Dynamic Mechanical Analysis and Tribological Properties of Fly Ash/Precipitated Silica Hybrid Filled-NR Composites

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Abstract

This work involved dynamic mechanical analysis (DMA) and tribological properties of natural rubber (NR) filled with silica hybrid filler, the silica fillers including fly ash silica (FASi) and precipitated silica (PSi). The silica contents to be filled into the NR compound were 10 and 40 parts per hundred of rubber (phr). The weight fraction FASi:PSi ratio used were 100:0, 75:25, 50:50, 25:75 and 0:100. The effect of particle size of fly ash particles was also of interest. The coefficient of frictions, specific wear rates, and wear morphology of the rubber composites were determined using Ball-on-Disc tribometer and scanning electron microscopy (SEM). The rubber-filler interaction was assessed through DMA. The results suggested that the reinforcement in this silica hybrid were obvious at the silica content of 40 phr. The mechanical properties of the FASi/PSi-filled NR vulcanizates were improved with increasing PSi fraction. There was also evidence from a decrease in tan δ_{max} and increase in T_g that some interaction between rubber and fillers in the silica hybrid-filled NR compounds. The rubber composites filled with small fly ash particles had greater overall mechanical properties than those with large particles. Weight fraction of 75% PSi was recommended for the optimum mechanical properties and wear resistance of FASi/PSi-filled NR vulcanizates.

Introduction

Natural rubber (NR) compounds have a wide range of applications, such as footwear, mat, tires, seals, etc. Generally, silica is used as reinforcing filler in non-black rubber products to improve the mechanical properties, particularly tensile strength, tear resistance, abrasion resistance, and hardness. Many attempts [1-6] have been made to use silica from natural resources, i.e. rice husk ash and fly ash, as an alternative reinforcing filler in natural rubber because of cost savings, good mechanical properties (if properly used), better dimensional stability, and environmental issues. Most articles have suggested that the white and black rice husk ash have a high silica content (ca. 90–95%), they could, in combination with a silane coupling agent, be used to replace the silica in rubber compounds. The curing rate and mechanical properties of the rice husk ash-filled rubber compounds are improved due to additional crosslinking and better filler dispersion in the matrix phase [1-3]. Thongsang et al. [4-6] found that fly ash and precipitated silica showed different reinforcing effects on NR compounds. However, the Si69-treated fly ash showed better tensile modulus and elastic properties than Si69-treated precipitated silica. Filler dispersion is an important parameter in controlling the mechanical properties of silica-filled rubber compounds.

Rubbers are known as viscoelastic materials exhibiting both elastic and damping behavior. One method which has been used to evaluate the viscoelastic behavior is dynamic mechanical analysis (DMA). This is particularly useful for identifying the molecular mechanisms of polymer materials, reinforcement, and filler dispersion in polymer composite systems [7-8]. Thongsang et al. [7] indicated that the treatment of the fly ash and precipitated silica fillers with Si69 coupling agent was hardly beneficial to the dispersion of the fillers in the rubber and rubber viscosity. The storage and loss moduli, and tan δ were marginally improved over a certain temperature range. Wang et al. [8] found that the silica containing fillers of carbon-silica dual phase, measured by the strain dependence of the elastic modulus, gave significantly higher adsorption energies than conventional silica and carbon black.

The relative motion between rubber surface and contacting substances is a topic of considerable practical importance. It has been recognized that tribological properties of rubber can be tailored upon request. Felhös et al. [9] and Karger-Kocsis et al. [10] found that the coefficient of friction (COF) and specific wear rate of EPDM rubbers were reduced with increasing carbon black. However, the COF and wear parameters strongly depended on the related test configuration.

Kaushik et al. [11] reviewed abrasion of rubber. During abrasion, ridge formation takes place on the rubber surface, which is indicative of the mechanism of wear.

The present work investigated dynamic mechanical analysis and tribological properties of NR filled with fly ash silica (FASi) and precipitated silica (PSi). The silica contents to be filled into the NR compound ranged from 10 to 40 parts per hundred of rubber (phr). The weight fraction FASi:PSi ratio used were 100:0, 75:25, 50:50, 25:75 and 0:100. The effect of particle size of fly ash particles was also of interest.

Experimental

Raw materials

Rubber and compounding ingredients

The raw rubber used in this study was natural rubber grade STR20 supplied by Creative Polymers Co.,Ltd. (Bangkok, Thailand). The formulation of the natural rubber (NR) compounds was as follows: 100 phr rubber, 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. Polyethylene glycol was added at 2 phr for precipitated silica systems.

Silica Hybrid Fillers

The silica hybrid fillers were the mixture of fly ash silica (FASi) and precipitated silica (PSi). The FASi particles were supplied by MaeMoh Power Station of KNR Group Co., Ltd. (Lampang, Thailand). The silica content in the fly ash particles used in this study was approximately 33%. The required silica content for FASi added to the rubber compounds had to be calculated with the information on the fly ash chemical compositions. The fly ash particle size of less than 25 micron and 45-74 micron were studied. The precipitated silica was Hi-Sil 233-S supplied by Tokuyama Siam Silica Co., Ltd. (Bangkok, Thailand) and had a pH of 6.8, a bulk density of 0.235 g/cm^3, and a surface area of 128 m^2/g. The silica contents to be filled into the NR compounds were 10 and 40 phr. The weight fraction FASi:PSi ratio used were 100:0, 75:25, 50:50, 25:75 and 0:100.

Surface Treatment by Si69 Coupling Agent

Bis(3-triethoxysilylpropyl)tetrasulfane (designated as Si69), [$(C_2H_5O)_3$-Si-$(CH_2)_3$-S_4-$(CH_2)_3$-Si-$(C_2H_5O)_3$], supplied by Behn Meyer Chemical (T) Co., Ltd. (Bangkok, Thailand) was used as a chemical coupling agent. In this work, the Si69 was utilized

for surface treatments of the FASi and PSi at 2.0 wt%. The surface treatment procedure was commenced by mixing 2.0 g of Si69 with 100 mL of ethanol, and then stirring for 30 min. 100 g of FASi or PSi were then added to the solution with a further 15 min stirring in order to ensure a uniform distribution of the coupling agent on the FASi and PSi surfaces. The treated FASi and PSi were then dried at 100°C for 12hrs in an oven until a constant weight was achieved.

Sample Preparation

The rubber sample preparation involved mastication, compounding, and molding processes. In the mastication step, the natural rubber was masticated on a laboratory two-roll mill (Yong Fong Machinery Co., Ltd., Samutsakon, Thailand) for 5 min and was then mixed with a specified content of treated fly ash for another 10 min. In the compounding step, the rubber and filler were compounded with prepared vulcanization chemicals on the two-roll mill for another 15 min, and the compounds were then kept at 25°C at 50% humidity before further use. The resultant rubber compound was then compression-molded to a 90% cure using a hydraulic press (Model MGLP-50AT, Mach Group Co., Ltd., Samutprakarn, Thailand) at 150 kg/cm^2, using 160°C cure temperature, in order to give vulcanized rubber. The cure time used for any individual compound was pre-determined by an oscillating disk rheometer (ODR) before conducting the vulcanization process.

Characterizations

Cure Characterizations

Cure time and delta torque values of the rubber compounds were measured with an oscillating die rheometer (Model ODR GT 7070, GOTECH Testing Machine, Taiwan) at a test temperature of 160°C.

Mechanical Properties

Tensile properties (modulus at 300% elongation, tensile strength, and elongation at break) of the rubber vulcanizates were tested according to ASTM D 412–98a (2002) with use of dumbbell-shaped samples, the tests being carried out using a universal testing machine (Model LR 50 K, LLOYD Instrument Ltd, England). Tear strength was determined according to ASTM D 624–00 (2007) 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 hardness durometer (Shore A Model 475, PTC instruments, MA, USA) was used for hardness tests, the test conditions being in accordance with ASTM D 2240–97 (1997). The abrasive resistance was evaluated with a standard test according to DIN 53516 and performed on a DIN abrader (Hampden Test Equipment, Ltd., Northants, UK).

Dynamic Mechanical Analysis (DMA)

The dynamic mechanical properties of the silica-filled NR compounds were determined using dynamic mechanical analyzer (Model DMA242, NETZSCH-Gerätebau GmbH, Selb, Germany). DMA measured the mechanical modulus and damping properties of the rubbers filled with FASi/PSi hybrid fillers. The test samples were rectangular in shape, 60mm long, 10mm wide and 2.5mm thick. Thermal scanning was performed between -80 to 20◦C, with a heating rate of 5◦C/min. A sinusoidal strain with a frequency of 1 Hz and amplitude of 50 μm was applied to the specimen. The storage and loss moduli, tan δ, and glass transition temperature (T_g) of the rubber vulcanizates were subsequently measured from these tests.

Tribological properties

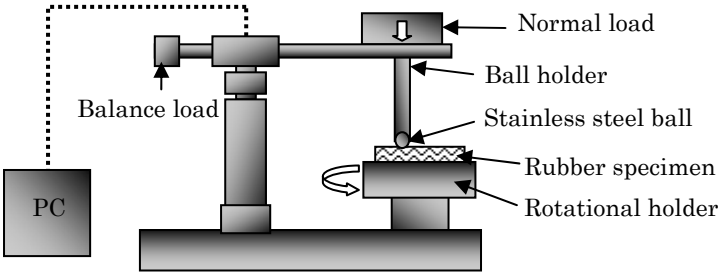


Figure 1. Tribological set-up ball-on-disc test.

Tribological set-up consisted of a ball-on-disc configuration, as shown in Figure 1, using a CSM tribometer (Peseux, Switzerland), with a stainless steel ball (diameter: 6 mm, R_a: 0.0046 μm) sliding over a rubber specimen along a circular path of 7.26 mm diameter. The specimen rotated against the stainless ball with the constant linear speed of 10 cm/s for a sliding distance of 250 mm under the normal load 2 N. The tribometer was equipped with a multi-channel electronic PC measurement unit for data acquisition. Measuring simultaneously both normal and the friction force components during the tests of coefficient of friction values were calculated and monitored during the test. The specific wear rate (W_s) was calculated by:

$$W_s = \frac{\Delta m}{\rho F L} \qquad \text{Eq. 1}$$

Where Δm is the mass loss, ρ the density of cured rubber, F the normal force, and L is the overall sliding distance.

Morphological Studies

Morphology of worn surface was studied using a SEM machine (JSM-6380LV, JEOL Ltd., Akishima-Shi, Japan) at 15 kV accelerating voltage. The experimental procedure can be obtained elsewhere.

Results and discussion

Cure characteristics of FASi/PSi-filled NR compounds

Table 1. The cure time (t_c90) and torque difference of the FASi/PSi-filled NR vulcanizates with varying weight fraction of PSi.

Cure Properties	Silica content (phr)	FASi particle size (μm)	Weight fraction of PSi (%)				
			0	25	50	75	100
Cure time; t_c90 (min:sec)	10	45-74	5:17	5:24	5:11	5:09	5:03
	40	<25	4:52	5:18	5:26	6:02	6:17
		45-74	5:06	5:22	5:28	6:13	6:17
Delta torque (dN-m)	10	45-74	52.13	57.49	58.10	58.64	57.49
	40	<25	75.52	83.73	84.74	88.47	87.11
		45-74	64.34	84.74	86.71	89.62	87.11

Table 1 shows the t_c90 cure time and torque difference for NR compounds filled with various weight fraction of precipitated silica (PSi) in fly ash silica (FASi) and precipitated silica hybrid filler. It was found that the PSi fraction had no effect on the cure time of the sililca hybrid-filled NR compounds at low silica loading. The cure time was approximately 5 min. For high silica loading, the cure time of NR compounds appeared to slightly increase with PSi fraction. Delta torque is the difference between the maximum and minimum torques obtained from the ODR rheograph which indicated the formation of crosslinks and rubber-filler interaction in the vulcanizates. It seems that the incorporation of PSi had a significant effect on the delta torque. It was also interesting that the delta torque for NR vulcanizates filled with FASi particle size of less than 25 micron was greater than that with FASi particle size of 45-74 micron, which was probably caused by the higher surface area giving the higher rubber-filler interaction in the rubber vulcanizates.

Mechanical properties of FASi/PSi-filled NR vulcanizates

Table 2 shows the mechanical properties for NR composites containing different PSi fractions in FASi/PSi hybrid filler. It can be observed that the overall mechanical properties of all silica-filled NR vulcanizates were higher than those of the unfilled vulcanizates, especially at high PSi fraction. The FASi/PSi-filled NR composites filled with small FASi particles had greater mechanical properties than those with large particles at any given PSi fractions. The change in mechanical properties of the rubber composites was corresponded with that in delta torque results, which was referred to as crosslink density and rubber-filler interaction. This involved the continual deformation in the FASi/PSi-filled NR compounds.

Table 2. The mechanical properties of unfilled and FASi/PSi-filled NR vulcanizates with different PSi weight fraction.

Properties	Unfilled	Silica content (phr)	FASi particle size (μm)	Weight fraction of PSi (%)				
				0	25	50	75	100
Modulus at 300% Elongation (MPa)	2.36 ±0.17	10	45-74	2.40 ±0.10	2.57 ±0.18	2.51 ±0.13	3.01 ±0.32	3.29 ±0.14
		40	<25	5.51 ±0.80	6.44 ±0.10	6.80 ±0.20	6.25 ±0.08	5.86 ±0.07
			45-74	4.06 ±0.16	4.40 ±0.05	5.51 ±0.14	5.87 ±0.14	5.86 ±0.08
Tensile strength (MPa)	20.39 ±1.19	10	45-74	17.74 ±0.88	17.51 ±1.20	17.50 ±0.32	19.93 ±1.04	21.72 ±1.07
		40	<25	10.43 ±0.73	12.62 ±0.50	15.28 ±1.65	19.36 ±0.41	23.97 ±0.97
			45-74	8.21 ±0.38	10.74 ±0.14	13.95 ±0.51	18.29 ±0.45	23.97 ±0.97
Elongation at break (%)	827.82 ±23.45	10	45-74	816.82 ±14.43	776.15 ±14.93	761.15 ±3.42	794.76 ±10.94	868.97 ±28.94
		40	<25	527.72 ±22.14	507.53 ±8.39	556.93 ±13.97	629.04 ±21.95	711.07 ±9.03
			45-74	515.68 ±16.32	547.57 ±14.83	552.49 ±7.31	633.24 ±6.43	711.07 ±9.03
Tear strength (MPa)	28.39 ±0.45	10	45-74	26.26 ±1.12	33.75 ±2.99	35.00 ±2.55	38.13 ±2.09	39.95 ±2.24
		40	<25	24.16 ±1.89	26.55 ±1.17	31.85 ±1.96	59.84 ±5.69	91.74 ±8.36
			45-74	21.54 ±1.03	30.20 ±1.81	35.58 ±1.92	57.88 ±3.48	91.74 ±8.36
Hardness shore A	42.50 ±0.21	10	45-74	45.50 ±0.55	41.33 ±0.26	42.16 ±0.29	42.33 ±0.26	41.33 ±0.26
		40	<25	59.00 ±2.04	60.42 ±0.58	60.16 ±1.47	63.67 ±0.81	63.10 ±0.65
			45-74	58.50 ±0.63	57.50 ±0.7	56.50 ±0.7	62.80 ±0.38	63.10 ±0.65
Volume loss (mm^3)	46.29 ±0.20	10	45-74	49.60 ±2.48	39.40 ±1.97	39.80 ±1.99	33.80 ±1.69	22.00 ±1.10
		40	<25	65.10 ±3.26	46.60 ±2.33	44.20 ±2.21	33.40 ±1.67	30.00 ±1.50
			45-74	78.60 ±3.93	58.20 ±2.91	43.40 ±2.17	37.40 ±1.87	30.00 ±1.50

Dynamic mechanical analysis

The variations in viscoelastic properties, which are usually expressed by storage and loss moduli and tan δ, with temperature for the unfilled and the FASi/PSi-filled NR compounds with different silica loadings are given in Figure 2. It can be seen that tan δ showed a peak around temperatures between -44 to 39 ºC, this being known as a glass transition temperature (T_g). The results from DMA can be summarized in terms of T_g and tan δ_{max} of NR compounds listed in the Table 3. Usually, the reinforcement of rubber caused by the rubber-filler interaction can be indicated by either decreasing tan δ_{max} or increasing T_g. The results suggest that there was some reinforcement in the FASi/PSi-filled NR compounds, especially at

high silica loadings; indicated by higher T_g and lower tan δ_{max} as compared to the unfilled NR vulcanizates. The results involved a reduction in energy loss or viscous behavior at interface region of fillers and rubber matrix [7]. The T_g of the FASi/PSi-filled NR vulcanizates was unaffected by the addition of PSi, while the change of tan δ_{max} was dependent on the level of silica loading. It can be observed that the incorporation PSi had a small effect on tan δ_{max} value of NR compounds filled with low silica content. This was not the case for high silica content; the intensity of tan δ_{max} decreased with increasing PSi weight fraction, which corresponded very well to the mechanical properties results as shown earlier.

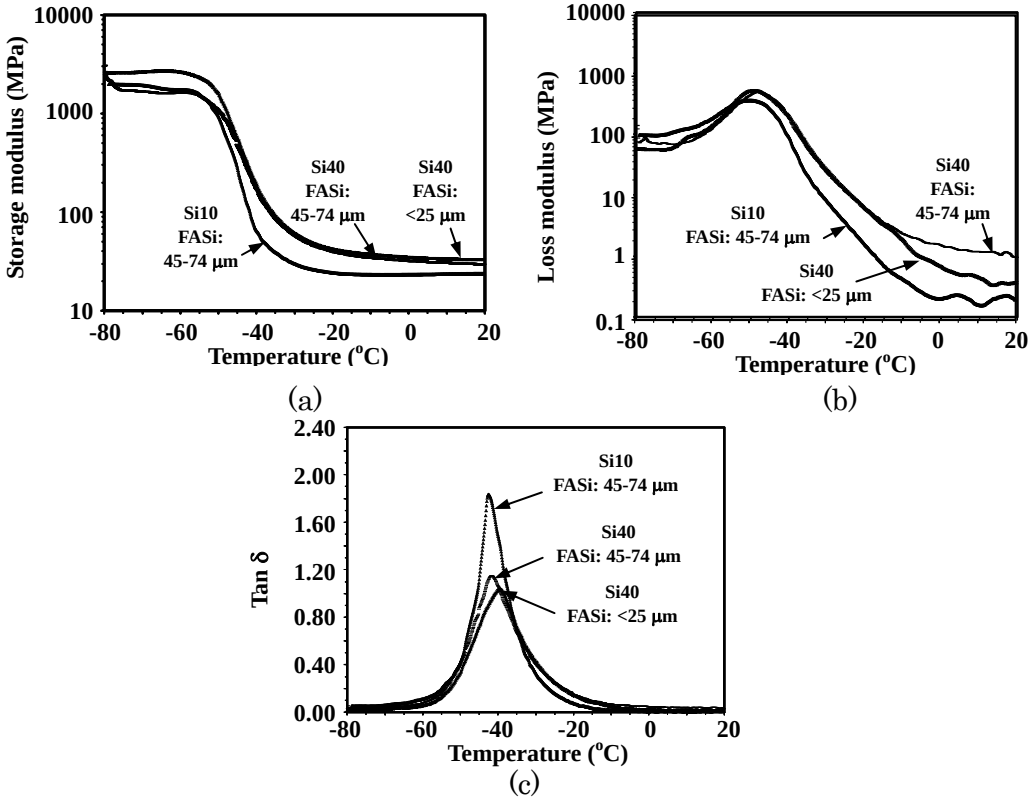


Figure 2. Storage modulus VS. temperature (a), loss modulus VS. Temperature (b) tan δ VS. Temperature (c) traces of NR vulcanizates filled with 75% of PSi weight fraction in FASi/PSi hybrid filler.

Table 3. Tan δ_{max} and T_g values of unfilled NR and FASi/PSi-filled NR composites.

Properties	Unfilled	Silica content (phr)	FASi particle size (µm)	Weight fraction of PSi (%)				
				0	25	50	75	100
T_g (°C from tan δ_{max})	-44.3	10	45-74	-41	-41.7	-43.1	-42.5	-40.4
		40	<25	-41.5	-40.8	-42.3	-39.6	-39.8
			45-74	-40.7	-39.2	-41.4	-41.8	-39.8
Tan δ_{max}	1.78	10	45-74	1.52	1.95	2.03	1.84	1.95
		40	<25	1.78	1.62	1.49	1.03	0.95
			45-74	1.72	1.76	1.56	1.15	0.95

Tribological properties

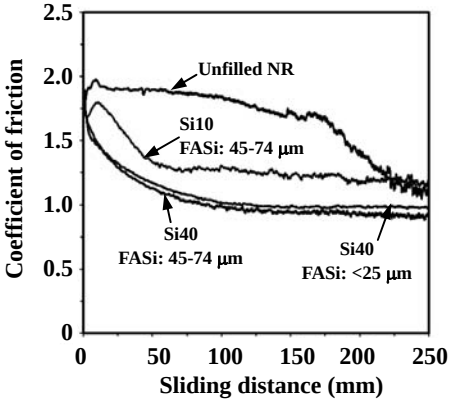


Figure 3. Change of the coefficient of friction (COF) during the ball-on-disc tests for the unfilled and the FASi/PSi-filled NR composite having 50% of PSi weight fraction.

Figure 3 illustrates the changes in the coefficient of friction (COF) of the unfilled and the FASi/PSi-filled NR compounds as a function of sliding distance. It can be seen that the initial COF value measured was very high owing to the formation of wear track by ploughing of the stainless steel ball that penetrates into the rubber to change the contact geometry. One can observe a decrease in the COF value prior to its leveling off (reaching a steady-state friction value) since the real contact area was decreasing in the sliding process. There was a significant difference in the COF behavior between the unfilled and the filled rubber vulcanizates. The COF-drop region of unfilled NR compound seemed to be the longest; the COF-drop region was shorter with higher silica loading. This was expected since the rubber initially removed and then fully formed the wear track due to the different real contact area relating to hardness of rubber compounds as given in Table 2. The greater the hardness value, the lower the real contact area and the deformation of the composites under the same applied load. Therefore, the high hardness value of the filled NR compounds with high silica content had a low real contact area concerned an ease for wear track formation on rubber surface.

The friction and wear were associated with a steady-state COF. The steady-state behavior for COF value, after around 225 m, is summarized in Figure 4. The friction force between rubber and hard surface has two contributions commonly described as the adhesion and hysteric component, respectively [12]. The friction force of rubber composite is expressed by the relationship: $F = F_{adh} + F_{hyst}$, where F_{adh} is adhesion force, and F_{hyst} is hysteric force. In this paper, part of the friction coefficient were obtained from the adhesion F_{adh} between the NR composites surface and the stainless steel ball relating to the hardness of rubber compounds,

leading to the real contact area formation. The higher hardness value the lower adhesion force. The other contribution was due to the friction force F_{hyst} from the internal friction of the rubber composites directly associated with elastic properties of rubber composites (i.e., elongation at break) [13]. It was found that the COF of the FASi/PSi-filled NR composites with high silica content were lower than that with low silica content due to low adhesion and hysteric force. The COF value increased with increasing PSi fraction both silica loading of 10 and 40 phr because of an increase in hysteric force of rubber composites, whereas the adhesion force of composites were resembled. The particle size of fly ash had also a significant effect on the COF. It can be observed that the COF of the FASi/PSi-filled NR composites with small fly ash particle were greater than those with large particles due to a higher hysteric force of rubber composites, whereas the adhesion force of composites were resembled.

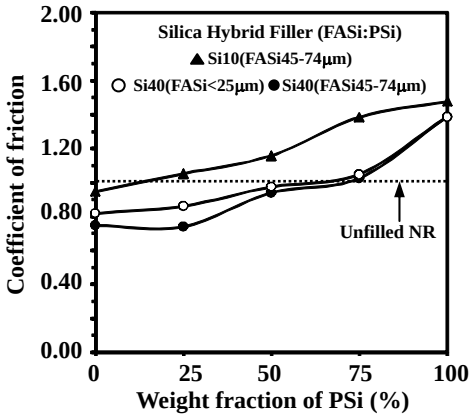
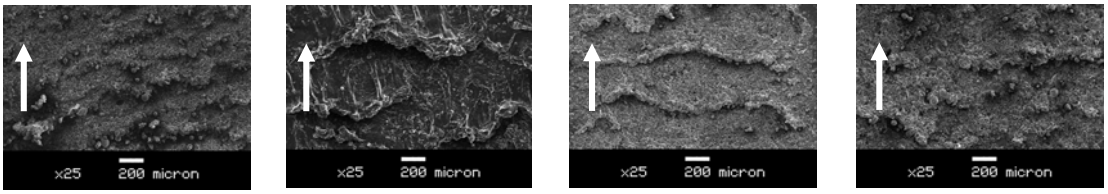


Figure 4. Steady-state COFs for the FASi/PSi-filled NR vulcanizates at various weight fraction of PSi.



(a) (b) (c) (d)

Figure 5. SEM micrographs of worn surface of the FASi/PSi-filled NR compounds with silica content of 40 phr. The arrow indicates the sliding direction.

(a) 0% of PSi (100% of FASi) (b) 100% of PSi

(c) 50% of PSi (FASi <25μm) (d) 50% of PSi (FASi 45-74μm)

Figure 5 illustrates SEM micrographs of the FASi/PSi-filled NR compounds. It can be seen that it had small ridges formation or torn tongues which became easily fragmented for silica hybrid-filled NR composites with 0% of PSi fraction (cf. Figure 5a), the reasons being associated with low tensile and tear strength, and low

elongation at break. In contrast, the silica hybrid-filled NR composites with 100% of PSi fraction had stiff roll formation or wavy pattern (cf.Figure 5b). This could be reasoned by the continuous phase and high elongation, leading to the compressed rubber surface in front of the contact area undergoing a buckling which produced detachment waves which propagate from the front-end to the back-end of the contact area, called Schallamach waves [12]. It seemed that the high elongation of filled NR compounds with small fly ash particle showed more evident roll formation than that with large particle size (cf. Figure 5c and 5d). The kind of rubber formation on worn surface may be another reason for the different COF values of NR compounds.

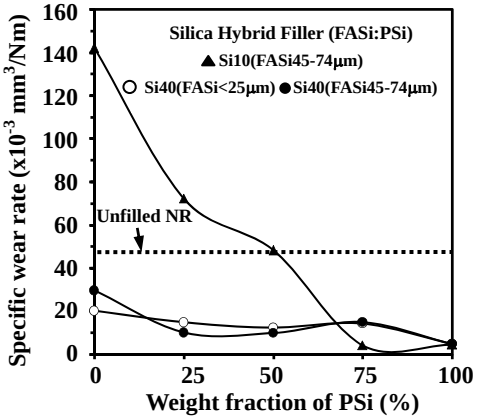


Figure 6. Specific wear rate as a function of PSi weight fraction for the FASi/PSi-filled NR composites.

Figure 6 shows the specific wear rate as a function of PSi weight fraction for the FASi/PSi-filled NR composites. The results are reported in terms of the specific wear rate of the vulcanizates; the greater the specific wear rate, the lower the wear resistance. It was found that for the FASi/PSi-filled NR vulcanizates at low silica content, wear resistance sharply increased with increasing PSi fraction. The NR composites filled with high silica content had higher wear resistance than those of rubber vulcanizates with low silica content. The wear resistance of NR compounds increased with incorporation of PSi.

Conclusions

In this work, the dynamic mechanical analysis and tribological properties of FASi/PSi-filled NR composites were studied by varying the silica content, fly ash particle size and weight fraction of PSi in hybrid filler. The results indicated that the rubber-filler interaction and the reinforcement in this silica hybrid were obvious at the silica content of 40 phr. The mechanical properties of the

FASi/PSi-filled NR vulcanizates were improved with increasing PSi fraction. There was a reinforcement in the silica hybrid-filled NR compounds by a decrease in tan δ_{max} and increase in T_g. The rubber composites filled with small FASi particles (<25 micron) had greater overall mechanical properties than those with large particles (45-74 micron) at any given PSi fractions. The weight fraction of PSi was 75% for optimum mechanical properties and wear resistance of FASi/PSi-filled NR vulcanizates.

Acknowledgments

The authors would like to thank the Commission on Higher Education and the Thailand Research Fund (MRG5280045 and RTA5280008) for financial support throughout this research work.

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