-NR coagulum into NaOH aqueous solution can further decrease the nitrogen content. This is attributed to the removal of decomposed proteins, which is adsorbed on the surface of the coagulum.

The fatty acid ester content and gel content of the rubber samples are also shown in Table III. The fatty acid ester content of NR showed no change after deproteinization, while it decreased after saponification and further decreased after soaking with NaOH. The fatty acid ester content of SAP-NR was 24.7 mmol/kg-rubber, whereas that of SAP/SK-NR was 20.7 mmol/kg-rubber. It is clear that the saponification can decompose some fatty acid ester groups in lipids and further removed after soaking with NaOH. The treatment of NR latex with strong alkali (i.e., NaOH) can decompose the proteins and some solubilized lipids covering the rubber particle and the soaking process of rubber coagulum can further remove the decomposed proteins and some solubilized lipids. In this work coagulation was carried out by the combination of a cationic high-polymer flocculant and formic acid to obtain instantaneously the coagulum composed of fine crumbs, which has facilitated the subsequent soaking and washing process. The removal of proteins by deproteinization and saponification resulted in the decomposition of gel fraction in NR, as is evident by the decrease of

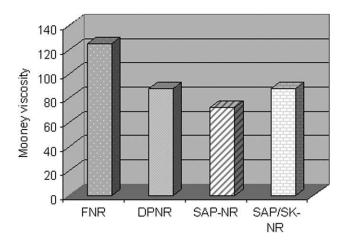


Figure 1 Mooney viscosity of rubber sample in un-vulcanized state.

gel content. The gel content of SAP-NR is almost the same as that of DPNR. Figure 1 shows Mooney viscosity of the rubber samples. It is clear that the Mooney viscosity value of SAP-NR was lower than that of DPNR and apparently lower than that of FNR. This is in accordance with the reduction of gel content after deproteinization and saponification. It is remarkable that the soaking of SAP-NR with NaOH gave slightly higher gel content and Mooney viscosity compared with SAP-NR. However, they are significantly lower than those of FNR. These findings suggest that the processability of NR can be improved by saponification.

The stress–strain curves of these rubber samples were shown in Figure 2. The green strength of FNR was 5.51 MPa, while that of DPNR, SAP-NR and SAP/SK-NR were 3.22, 1.97, and 1.86 MPa, respectively. It is clear that the green strength of SAP-NR is very low. This is one of the characteristics of the saponified rubber. This behavior may be attributed to the disintegration of branch-points via the decom-

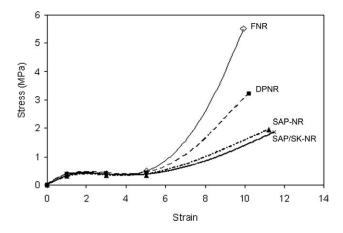


Figure 2 Stress–strain curves of raw or un-vulcanized rubbers.

position of proteins and fatty acid ester groups in lipids by saponification. The strain at break of rubber slightly increased after deproteinization and saponification. In the previous work,¹⁷ DPNR was reported to show a similar green strength as NR. However, DPNR, presented in this work, showed the lower green strength than NR. The inconsistent properties of DPNR may be due to the variation of the starting NR, i.e., freshly tapped latex or preserved fresh latex, which affect the number of branch-points. Nevertheless, it is interesting to note that DPNR always showed the lower modulus and higher strain at break than NR.

Cured rubber properties of the rubber samples vulcanized with sulfur at 155°C were shown in Table IV. The scorch time of SAP-NR and SAP/SK-NR was about 1.4 min, similar to that of FNR, while DPNR showed the highest scorch time of about 4.15 min. The cure time of FNR was 6.19 min, while SAP-NR and SAP/SK-NR showed shorter cure time. DPNR showed the highest cure time of about 11.23. It can be observed that DPNR showed the longest cure time and scorch time. It can be explained by the reduction of proteins and nitrogenous constituents in NR after deproteinization since they were reported to be the activators for the MBT-accelerated vulcanization.¹⁸ The key function of the nitrogenous compound is to solubilize the zinc salt of MBT, which is regarded as the actual accelerator in MBTaccelerated system.¹⁹ Although SAP-NR has a similar nitrogen content as DPNR, SAP-NR showed the shorter scorch time and cure time than DPNR. The difference between SAP-NR and DPNR is the partly decomposition of lipids by saponification. The fast cure of saponified rubber, i.e., SAP-NR and SAP/ SK-NR, may be the effect of by-product of saponification, which may be the compound having an ability to accelerate the vulcanization. The decomposition of lipids by saponification is considered to be a factor affecting the vulcanization. Glycerides, major component of lipids in NR, can be decomposed by NaOH to form long-chain fatty acids. One of them is stearic acid, which is known to be an activator for

TABLE IV Cured Properties of Rubber Samples Vulcanized with Sulfur at 155°C

Rubber	T _{Max}	T _{Min}	ΔT	t _S	t ₉₅
sample	(N m)	(N m)	(N.m)	(min)	(min)
CV60	16.04	5.64	10.40	2.55	7.38
FNR	15.69	4.85	10.84	1.43	6.19
DPNR	13.89	3.79	10.10	4.15	11.23
SAP-NR	15.63	5.57	10.06	1.42	5.52
SAP/SK-NR	15.42	5.14	10.00	1.42	4.36

 $T_{\text{max}} = \text{maximum torque}, T_{\text{Min}} = \text{minimum torque}, \Delta T$ = delta torque, t_S = scorch time, t_{95} = cure time 25

20

10

5

0

0

Stress (MPa) 15

Figure 3 Stress-strain curves of vulcanized rubbers.

Strain

6

SAP-NR

CV-60

8

SAP/SK-NR

10

the vulcanization. Even through the ester content of rubber decrease after saponification, the amount of fatty acids (i.e. stearic acid) may be higher.

The delta torque (ΔT) is the difference between the maximum and minimum torques. Because the torque in the rheological measurement starts to increase by the formation of crosslinks, the delta torque is very closely related to the crosslink density.^{20,21} The high value of delta torque means the high crosslink density. As shown in Table IV, the ΔT values of all rubber samples were almost the same of about 10 N.m, indicating the similar crosslink density can be presumed for all rubber samples.

The stress-strain curve of vulcanized rubbers was shown in Figure 3. The tensile strength of vulcanized SAP-NR and SAP/SK-NR was higher than that of vulcanized FNR and vulcanized CV60. The tensile strength of vulcanized DPNR was almost similar to that of vulcanized FNR, while the strain at break of vulcanized DPNR was higher. It is clear that the vulcanized saponified rubber showed the highest tensile strength among the samples.

The dynamic mechanical properties can be expressed in the term of storage modulus (E'), loss modulus (E'') and tan δ . The storage modulus represents the immediate response to the applied force. The loss modulus represents energy dissipated as heat. The ratio of E'' to E' is tan δ , which is generally known as hysteresis factor or loss tangent. The hysteresis factor in a material is a measure of energy

TABLE V Dynamic Mechanical Properties of Vulcanized Rubbers

		-			
Properties	CV60	FNR	DPNR	SAP-NR	SAP/ SK-NR
Storage modulus, E' (MPa)	1.26	1.51	1.16	1.70	1.69
Loss modulus, E'' (MPa)	0.04	0.04	0.07	0.03	0.03
Tan δ	0.04	0.03	0.06	0.02	0.02

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dissipated by various processes such as molecular mobility,^{22,23} breakdown and reformation of the filler particles²⁴ or slippage of rubber molecules under high strain amplitude.25 The dynamic mechanical properties of vulcanized CV60, FNR, DPNR, SAP-NR, and SAP/SK-NR are shown in Table V. The storage modulus of SAP-NR and SAP/SK-NR showed the highest value among the samples. The storage modulus of FNR was higher than that of CV-60. This may be due to the lower Mooney viscosity of CV-60 by the effect of treatment with hydroxylamine. The storage modulus of DPNR was the lowest among the samples. The loss modulus and tan δ of vulcanized SAP-NR and SAP/SK-NR showed the lowest value among the samples, whereas those of DPNR showed the highest values. The results suggest that the elasticity of rubber samples is align in the order of SAP-NR \sim SAP/SK-NR > FNR > CV60 > DPNR.

The heat build-up is the temperature rising from a chamber's temperature during dynamic measurement. The heat build-up of rubbers is shown in Figure 4. The heat build-up of DPNR was very high, compared with other samples. It is observed that the heat build-up of SAP-NR and SAP/SK-NR are the lowest among the rubber samples. Dynamic compression set is one of factors referring to the elasticity of rubber sample. Basically, the high elastic rubber can retain the original shape after applying the force. Consequently, it will show the low compression set. In other words, the rubber having low elasticity will give the high compression set. It is clear that SAP-NR and SAP/SK-NR showed the lowest dynamic compression set among the rubber samples as shown in Figure 5. The dynamic compression set of DPNR was very high, compared with other samples.

All the results presented above clearly showed that the purification of NR by saponification and soaking process give the rubber having good

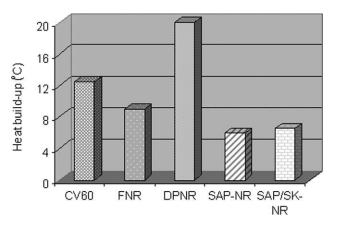


Figure 4 Heat build-up of vulcanized rubbers.

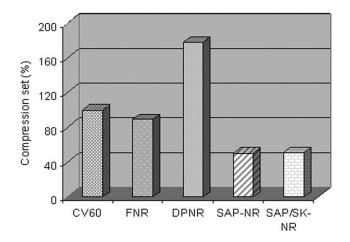


Figure 5 Dynamic compression set of vulcanized rubbers.

performance such as low Mooney viscosity, short cure time, high tensile strength, good dynamic mechanical properties and low heat build-up. By considering that the purification of NR by deproteinization give the rubber, which need long time to cure and showed the decrease in physical and dynamic mechanical properties, compared with the ordinary NR. Consequently, saponification of NR latex and soaking process of NR coagulum is a beneficial method for purification of NR.

Since SAP-NR and SAP/SK-NR showed better dynamic mechanical properties and lower heat build-up than that of the ordinary NR, these rubbers are expected to be a good new material for making rubber tire. To study this possibility, SAP-NR and SAP/SK-NR were blend with SBR according to the recipe of tire for carcass and investigated the physical and dynamic mechanical properties of the rubber blends, compared with the ordinary NR blend. Table VI shows the cure properties of rubber blends. The scorch time and cure time of SAP-NR blend and SAP/SK-NR blend were slightly lower than that of FNR blend and CV60 blend. The delta torque, which represents the crosslink density of rubber, showed

TABLE VICure Properties of Rubber Blends Vulcanized with
sulphur at 155°C

	····F·····				
Rubber sample	T _{Max} (N m)	T _{Min} (N m)	ΔT (N m)	t _S (min)	t ₉₅ (min)
CV60 blend FNR blend SAP-NR blend SAP/SK-NR blend	35.01 38.98 38.70 38.60	5.89 6.26 6.70 6.57	29.12 32.72 32.00 32.03	1.54 1.50 1.45 1.40	3.43 3.42 3.30 3.13

 $T_{\text{max}} = \text{maximum torque}, T_{\text{Min}} = \text{minimum torque}, \Delta T$ = delta torque, t_S = scorch time, t_{95} = cure time.

14 FNR 12 SAP/SK-NF 10 Stress (MPa) 8 CV-60 6 SAP-NE 4 2 Π 2 3 Strain

Figure 6 Stress-strain curves of rubber blends.

almost similar value between FNR blend, SAP-NR blend and SAP/SK-NR blend. This indicates that the crosslink density of these rubbers was almost the same. The delta torque of CV-60 blend showed slightly lower than other samples. Therefore, the crosslink density of CV-60 blend may be slightly lower than other samples.

The stress–strain curve of CV-60, FNR, SAP-NR and SAP/SK-NR blends is shown in Figure 6. The tensile strength of FNR blend showed similar value to that of SAP-NR and SAP/SK-NR blends. The tensile strength of CV-60 blend showed the lower value than others. This can be explained by its lower crosslink density. It is noticeable that the ratio of SBR in the recipe of rubber blend is higher than that of NR (SBR 70 phr per NR 50 phr). Thus, the tensile properties of rubber blend may be mainly affected by properties of SBR.

The dynamic mechanical properties and abrasion resistance of rubber blends are shown in Table VII. The storage modulus of CV-60 blend and FNR blend was higher than that of SAP-NR blend and SAP/SK-NR blend. The loss modulus, tan δ and abrasion loss of all rubber blends were almost the same. The heat build-up and compression set of rubber blends are shown in Figures 7 and 8, respectively. The heat build-up of SAP-NR blend and SAP/SK-NR blend

TABLE VII
Dynamic Mechanical Properties and Abrasion Loss of
Rubber Blends

Properties	CV60 blend	FNR blend	SAP-NR blend	SAP/SK-NR blend
Storage modulus E' (MPa)	5.51	5.17	5.08	5.07
Loss modulus E" (MPa)	0.15	0.16	0.15	0.15
Tan δ	0.03	0.03	0.03	0.03
Abrasion loss (cm ³)	0.11	0.10	0.09	0.10

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was similar to that of CV-60 blend, while it slightly higher than that of FNR blend. The insignificant difference in dynamic mechanical properties and heat build-up between the saponified rubber blends and the ordinary rubber blends is possibly due to the high amount of carbon black and SBR content, which dominate the whole properties of vulcanized rubber. However, the significant difference was observed on the dynamic compression set of rubber blends, as shown in Figure 8. It is clear that SAP-NR blend and SAP/SK-NR blend showed almost zero percentage of the dynamic compression set while CV-60 blend and FNR blend showed 7.6% and 2.7%, respectively. This suggests that SAP-NR blend and SAP/SK blend have very high elasticity.

The properties of rubber blends clearly showed that the saponified rubber will be a good material for making rubber tire, based on the following characteristics. First, the saponified rubber has low Mooney viscosity to make it easy to process. Secondly, the dynamic mechanical properties and heat build-up of the saponified rubber blend were almost similar to the ordinary rubbers. Finally, the most advantageous point of the saponified rubber blend is very low dynamic compression set.

CONCLUSION

The purification of NR by saponification of latex resulted in the decrease of nitrogen content, fatty acid ester content and gel content of NR. SAP-NR and SAP/SK-NR showed a clear decrease in Mooney viscosity and green strength. This demonstrates that the removal of a part of non-rubber components, i.e. proteins and lipids, brings about good processability of NR. The enzymatic deproteinization of NR latex resulted in the high scorch time and cure time, whereas the saponification of NR latex contributed to the low cure time. Vulcan-

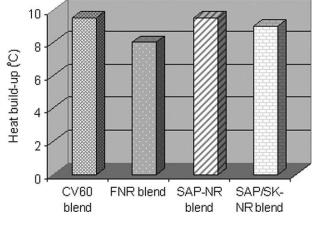


Figure 7 Heat build-up of rubber blends.

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Figure 8 Dynamic compression set of rubber blends.

ized DPNR showed low tensile properties, low storage modulus, high heat build-up and high dynamic compression set. In contrast, vulcanized saponified rubber showed the high tensile properties, high storage modulus, low heat build-up and low dynamic compression set. This clearly indicates that the purification of NR by saponification provide good physical and dynamic mechanical properties of cured rubber. Furthermore, the saponified rubber blend with SBR also showed a good performance suitable for making rubber tire.

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