3 RESULTS AND DISCUSSION

3.1 INFLUENCE OF ROTOR SPEED

Figure 1 shows that Mooney viscosity does not change significantly with increasing rotor speed, indicating low degree of mastication effect. In theory, an increase in rotor speed and thus shear rate should increase shear stress, promoting mastication. By contrast, an increase in rotor speed leads to a rise in bulk temperature, due to shear heating, which causes a decrease in shear viscosity. The decrease in shear stress therefore results and decrease the efficiency in mechanical mastication. In other words, from the result obtained, it is possible that an increase in bulk temperature as a function of rotor speed might cancel out the mastication effect.

Results of compound morphology are shown in Figures 2a to 2d. It is evident that the NR dispersed phase appears to be in an elongated structure rather than a droplet structure, which could be attributed to insufficient shear stress for disrupting the elongated dispersed phase into the droplet structure. The viscosity of NR dispersed phase might be too high and/or that of NBR matrix might be too low. However, at the rotor speed as fast as 60 rpm, the NR dispersed phase appears to be less elongated-like because of the pseudoplasticity of the blends. It is known that elastomers are highly pseudoplastic and their viscosity therefore reduces as shear rate increased.

The lower viscosity during blending at higher rotor speed promotes the droplet-like formation through the strain recovery of the elongated structure.

Additionally, phase size of NR does not depend strongly on rotor speed used for blending, which could be explained by an increase in bulk temperature due to shear heating generated as a function of rotor speed, resulting in a decrease in shear stress required for disrupting the dispersed phase. The insensitivity of phase size to a change in rotor speed has been reported previously by Favis[20] in a blend system of polycarbonate/polypropylene.

Figure 3 (and also Table 1) illustrate the relationship between relative tensile strength (used for determining oil resistance) and rotor speed. It is clear that the relative tensile strength does not change significantly with increasing rotor speed. Obviously, the results of morphology and relative tensile strength are in good agreement which leads to a preliminary conclusion that oil resistance of NR/NBR compounds is controlled by size of the NR dispersed phase. Nonetheless, further investigation of the dependence of oil resistance on blending time needs to be carried out before the final conclusion can be drawn.

3.2 INFLUENCE OF BLENDING TIME

Unlike the rotor speed, blending time appears to affect strongly the Mooney viscosity as shown in Figure 4. The longer the blending time, the lower the

Mooney viscosity. Certainly, the mastication effect is responsible for the decrease in compound viscosity.

The morphology of the blends prepared from various blending times is shown in Figures 5a to 5e. It is obvious that size of the NR dispersed phase decreases with increasing blending time up to 25 minutes. Then, the size of the dispersed phase increases again. The decrease in dispersed-phase size is attributed to the increase in total shear strain applied to the compounds. At a given shear rate, the longer blending time gives the larger total shear strain and thus the smaller dispersed-phase size. The increase in phase size of the dispersed phase with the blending time longer than 25 minutes might be the result of sufficiently long time available for collision of the unstabilised dispersed phase leading to phase coalescence.[21-22]

Figure 6 (and also Table 2) reveals the relationship between relative tensile strength and blending time. It is clear that the relative tensile strength increases with increasing blending time up to 25 minutes and then decreases with further increasing blending time. The results of morphology and relative tensile strength are in good agreement, similar to the case of rotor speed mentioned previously.

From all results obtained, it can be concluded that phase morphology of the blends plays strongly role in oil resistance as a function of relative tensile strength. The smaller the dispersed-phase size, the higher the relative tensile strength and thus, the higher the oil resistance. The proposed explanation is as follows: compared to NR, NBR possesses excellent resistance to hydrocarbon liquids. Thus, when the blends are immersed in oil, NR dispersed phase swells markedly leading to low resistance to failure of the blends. In the case of small dispersed-phase size of NR, the large surface area of the small dispersed phase of NR is surrounded by NBR phase, possessing high resistance to oil. Thus, oil swelling occurring mainly within small phase size of NR will be stopped by the surrounding NBR, resulting in high value of relative tensile strength. By contrast, a large degree of swelling in a large NR dispersed phase would be stopped ineffectively by NBR due to a small surface area of NR dispersed phase surrounded by NBR. This leads to low resistance to failure and thus low relative tensile strength.

4 Conclusions

The relationship between phase morphology, blending conditions (i.e. rotor speed and blending time) and oil resistance in 20/80 NR/NBR blend was investigated. It was found that Mooney viscosity of the blends depends more strongly on blending time than rotor speed. Size of the NR dispersed phase is approximately independent of rotor speed, but decreases with increasing blending time up to 25 minutes before increases again with a further increase in blending time. Results of relative tensile strength which is an indicator for oil resistance in the present study are in agreement with those of the blend morphology, indicating that the oil resistance in 20/80 NR/NBR blend depends significantly on phase morphology of the blend. The smaller the size of NR dispersed phase, the higher the resistance to oil of the blend.

5. Acknowledgement

Support of the present study by the Thailand Research Fund (TRF) is gratefully acknowledged.

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Table 1 Relative tensile strength of 20/80 NR/NBR blends as a function of rotor speed

Rotor speed (rpm)	Relative tensile strength
40	0.73
45	0.67
55	0.71
60	0.71

Table 2 Relative tensile strength of 20/80 NR/NBR blends as a function of mixing time

Mixing time (minutes)	Relative tensile strength
15	0.62
20	0.69
25	0.79
30	0.62
40	0.62

Figure Captions

Figure 1	Relationship between Mooney viscosity and rotor speed
Figure 2	Micrographs (with a magnification power of 200) of blends prepared from various rotor speeds : 40 rpm (a); 45 rpm (b); 55 rpm (c); 60 rpm (d)
Figure 3	Relationship between relative tensile strength and rotor speed
Figure 4	Relationship between Mooney viscosity and blending time
Figure 5	Micrographs (with a magnification power of 400) of blends prepared from various blending times: 15 mins. (a); 20 mins. (b); 25 mins. (c); 30 mins. (d); 40 mins. (e)
Figure 6	Relationship between relative tensile strength and blending time

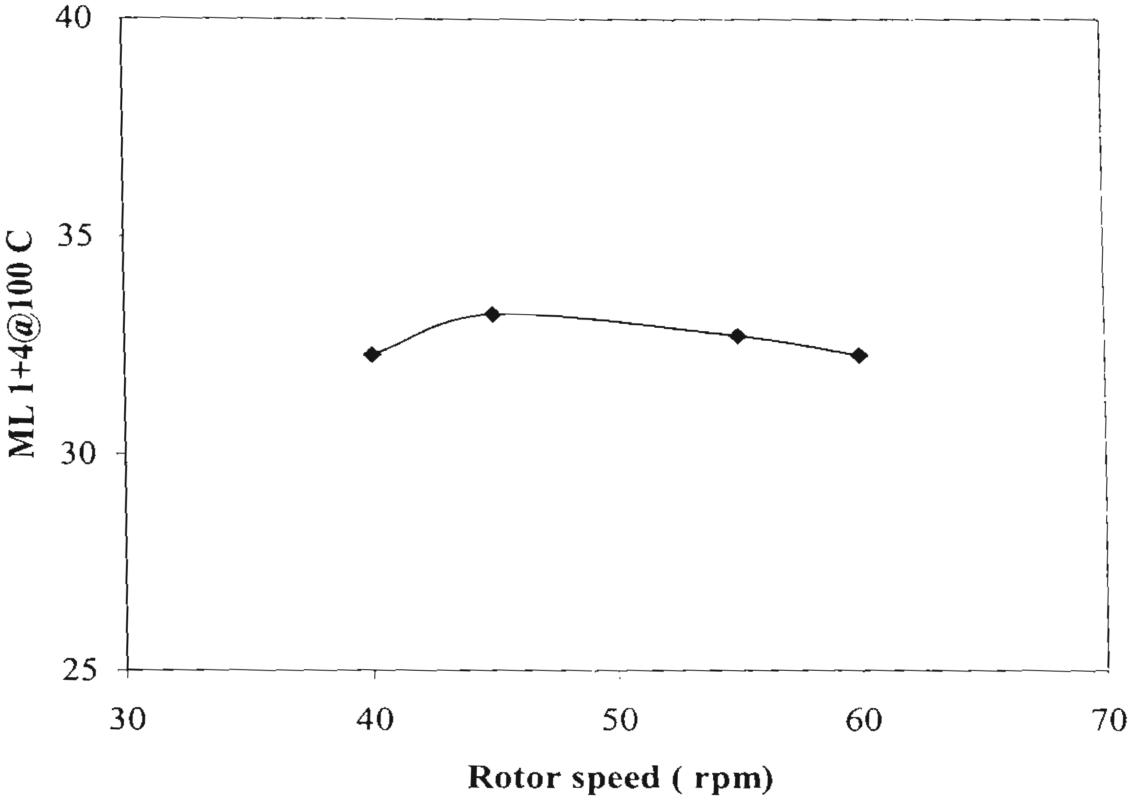
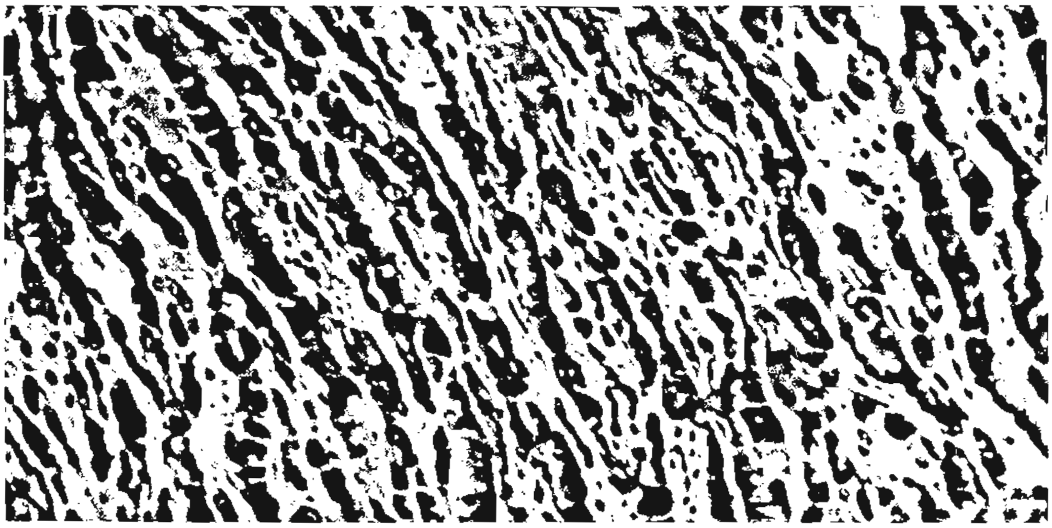


Figure 1

(a)



(b)

Figure 2a and 2b

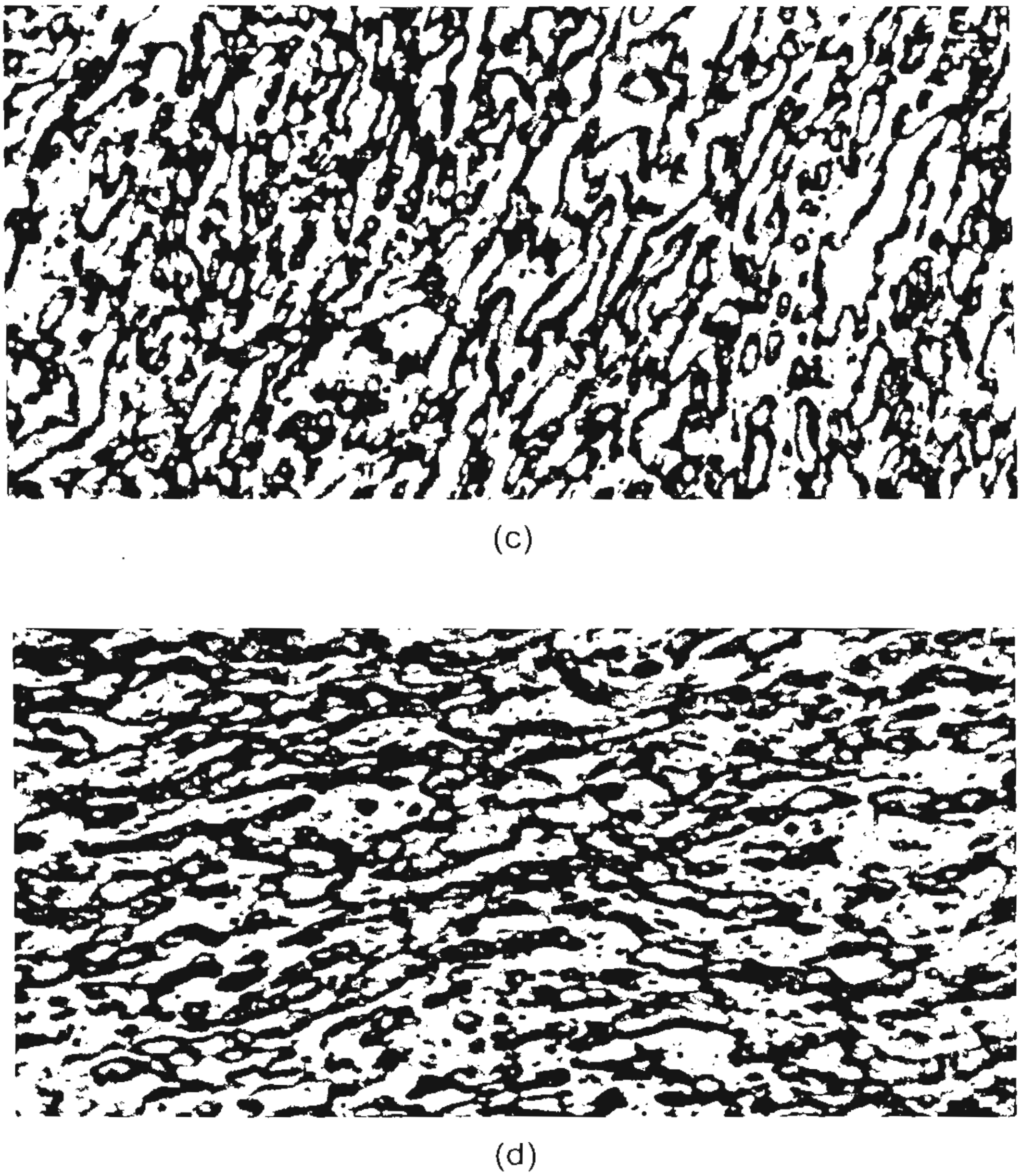


Figure 2c and 2d

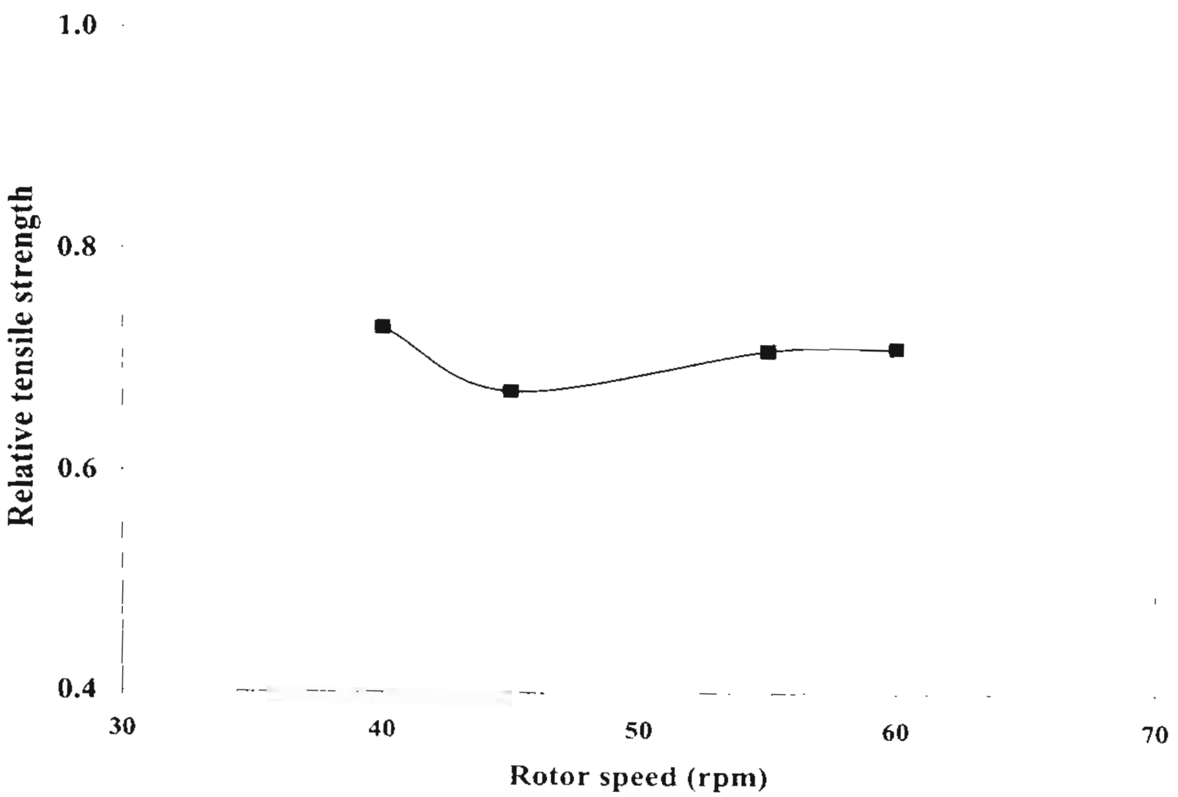

1.0
0.8
0.6
0.4
Relative tensile strength
30
40
50
60
70
Rotor speed (rpm)

Figure 3

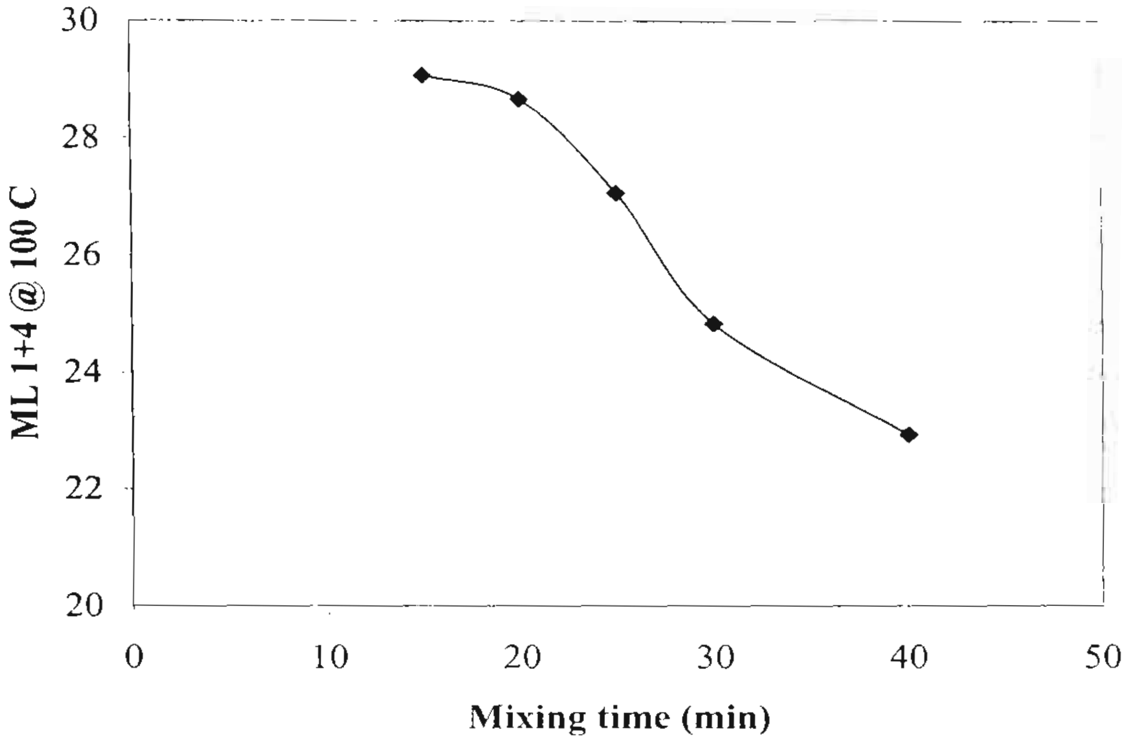
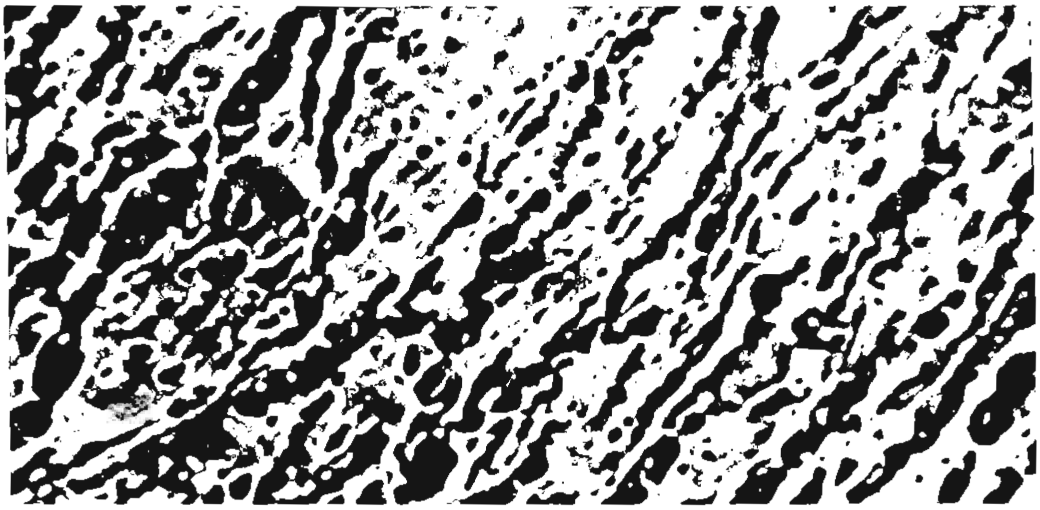
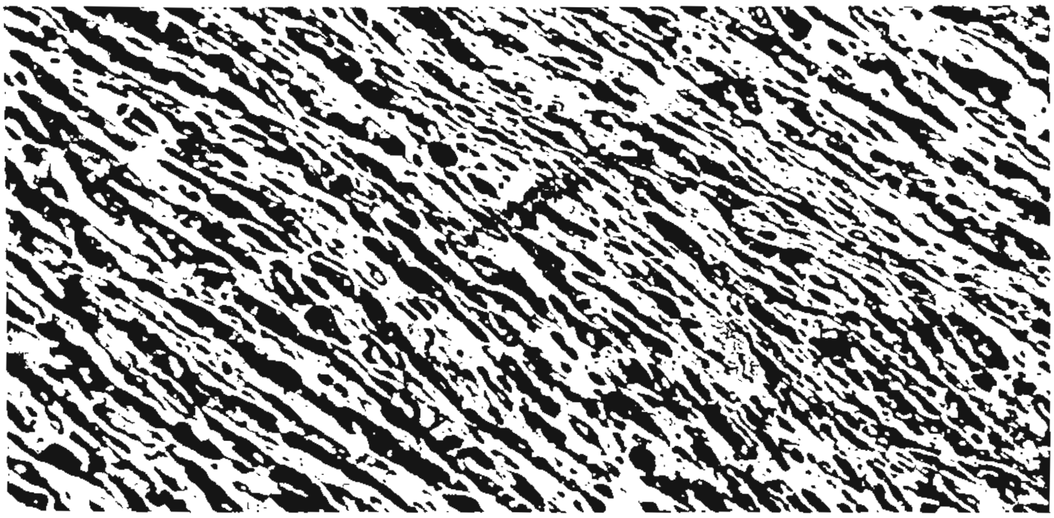


Figure 4

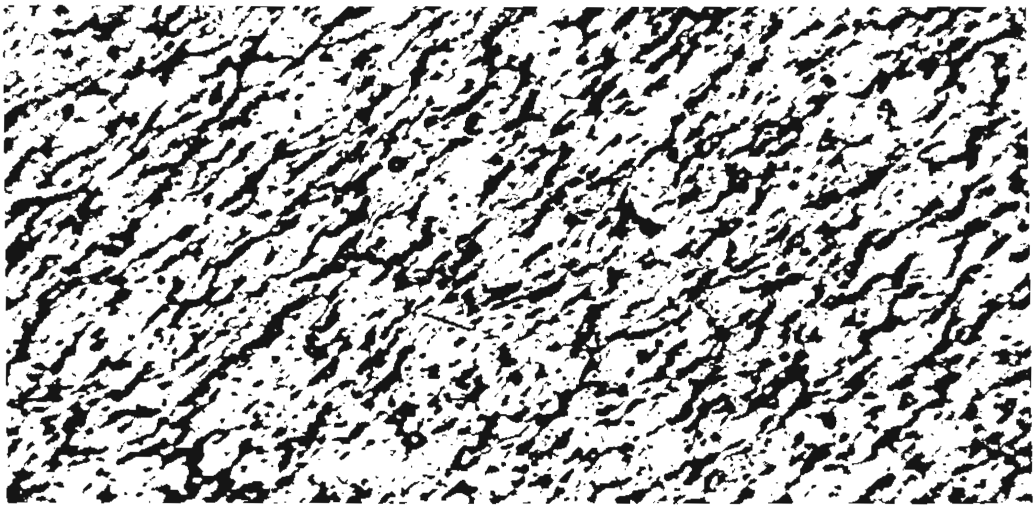


(a)

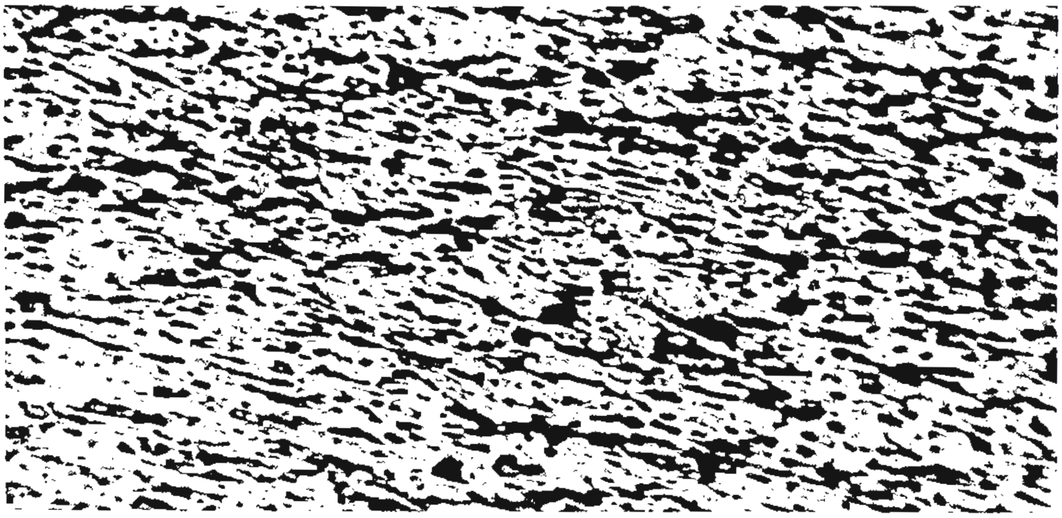


(b)

Figure 5a and 5b

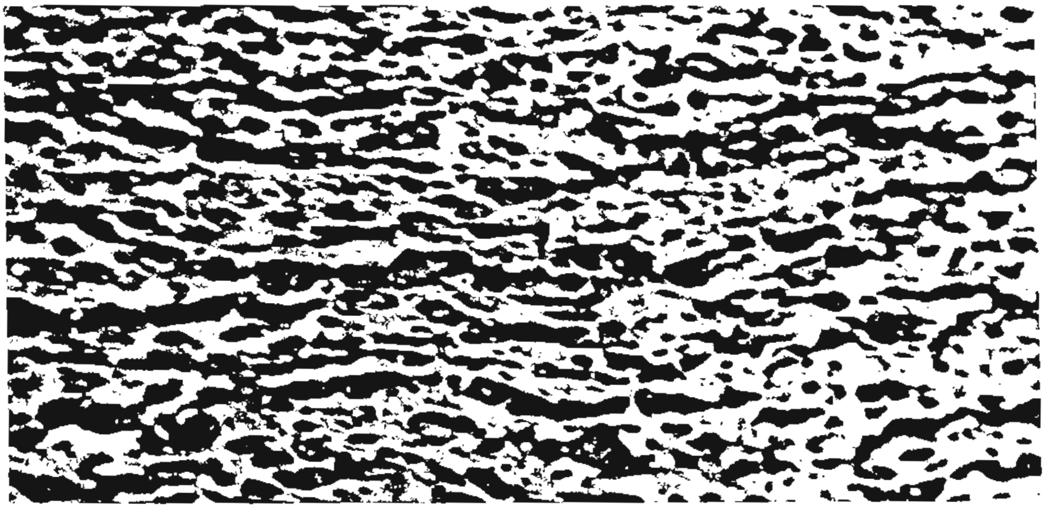


(c)



(d)

Figure 5c and 5d



(e)

Figure 5e

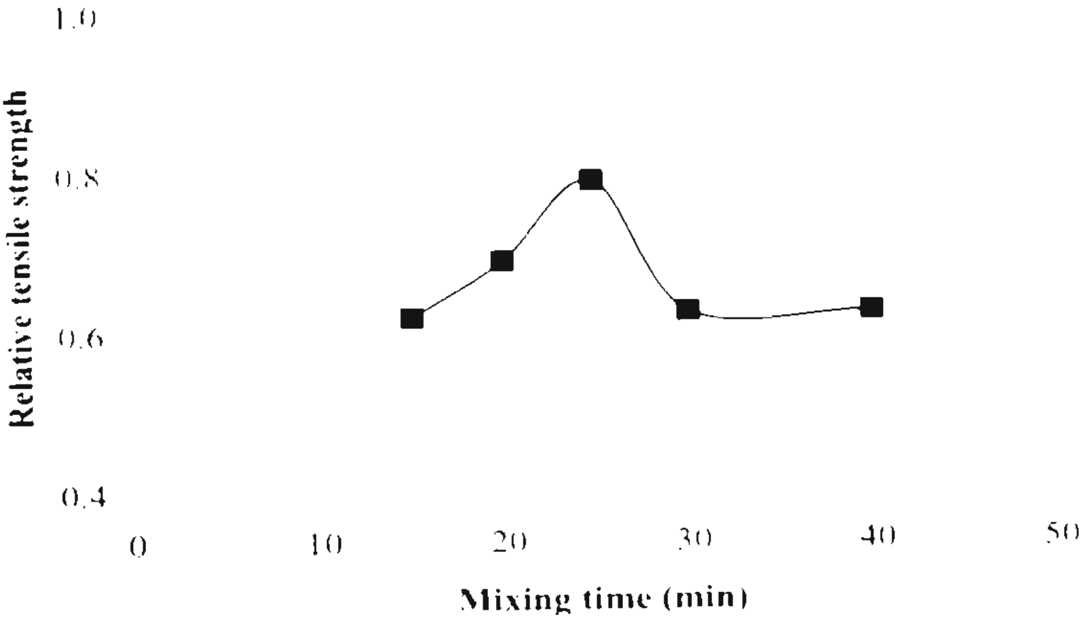

1.0
0.8
0.6
0.4
Relative tensile strength
0
10
20
30
40
50
Mixing time (min)

Figure 6

Manuscript ในหัวข้อเรื่อง

"Changes in Morphology and properties of NR/NBR blends: effect of viscosity ratio modified by LNR and ELNR "

ที่อยู่ระหว่างรอการตอบรับการตีพิมพ์ในวารสาร Plastics, Rubber and Composites

Changes in Morphology and properties of NR/NBR blends: effect of viscosity ratio modified by LNR and ELNR

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Abstract

Changes in rheological properties, morphology and oil resistance in NR/NBR blends by viscosity ratio have been investigated. In this study, the viscosity ratio was modified by mechanical mastication and an addition of liquid natural rubber (LNR) and epoxidised liquid natural rubber (ELNR). The results reveal that as viscosity ratio increased from 0.5 to 1.0, Mooney viscosity of the blends increases, and then decreases sharply as the viscosity ratio further increased from 1.0 to 2.0. The addition of LNR and ELNR for plasticising NR and NBR, respectively, does not significantly affect cure properties of the blends. Phase size of NR dispersed phase depends strongly on viscosity ratio. The high viscosity of the matrix and/or the low viscosity of the dispersed phase promote breaking up of the dispersed phase. Unexpectedly, a decrease in size of the dispersed phase by the modification of viscosity ratio via the use of low molecular weight rubber (i.e. LNR and ELNR) did not result in an improvement in oil resistance.

INTRODUCTION

Nitrile rubber (NBR) is a copolymer of acrylonitrile and butadiene. It possesses outstanding oil resistance. Natural rubber (NR) has excellent mechanical properties with relatively poor resistance to hydrocarbon liquids. The blending of NR together with NBR is intended to produce a vulcanisate with good properties from each component. However, poor properties of the blends are often obtained, mainly attributed to phase incompatibility, non-uniformity in distribution of filler[1-5] and of curatives[6-7]. Typically, morphological study has been used for characterising compatibility of the blends which significantly affects blend properties. Factors influencing blend morphology include blend composition[8-10], blending time[11], blending temperature[12], speed of mixing[12], mixing energy[12] and viscosity ratio[1,8,13]. Generally, the small phase size of the dispersed phase could be obtained by the use of long blending time, high blending temperature and similar viscosity of the blended polymers[14]. Recently, it has been reported that the addition of carbon black to NR/NBR blends could decrease dispersed phase size drastically[15]. There is an attempt to correlate the morphology of the unfilled NR/NBR blend to oil resistance of the blends[16]. It has been found that smaller size of the dispersed phase yields a higher resistance to oil. Consequently, the objective of the present paper is to further the previous study[16] where the influences of blending conditions on blend morphology and oil resistance have been reported. In the present study, the influence of the viscosity ratio on changes in rheological properties, morphology and oil resistance of NR/NBR blends will be investigated.

EXPERIMENTAL

Materials

Natural rubber or the so-called NR (STR 5, Thailand) and nitrile rubber or the so-called NBR with an acrylonitrile content of 35% (N230S, JSR), having Mooney viscosity (ML1+4 @ 100°C) of 74 and 56 respectively, were used. Peroxide curing system was chosen in order to minimise the possibility of non-uniform curative distribution between phases in the blends. In the present study, dicumyl peroxide (DCP) was used as a curing agent. Liquid natural rubber (LNR) with a viscosity-average molecular weight (M_v) of 8,413 was in-house prepared from natural rubber latex by the use of phenylhydrazine and oxygen peracid in our laboratory. Epoxidised liquid natural rubber (ELNR) with a viscosity-average molecular weight (M_v) and %epoxidation of 9,772 and 24.5, respectively was prepared from LNR using acid-hydrogen peroxide.

Mixing procedure

NR/NBR blend ratio by weight of 20/80 was chosen to ensure the morphology with NR phase dispersed in NBR matrix. Three viscosity ratios of NR to NBR used for preparing the blends were 0.5, 1.0 and 2.0, as illustrated in Table 1. To prepare the blend with viscosity ratio of 1.0, NR was masticated from the

Mooney viscosity of 74 to 56 (i.e., equivalent to NBR Mooney viscosity). For the viscosity ratio of 0.5, the masticated NR with the Mooney viscosity of 56 was further masticated with 5-phr LNR to yield the Mooney viscosity of 28. Likewise, as for the viscosity ratio of 2.0, the NBR was masticated with 5-phr ELNR to give the Mooney viscosity of 37.

The blending process was carried out in a Banbury-type internal mixer with a fill factor of 0.6, a circulating water of 40 °C and a rotor speed of 55 rpm for 25 minutes. The mixes were then sheeted on a cooled 2-roll mill and, finally, compression moulded into 2-mm thick sheets.

Mooney viscosity measurement

Mooney viscometer (Monsanto model 1500) with a large rotor at the test temperature of 100 °C was utilised for determining Mooney viscosity (ML 1+4 @100 °C), according to ASTM D1646-87 and reported in Mooney Unit. At least 5 samples were used for a measurement.

Cure property determination

Cure properties were determined from the oscillating disk rheometer (Monsanto model 100S) at the test temperature of 155 ^{o}C. At least 4 samples were determined for a measurement.

Morphological study

The vulcanisate samples were cryogenically microtomed using glass knives. The morphology of thin-sectioned samples was then observed using an optical microscope.

Oil resistance measurement

The dumbbell-shape (punched out using Die C - ASTM D412-92) test specimens were immersed in oil (Grena DX, Bangjak Petroleum, Co. Ltd., Thailand) at room temperature for 70 hours. Thereafter, the specimens were removed from the oil and quickly dipped in acetone and blotted lightly with filter paper in order to eliminate the excess oil on the specimen surfaces. Changes in tensile strength of specimens after oil immersion were used to determine oil resistance as shown in eq (1). In this study, the relative tensile strength (TS_{rel}), calculated from the ratio of tensile strength after to that before oil immersion, was used in order to eliminate the mastication effect during preparing the blends with viscosity ratios required.

$$TS_{rel} = \frac{TS_{after}}{TS_{before}} \qquad (1)$$

where TS_{before} and TS_{after} are tensile strength of specimens before and after oil immersion, respectively.

Tensile properties were measured using an Instron 4301 tensile tester with a crosshead speed of 500 mm/min and a full scale load cell of 1 kN in accordance with ASTM D638.

RESULTS AND DISCUSSION

Mooney viscosity results of the blends with different viscosity ratios are shown in Fig. 1. As viscosity ratio of NR/NBR increased from 0.5 to 1.0, the Mooney viscosity of the blends increases and then decreases sharply as the viscosity further increased from 1.0 to 2.0. The results obtained are not unusual, which are due to the dilution and plasticising effects. Since the Mooney viscosity of the blends depends strongly on matrix viscosity, the viscosity of the blends at the blend ratio of 20/80 used in the present study relies mainly on the viscosity of NBR matrix. As shown in Table 1, the blend with viscosity ratio of 2.0 is prepared by plasticising NBR matrix with ELNR. Thus, the viscosity of the NBR matrix reduces markedly leading to a substantial decrease in Mooney viscosity of the blend. For the blend with viscosity ratio of 0.5, the LNR added to plasticise NR does not reduce the Mooney viscosity of the blend system as much as does the ELNR plasticising NBR matrix, since NR is a dispersed phase in the blend ratio studied and thus does not play significant role in the viscosity of the system, compared with the matrix.

Results of cure properties are shown in Fig. 2. Only slight changes in scorch time (t_2) and cure time (t_{90}) are observed as viscosity ratio increased, indicating that the addition of LNR and ELNR for reducing the Mooney viscosity of NR and NBR, respectively, does not significantly affect cure properties of the blends.

Figs. 3a to 3c reveal the morphology of the blends with different viscosity ratios. At the high viscosity ratio of 2.0 (i.e. viscosity of the dispersed NR is higher than those of NBR matrix), the NR phase size is remarkably larger than that at the low viscosity ratios of 0.5 and 1.0, respectively. This is an evidence that high shear stress caused by high shear viscosity of the NBR matrix in the blends with viscosity ratios of 0.5 and 1.0 could transfer to the NR dispersed phase and then promotes the disruption of the dispersed phase. The results obtained are in good agreement with previous workers[12,17]. In addition, NR dispersed phase appears to be in an elongated structure rather than a droplet structure, particularly at the viscosity ratios of 0.5 and 1.0. This could probably be attributed to the low elasticity of the dispersed phase and high viscosity of the matrix, leading to a decrease in elastic recovery of the dispersed phase.

Regarding the relationship between phase morphology and relative tensile strength, which is an indicator for oil resistance used in the present study, according to the previous work[16], it has been reported that the smaller the NR dispersed phase in NBR matrix, the higher the resistance to the hydrocarbon liquid. The proposed model used for explanation (see Fig. 4) is that compared to NR, NBR possesses excellent resistance to hydrocarbon liquids, and when the blends are immersed in oil, NR dispersed phase swells markedly leading to low resistance to failure of the blends. In the case of small dispersed-phase size of NR, the large surface area of the small dispersed phase of NR is surrounded by NBR phase, possessing high resistance to oil. Thus, oil swelling occurring mainly within small phase size of NR will be stopped effectively by the surrounding NBR, resulting in high value of relative tensile strength. By contrast, the large size of the dispersed NR should yield a low relative tensile strength and thus low oil resistance. However, Fig. 5 shows the unexpected results of relative tensile strength. It appears that the blend with viscosity ratio of 2.0, possessing relatively large dispersed phase size of NR, shows higher relative tensile strength than that with viscosity ratio of 0.5. In other words, the blend with smaller size of NR dispersed phase results in lower oil resistance, which is contrary to the proposed model and to the result obtained in the previous work[16]. The peculiar result could be explained by the dilution effect. Since the blend with viscosity ratio of 0.5 was prepared by the addition of 5-phr LNR possessing poor oil resistance to the blend, the blend contains relatively large amount of the phase with poor resistance to oil (i.e. NR phase). This could lead to a significant decrease in oil resistance of the blend with viscosity of 0.5, despite of the relatively small size of NR dispersed phase. On the other hand, the blend with viscosity ratio of 2.0 was prepared by plasticising NBR with epoxidised low molecular weight natural rubber, i.e., ELNR. As a result, the blend with viscosity ratio of 2.0 would contain relatively large amount of the phase having high resistance to oil, leading to an increase in oil resistance of the blend, although the NR phase size in this blend is relatively large. Clearly, from the results obtained, it can be concluded that although the use of liquid natural rubber (LNR) could reduce the phase size of NR as a dispersed phase in NBR matrix, the addition of LNR appears to reduce oil resistance of the blends.

CONCLUSIONS

The relationship between phase morphology, viscosity ratio and oil resistance in 20/80 NR/NBR blend was investigated. It has been found that as viscosity ratio increased from 0.5 to 1.0, Mooney viscosity of the blends increases and then decreases sharply as the viscosity further increased from 1.0 to 2.0. The addition of LNR and ELNR for reducing the Mooney viscosity of NR and NBR, respectively, does not affect significantly the cure properties of blends. Phase size of the NR dispersed phase is controlled strongly by viscosity ratio. The high viscosity of the matrix and/or low viscosity of the dispersed phase promote breaking up of the dispersed phase. However, the small phase size of NR obtained by the use of low molecular weight natural rubber to modify viscosity ratio does not result in an enhancement of oil resistance which might be attributed to the dilution effect.

Acknowledgement

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Figure Captions

Fig. 1 Mooney viscosity of the blends prepared from different viscosity ratios

Fig. 2 Scorch time (t_2) and cure time (t_{90}) of the blends with various viscosity ratios

Fig. 3 Micrographs of the blends prepared from different NR/NBR viscosity ratios (x200): 0.5 (a); 1.0 (b); 2.0 (c)

Fig. 4 Proposed model of the blends with different sizes of the dispersed phase: large size (a); small size (b)

Fig. 5 Relationship between viscosity ratio and relative tensile strength

Table 1 Mooney viscosity ratio used in the present study

Viscosity ratio of NR to NBR	Mooney viscosity ratio of NR to NBR
0.5	28:56
1.0	56:56
2.0	74:37

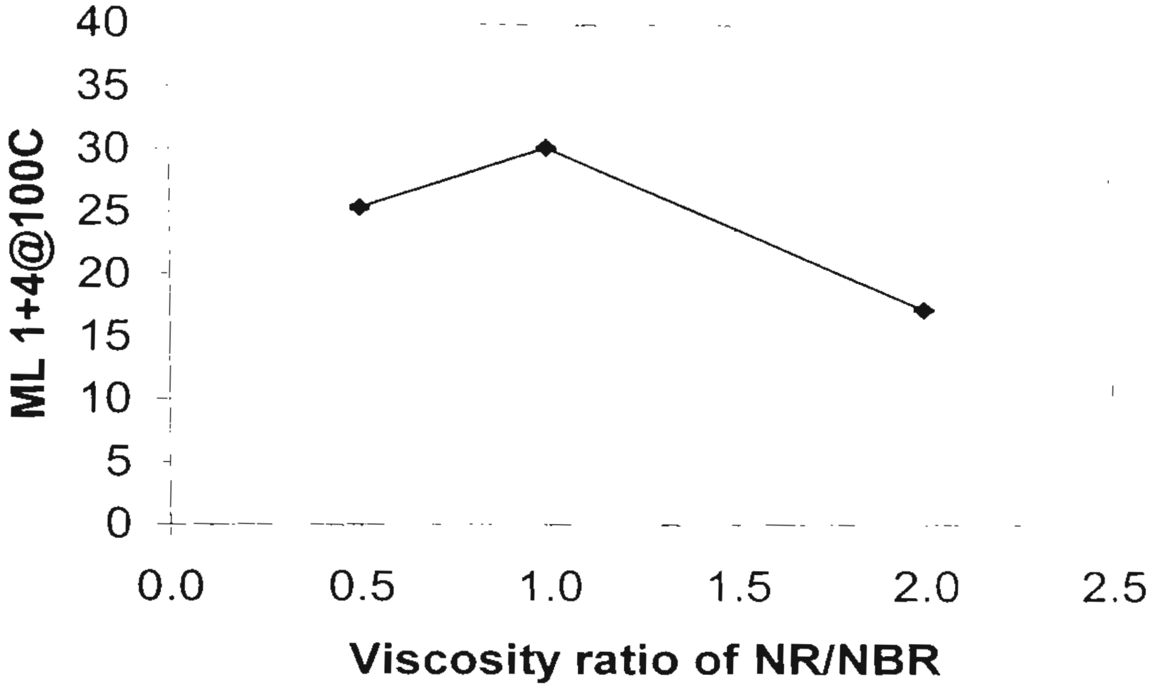


Fig. 1

C. Sirisinha et al.

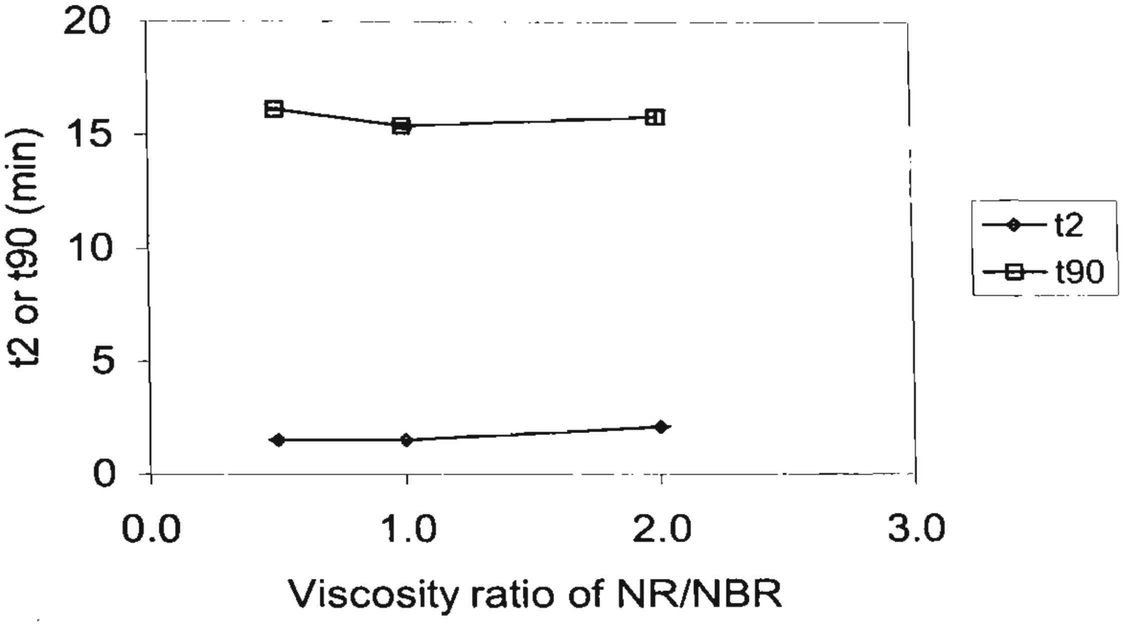
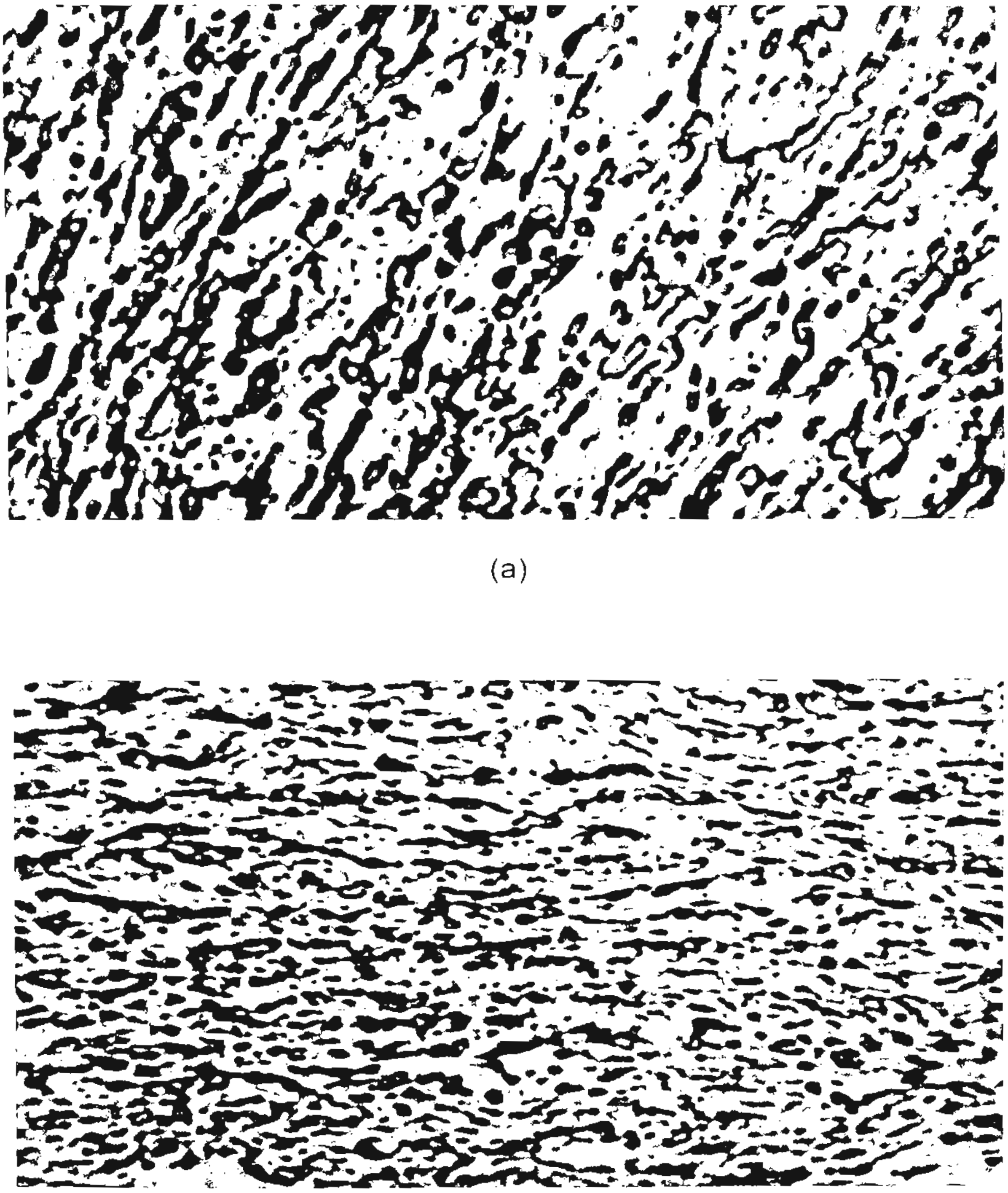


Fig. 2

C. Sirisinha et al.

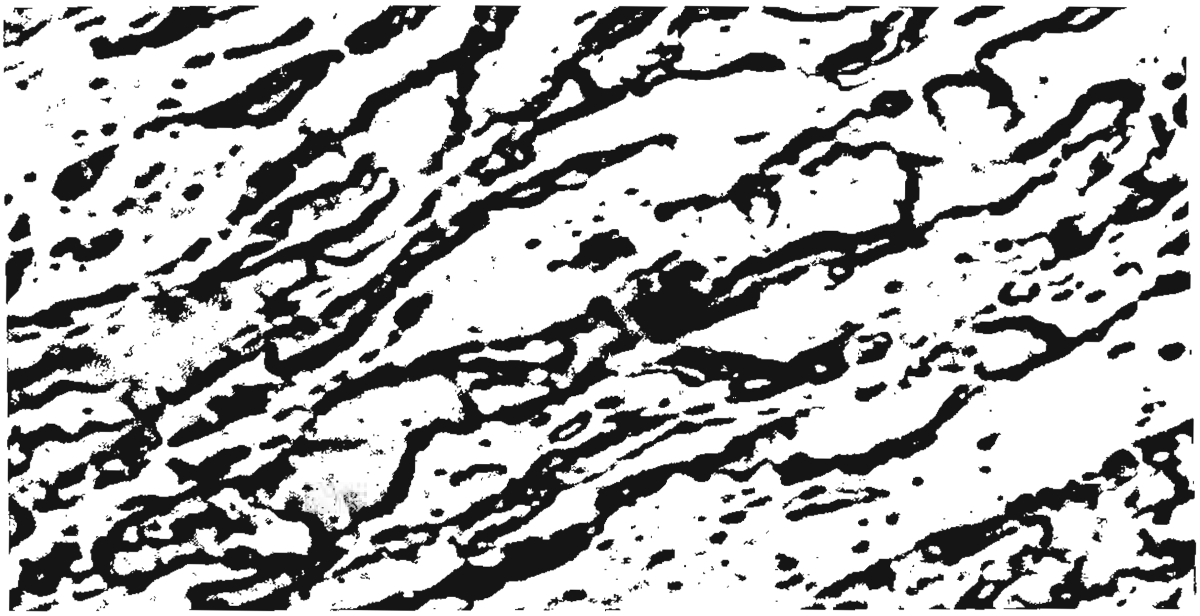


(a)

(b)

Fig. 3

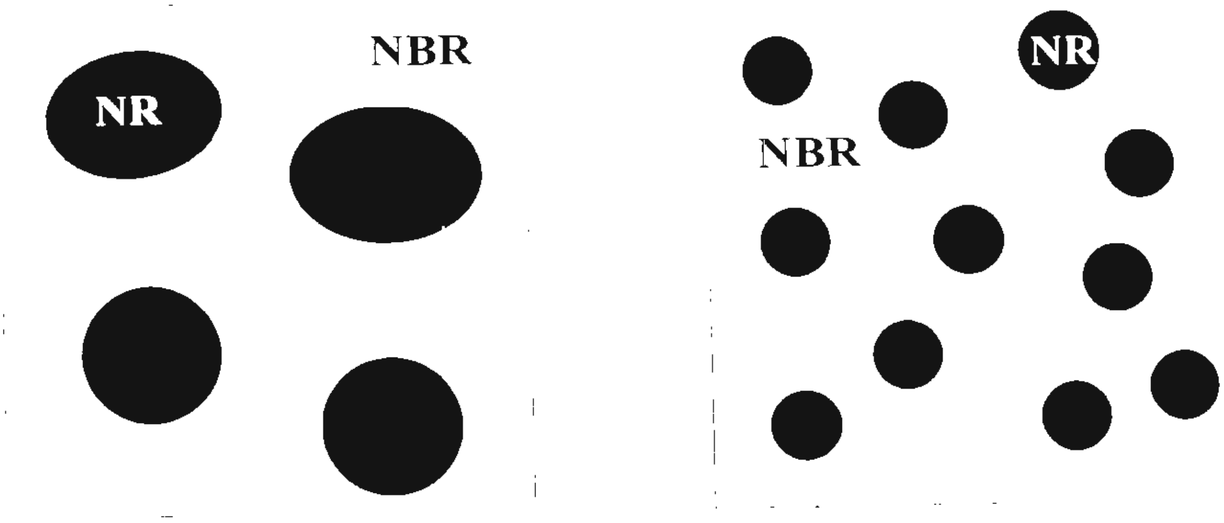
C. Sirisinha et al.



(c)

Fig. 3 (cont')

C. Sirisinha et al.



(a)

(b)

Fig. 4

C. Sirisinha et al.

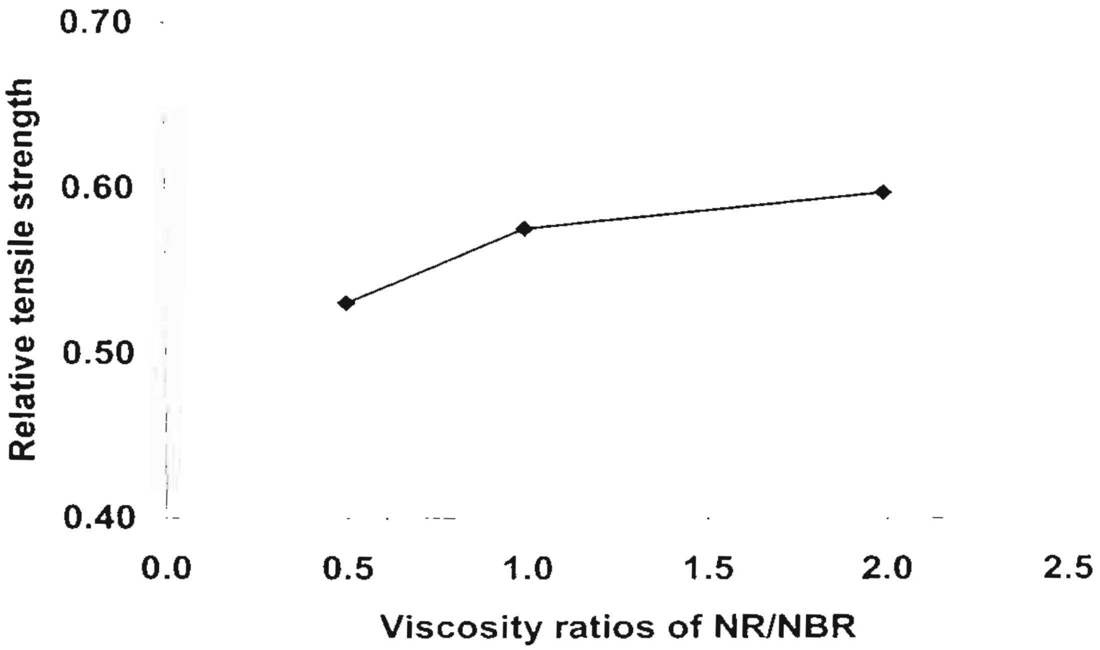


Fig. 5

C. Sirisinha et al.

Abstract ของผลงานที่เสนอ (oral presentation)ในงานประชุมนานาชาติ 8th International Seminar on Elastomer ณ ประเทศฝรั่งเศส ที่จัดขึ้นระหว่างวันที่ 9-11 พฤษภาคม 2544

FACTORS INFLUENCING MORPHOLOGICAL CHANGE AND PROPERTIES IN NR/NBR BLENDS

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Abstract

Rheological properties, morphology and oil resistance in NR/NBR blends has been investigated as functions of blending conditions. It has been found that Mooney viscosity of the blends depends more strongly on blending time than rotor speed. Size of the NR dispersed phase is approximately independent of rotor speed, but decreases with increasing blending time up to 25 minutes. Further increase in blending time, NR dispersed phase size decreases. Results of relative tensile strength which is an indicator for oil resistance in the present study are in agreement with those of the blend morphology, indicating that the oil resistance in 20/80 NR/NBR blend depends strongly on phase morphology of the blend. The smaller the size of NR dispersed phase, the higher the resistance to oil of the blend.

In addition, the influence of blend viscosity ratio modified by mechanical mastication and an addition of liquid natural rubber (LNR) and epoxidised liquid natural rubber (ELNR) on blend properties was investigated. The results reveal that as viscosity ratio increased from 0.5 to 1.0, Mooney viscosity of the blends increases, and then decreases sharply as the viscosity ratio further increased from 1.0 to 2.0. Phase size of NR dispersed phase depends strongly on viscosity ratio. The high viscosity of the matrix and/or the low viscosity of the dispersed phase promote breaking up of the dispersed phase. Unexpectedly, a decrease in size of the dispersed phase by the modification of viscosity ratio via the use of low molecular weight rubber (i.e. LNR and ELNR) did not result in an improvement in oil resistance.