Epoxidised Natural Rubber - An Interface Modifier for Natural Rubber - Silica Composites

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Abstract

Low rolling resistance tyres are preferred, in automobiles for fuel saving. Replacement of carbon black with silica, in tyres, is reported to give low rolling resistance. In hydrocarbon rubbers, unlike carbon black, use of silica leads to incompatibility of the two, resulting in poor reinforcement. This problem is conventionally being overcome, by using silane coupling agents. Higher cost of coupling agents is a limitation to its widespread use in tyres. Silica filler is reported to have strong interaction with epoxidised natural rubber (ENR). Present work is an attempt, to replace coupling agent used in silica filled natural rubber (NR) based tyre compounds, by substituting a part of natural rubber with small quantities of ENR. ENR is expected to perform as a silica-rubber coupling agent or as an interface modifier. The property variations with replacement of NR by ENR was studied. Two cure systems were tried viz; cyclohexyl benzthiazyl sulphenamide (CBS) alone and in combination with diphenyl guanidine (DPG). In CBS accelerated systems, substitution of NR to a limiting concentration by ENR, resulted in improved cure characteristics and technological properties. With dual accelerator system also, improved technological properties were observed with the incorporation ENR. ENR modified composites, were compared with unmodified NR-silica compounds and those containing modifiers such as glycol and silane coupling agents and also with conventional CBS accelerated carbon black filled system. The thermal degradation behaviour and filler morphology of the different systems were also compared.

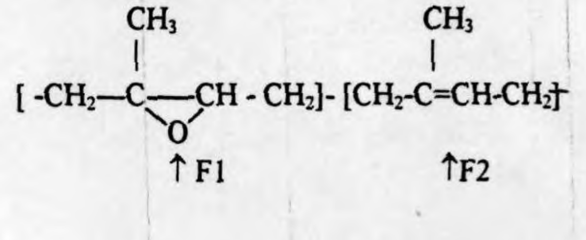
1. Introduction

The conventional reinforcing filler being used in various rubber products is carbon black, a petroleum derivative. The sources of petroleum are fast depleting. For a sustainable environment, attempts are being made to reduce the dependence on petroleum and its derivatives. Moreover burning of petroleum fuels causes emission of several gases, which pollute the air and contributes to the so called 'green house effect'. Automobile sector depends to a large extent on petroleum and its derivatives, as a fuel and for reinforcement of tyres. Nowadays low rolling resistant, energy efficient tyres are preferred in automobiles for fuel saving and to preserve the environment. In tyres, replacement of carbon black with silica, a mineral filler is reported to reduce rolling resistance(1). Unlike carbon black, use of silica as a filler for hydrocarbon rubbers, will lead to the incompatibility of the two, resulting in poor reinforcement. This problem is conventionally being overcome by the use of silane coupling agent, which acts as an interface modifier, that combines silica and rubber. The higher cost of coupling agents is a limitation to the widespread use of silica as a filler in tyre and non tyre products(1) . This paper explores the possibility of using epoxidised natural rubber (ENR) as an interface modifier for the silica filled natural rubber (NR) compounds.

2. Scope of the work

ENR is reported to have strong interaction with silica (2). Silane coupling agent and epoxidised natural rubber possess bifunctionalities and their structures are given under(1,2) .

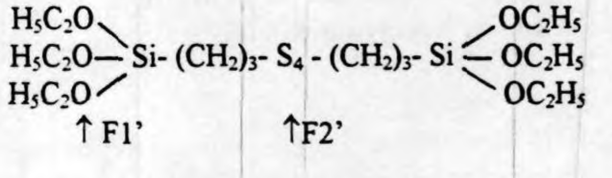
Epoxidised natural rubber (ENR-50)



$$[-CH_2-\overset{CH_3}{\overset{|}{C}}\underset{O}{\diagdown\diagup}CH-CH_2]-[CH_2-\overset{CH_3}{\overset{|}{C}}=CH-CH_2]$$

Silane coupling agent (Si 69)

[Bis - (3 - triethoxy propyl) silyl tetra sulphide]



$$(H_5C_2O)_3Si-(CH_2)_3-S_4-(CH_2)_3-Si(OC_2H_5)_3$$

Both materials possess functional groups that can interact with the surface hydroxyl groups of silica (F_1 and F_1') and with the rubber through sulphur bridges (F_2 and F_2') (1, 3). Hence it is likely that, ENR may serve also as a coupling agent. Moreover, the oxirane ring of the ENR, makes it more polar than natural rubber. Thus it may also serve as a compatibiliser for the nonpolar natural rubber and the polar hydrophilic silica particles(3). Chemisorptive linkages between carbon black and olefinic rubber has been reported elsewhere (4, 5, 6).

In the present work, silica filled NR compounds were modified with ENR, by subsituting small quantities of NR with ENR, without altering the total rubber content. Modified compounds were compared with those containing silane coupling agent (Si 69) and diethylene glycol (DEG) and also with NR- ISAF composites. In this laboratory we have also attempted the same technique for improving the silica reinforcement of styrene butadiene rubber and acrylonitrile butadiene rubber. Promising results were obtained(7).

3.Experimental

3.1. Materials

Natural rubber - ISNR 5, Pilot Crumb Rubber Factory, Rubber Board, Kottayam.
ENR (50 mole % epoxidised natural rubber) - prepared by epoxidising NR latex using performic acid generated *in situ*(8)
Precipitated silica - Ultrasil VN3,
Coupling agent - Si 69 - Degussa AG., F.R.G.

The other ingredients used were of rubber grade.

3.2. Mixing of compounds

Recipes of the various mixes used for the study are given in Tables 1 and 2. NR and NR/ENR blends were masticated/blended in the two roll mixing mill for five minutes. The masticated rubber/blend was used for making the compound. The compounds were made in the laboratory model Intermix (KO MK_3) with the following schedule.

0 - Rubber/blend
30 - Silica/carbon black (1/4) + coupling agent (1/4)
120 - Silica/carbon black (1/4) + coupling agenet (1/4)
210 - Silica/carbon black (1/4) + coupling agent (1/4)
300 - Silica/carbon black (1/4) + coupling agent (1/4) + Process oil
360 - Zinc oxide + stearic acid
420 - Accelerator + sulphur
600 - Dump

The final mix was homogenised in the two roll mixing mill.

3.3 Testing:

Cure characteristics of the compounds were determined using Monsanto Rheometer R-100 at 150°C. The test samples were prepared, by vulcanising the compound at 150°C upto their optimum cure times(t_{95}), in an electrically heated hydraulic press. Test procedures followed for various testings are given below.

Stress-strain properties (modulus, tensile strength, elongation at break) ASTM D-412-80 (zwick UTM)
Tear strength-ASTM D-624-91 (zwick UTM)
Compression set - ASTM D-395-89-Method B
Heat buildup - ASTM D-623 (Goodrich Flexometer)
Hardness - ASTM D-2240-95 (Shore A)
Resilience - ASTM D-1054 (Dunlop Tripsometer)
Abrasion resistance - DIN 53516
Ageing - ASTM D-573-88

Scanning electron microscopy was used for studying the dispersion of silica in the samples. Samples of vulcanised rubber were cut using a sharp blade. Cut surface (about 2mm^2) was gold coated and examined under the microscope.

4. Results and Discussion

4.1 Cure characteristics

It is generally seen that, with identical cure system, silica filled rubber compounds showed lower rate and extent of cure than that containing carbon black. This is caused by the active silanol groups that are present on the surface of the silica particles which absorb the polar chemical additives, such as accelerators, thus rendering it unavailable for curing (9).

Table 3 gives the cure characteristics of the mixes given in Table 1, for which, CBS was used as the accelerator. Changes in cure characteristics, on incorporation of various modifiers, into the silica filled natural rubber compounds were studied. Compounds containing ENR, Si 69 and DEG as modifiers were compared with an ISAF filled NR compound. ENR modified compounds showed, improved cure characteristics over the control compound. Scorch time was found increased and cure time reduced with ENR incorporation. With an ideal concentration of ENR, optimum cure time (OCT) and cure rate index (CRI) were comparable with those of the compounds containing coupling agent, while with regard to carbon black, OCT was higher and CRI was lower. With ENR incorporation, the improvements noted in cure behaviour may have been resulted from the silanol-epoxy interaction (2). This indirectly may have reduced the chances of accelerator absorption by the silanol groups. The increased cure torque (Δ, Rheometric torque) observed for NR/ENR blends over NR also supports this view.

Table 4 gives the cure characteristics of the dual accelerated compounds, the recipes of which are given in Table 2. In this case only minor increase in scorch time and optimum cure time was noted with ENR incorporation, while cure rate index does not show any difference from that of the NR-silica composite. The secondary accelerator added might have covered up the effect of accelerator sorption on the filler surface. Compared to modified and unmodified NR-silica composites, ENR-silica showed a higher rheometric torque. This might be a consequence of the very high interaction of ENR with silanol groups.

4.2 Strength properties.

a) Stress-strain properties

Stress-strain properties of selected samples from mixes given in Tables 1 and 2 are given in Figures 1 and 2 respectively . In Figure 1, CBS cured systems are compared. ENR containing samples showed improvement in stress-strain properties over those of NR-silica and NR-DEG-silica, while it is less significant compared to the composites based on NR-Si 69 silica and NR-ISAF. With the use of modifiers a higher elongation was imparted to the NR-silica composites. Figure 2 compares the stress-strain properties of ENR modified samples with those of the others. With ENR addition, the stress strain properties have improved over the control and are closer to those realised with 2% coupling agent. Samples with 8% Si 69 showed the highest stress improvement.

Modulus data of the samples made from the two systems of cure are given in Figure 3 and in Tables 5 and 6. Modulus at 10% elongation is considered to be a measure of filler dispersion, the lower the modulus, the better the dispersion (10). NR-ISAF and NR-DEG-silica showed the lowest 10% modulus. Any considerable variation in 10% modulus was not observed for ENR and Si 69 modified compounds over the control compound. 300% modulus was found increased with incorporation of modifiers except DEG. With CBS cure, ENR and coupling agent improved modulus. With combination of accelerators, modulus of the samples containing silane coupling agent was found enhanced further than the single accelerator, while ENR modified could not. In a two phase elastomer system, preferential migration of accelerators to any one phase may reduce the crosslink density in the other (11). NR-ENR blend being a two phase system, a similar phenomenon may have occurred, affecting its modulus. Silane coupling agent, on the other hand, also being a crosslinking agent, might have increased the crosslink density. Thus the modulus enhancement could be explained (12). DEG being only an interfacial agent could not improve the modulus, in both systems of cure.

ENR-silica and NR-ISAF composites exhibited closer modulus values with CBS cure, while with secondary accelerator a minor modulus drop was observed for the former. The low modulus observed with the combination of accelerators may be due to the reduced silanol epoxy interaction, caused by the secondary accelerator, DPG.

b) Tensile strength and elongation at break

Variations in tensile strength and elongation at break of ENR modified composites are compared in Figure 4. With ENR addition, tensile strength, a measure of reinforcement was found increased and elongation at break decreased. For rubber filler composites, the most important requirement for obtaining good reinforcement is the interaction between the two. The enhanced tensile strength and decreased elongation at break with ENR incorporation might be a consequence of the better rubber silica interaction achieved through ENR. Both cure systems compared in the figure showed similar trend. The strength enhancement with ENR addition is sharper with CBS cure, and it was found further improved, with incorporation of secondary accelerator. The strength enhancement is not continuous with ENR addition, but reaches a maximum with certain optimum concentration and then showed a declining trend. The drop in strength with higher concentration of ENR might be a consequence of the cure mismatch of the two different rubber phases (11).

Strength and elongation of silica filled NR modified with conventional interfacial agents and ENR are compared with those of the ISAF filled NR and silica filled ENR in Tables 5 and 6. The improvement with modifiers could be better identified with CBS cure. The highest strength was observed with NR-ISAF and NR-Si 69-silica followed by NR-ENR-silica, NR-DEG-silica and ENR-silica in the descending order. With DPG addition, silica filled compounds showed improved strength properties over CBS cure and is in the order of NR-Si 69 silica and NR-ENR-silica)NR-silica)ENR-silica. All silica composites exhibited enhancement in strength and elongation with CBS-DPG combination, except NR-Si69-silica, for which strength has improved and elongation decreased. ENR-silica composites vulcanised with binary cure system exhibited increased strength and elongation over that with CBS.

4.3 Tear strength and abrasion resistance

Improvement in tear strength and abrasion resistance is considered as a measure of enhanced filler reinforcement (13). Variation of these properties for different compositions of silica filled NR/ENR blends is given in Figure 5. Tear strength was found increased with ENR incorporation, for both the cure systems. The increase is sharper with CBS cure. Intermittent deviations in the direction of tear path was observed for the samples containing ENR and coupling agent as modifiers. The deviation in the tear path, normal to the direction of tear, also known as 'knotty tear' is associated with improved rubber-filler interaction of the ENR containing samples (14).

Table 5 gives some interesting features. With CBS cure silica filled NR/ENR blend showed higher tear strength than the corresponding NR or ENR; while the abrasion resistance of ENR and NR/ENR blend is very close and is superior to the control sample. Silane coupling agent performed well with CBS cure. Tear strength has improved on addition of coupling agent. Samples containing optimum concentration of ENR also gave higher tear strength, but was less than that which contained 8% coupling agent. DEG also could improve the tear strength and abrasion resistance of silica filled NR, but was lower than that of ENR or Si 69 modified. Data of the binary accelerated systems are given in Table 6. Tear strength and abrasion resistance of all the compounds have further improved over that of the single accelerator. Much variations in tear strength was not observed with concentration of coupling agent. Improved tear strength observed with coupling agent, could also be achieved with NR/ENR blends and is closer to that of the black filled. Incorporation of coupling agent and substitution of NR with ENR in NR-silica composites improved their abrasion resistance. Abrasion resistance of the silica filled NR containing 2% coupling agent and of optimum NR/ENR blend are comparable. With increased concentration of coupling agent, a corresponding improvement in abrasion resistance was noted, but was still lower than that of NR-ISAF. With combination of accelerators, abrasion resistance of silica filled NR was found better than that of the same modified with DEG and that of ENR-silica.

4.4 Heat buildup and compression set

Variations in heat buildup and compression set values of NR-silica composites modified with varying proportions of ENR is given in Figure 6. For CBS cured systems, a reduction in heat buildup was noted with ENR incorporation, while an enhancement was observed for binary accelerators. With single accelerator system the interface modification of NR and silica by ENR might have reduced the accelerator adsorption by the silica particles. This may have contributed to a higher crosslink density and a corresponding lower heat buildup. While the enhancement in heat buildup with binary accelerated systems for the samples containing ENR, over that of the control, may be a consequence of the difference in moduli of the two.

Compression set also showed a similar behaviour as that of heat buildup; ie with ENR concentration a reduction with CBS cure and a rise for combination of accelerators. Silane modified NR-silica composites exhibited lower heat buildup and compression set for both the systems of cure (Tables 5 and 6). However, NR-ENR-silica and NR-Si 69-silica cured with binary accelerators, exhibited comparable heat buildup values as those of NR-ISAF.

4.5 Resilience and hardness

Changes in resilience and hardness of silica filled NR compounds containing varying proportions of ENR are given in Figure 7. A progressive reduction in resilience with increased concentration of ENR was observed. Compared to NR, a higher glass transition temperature and lower resilience are characteristics of ENR (3). It is likely that these characters might have been imparted to the silica filled blend, depending on its concentration in the blend.

With binary cure system, hardness of ENR modified silica composites are comparable with that of the Si 69 and DEG modified composites.

4.6 Ageing resistance

Ageing resistance data of a few selected samples viz. NR-ISAF, NR-Silica, NR-ENR-Silica, NR-Si 69-Silica and ENR-Silica are compared in Table 7. Retention of strength and modulus are comparable for all the samples, while clear differences can be drawn between samples on retention of tear strength. NR-ISAF and ENR-silica showed the least retention, while NR-silica and NR-ENR-Silica showed very good retention even better than that containing 8% coupling agent.

4.7 Scanning electron microscopy

Dannenberg has reported that coupling agents not only contributes to an improvement in filler-rubber bonding, but also facilitates for a better dispersion of the fillers in the rubber matrix (15). The SEM photographs of the samples modified with Si 69 and ENR are compared with that of the control in Figures 8.a, 8.b, 8c. and 8d. From the figures it can be observed that the filler agglomerates contained in the silica composites modified with Si 69 and ENR is smaller in size and less in number than that in the unmodified. This observation indicates that ENR in small quantities, is effective in controlling the size and distribution of silica fillers. This finding also explains some of the improvements observed in other properties.

5. Conclusions

1. Small quantities of NR substituted with ENR of NR-silica composites was found to have modified their properties, in a pattern similar to that of the conventional modifiers such as coupling agent or diethylene glycol.
2. With optimum concentration of ENR in the blend some of the important technological

properties such as cure characteristics, modulus, tensile strength and abrasion resistance were found improved over the silica filled NR.

3. Though ENR-silica composites exhibited higher modulus similar to that of NR-ISAF or NR-Si 69-silica, other critical properties were inferior.
4. For the ENR modified system, though a considerable improvement in properties was achieved over the control, further work is required for closer matching with that of the coupling agent.
5. NR-ENR-silica composite exhibited very good ageing resistance, better than that of ENR-silica and NR-ISAF.
6. Scanning electron microscopy showed that ENR could enhance the efficiency of filler dispersion of NR-silica composites.
7. Improved properties of NR-ENR-silica composites over NR-silica indicates that ENR has served as an interface modifier in the former.

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Table 1 - Formulation of mixes*

Ingredient	A	B	C	D	E	F	G	H	I
Natural rubber	100	95	90	85	80	-	100	100	100
Epoxidised natural rubber	-	5	10	15	20	100	-	-	-
Diethylene glycol	-	-	-	-	-	-	-	2	-
Si-69	-	-	-	-	-	-	4	-	-

* Compounds given in Table 1 also contain the ingredients given below

Zinc oxide 5, Stearic acid 2, Naphthenic oil 5, N-Cyclohexyl-2-benzthiazyl sulphenamide (CBS) 1.5, Sulphur 2, Ultrasil VN_3 50 (for A to H) and ISAF 50 (for I only).

Table 2. Formulation of mixes*

Ingredient	A'	B'	C'	D'	E'	F'	G'	H'	I'	J'	K'	L'	M'
Natural rubber	100	97.5	95	90	85	80	75	-	100	100	100	100	100
Epoxidised natural rubber	-	2.5	5	10	15	20	25	100	-	-	-	-	-
Si-69	-	-	-	-	-	-	-	-	1	2	3	4	-
Diethylene glycol	-	-	-	-	-	-	-	-	-	-	-	-	2

* Compounds given in Table 2 also contain the ingredients given below:
Zinc oxide 5, Stearic acid 2, Naphthenic oil 5, N-Cyclohexyl-2-benzythiazyl sulphenamide (CBS) 1.5, Diphenyl guanidine(DPG) 2, Sulphur 2, Ultrasil VN_3 50

Table 3. Cure Characteristics (Mixes A to I)

Property	A (100 NR)	B (5 ENR)	C (10 ENR)	D (15 ENR)	E (20 ENR)	F (100 ENR)	G (8 % Si69)	H (4% DEG)	I (ISAF)
Δ, Rheometric torque,dN.m (Max . torque - Min. torque)	19	36	41	39	39	67	56	45	71
Scorch time, 150 ° C (ts_2) - minutes	5.5	8	8	8.5	8.5	8	5	4	3
Optimum cure time(t_{90}), 150° C -minutes	34	20	15	17.5	18.5	16	15	12.5	6
Cure rate index ($100/t_{90}-t_2$)	3.5	8.3	11.7	11.1	10	12.5	10	12	36

Table4.Cure characteristics (Mixes A' to M')

Property	A' (100 NR)	B' (2..5 ENR)	C' (5 ENR)	D' (10 ENR)	E' (15 ENR)	F' (20 ENR)	G' (25 ENR)	H' (100 ENR)	I' (2% Si69)	J' (4% Si69)	K' (6% Si69)	L' (8% Si69)	M' (4% DEG)
Δ, Rheometric torque,dN.m (Max.torque - Min. torque)	84.5	82	82	80	80	76	77	87	80	84	85	92	81.5
Scorch time, 150° C (ts_2) - minutes	2.5	3	3	3	3	3	3	2.5	2	2	2.5	3	1.5
Optimum cure time(t_{90}) 150 ° C -minutes	5.5	6	6	6	6	6	6	7	4.5	4.7	7	7	4
Cure rate index ($100/t_{90}-t_2$)	33.3	33.3	33.3	33.3	33.3	33.3	33.3	22.2	40	37	20	25	40

Table 5 - Technological Properties (CBS cure)

Property	Mix/Identification					
	A (100 NR)	F (100 ENR)	G (8% Si-69)	H (4% DEG)	I (ISAF)	C (10 ENR)
Modulus (10% elongation) N/mm^2	0.95	1.02	0.95	0.8	0.85	0.95
Modulus (300% elongation) N/mm^2	5.0	15.0	10.6	4.8	16.1	8.7
Tensile Strength (N/mm^2)	16.8	19.6	25.5	21.0	24.3	22.6
Elongation at break (%)	560	340	560	630	420	535
Tear Strength (N/mm)	33	47	107	72	115	89
Abrasion loss (mm^3)	312	185	136	252	97	200
Heat buildup (°C)	91	33	25	79	31	83
Compression set (%)	41	17.5	25	43	25	37
Resiliense (%)	49	29	59	53	49	43
Hardness (Shore A)	68	66	63	63	64	63

Table 6 Technological properties (CBS- DPG cure)

Property	Mix Identification								
	A' (100 NR)	H' (100 ENR)	I' (2% Si-69)	J' (4% Si-69)	K' (6% Si-69)	L' (8% Si-69)	M' (4% DEG)	I (ISAF)	D' (10 ENR)
Modulus (10% elonga-tion) N/mm^2	0.9	1.1	0.95	1.1	1.2	1.16	0..9	0.85	1.0
Modulus (300% elongation) N/mm^2	6.1	12.5	12.5	16	16.2	15.1	5.1	16.1	8.5
Tensile strength (N/mm^2)	25	24.1	27	28	28.4	27.5	22.1	24.3	27.0
Elongation at break (%)	620	520	530	440	490	470	630	420	590
Tear strength (N/mm)	96	82	111	109	113	110	85	119	110
Abrasion loss (mm^3)	170	178	144	126	121	111	186	97	145
Heat buildup (°C)	23	40	22	20	21	24	30	31	30
Compression set (%)	31	44	23	23	23	28	31	25	32
Resilience (%)	54	36	61	63	62	58	57	49	49
Hardness (Shore A	67	75	65	66	67	72	71	64	72

Table 7. Comparison of ageing resistance

Property	Condition	Mix/Identification				
		I (NR-ISAF)	A' (NR-SILICA)	D' (NR-ENR-SILICA)	L' (NR-8% Si69-SILICA)	H' (ENR-SILICA)
Modulus (N/mm^2)	Control	16.1	6.1	8.5	15.1	13.7
	Aged*	16.3	7.0	9.3	14.1	17.0
Tensile strength (N/mm^2)	Control	24.3	25	27	27.5	24
	Aged*	18	19	21	20	21
Tear strength (N/mm)	Control	113	96	110	110	82
	Aged*	63	97	103	75	57

* Aged at 100°C, for 48 hours

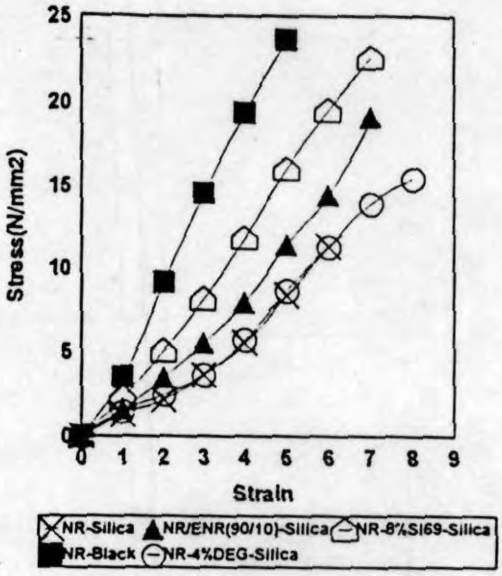


Fig.1 Comparison of stress- strain property
(CBS cure)

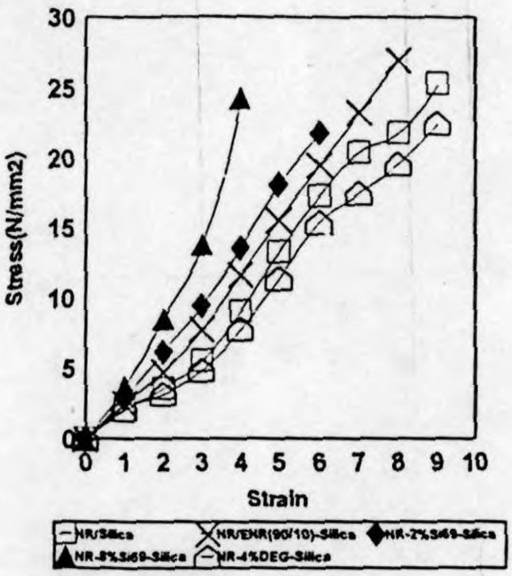


Fig.2 Comparison of stress- strain property
(CBS- DPG cure)

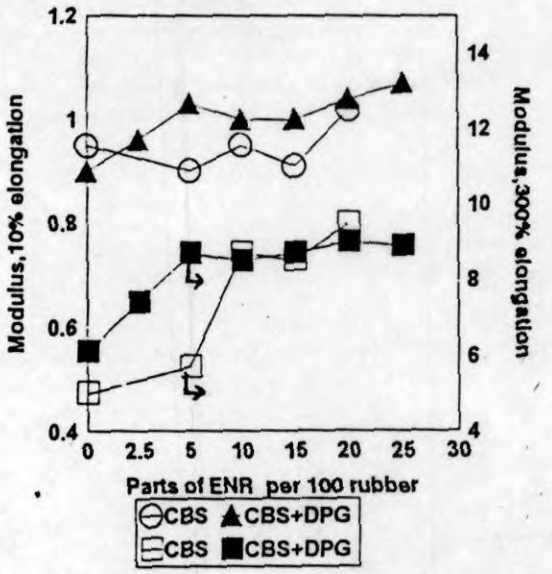


Fig.3 Variations in modulus of silica filled NR with ENR incorporation

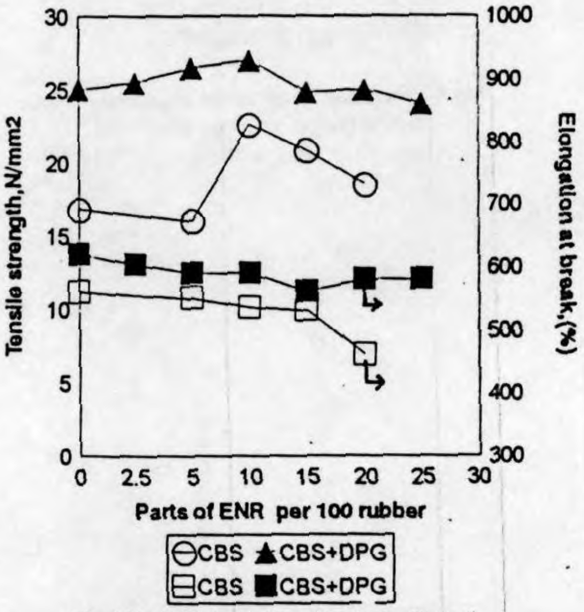


Fig.4 Variations in tensile strength and elongation at break of silica filled NR with ENR incorporation.

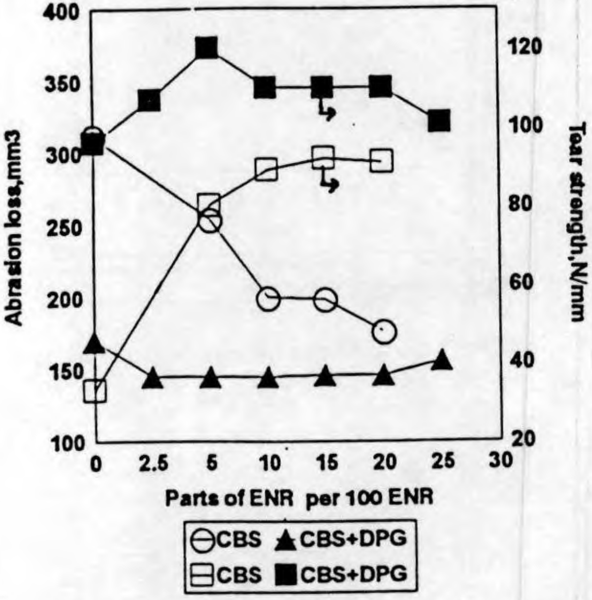


Fig.5 Variations in abrasion loss and tear strength of silica filled NR with ENR incorpration

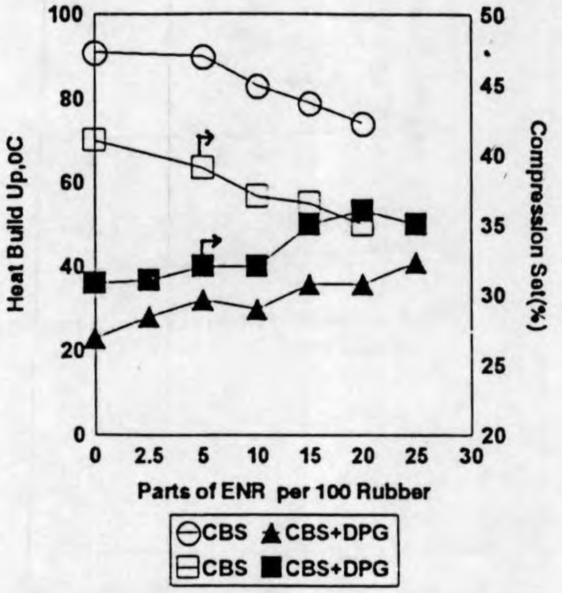


Fig.6 Variations in heat build up and compression set of silica filled NR with ENR incorporation

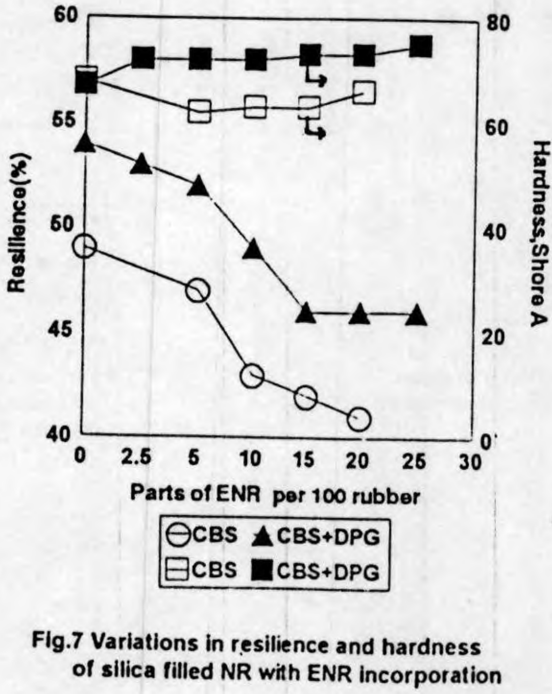
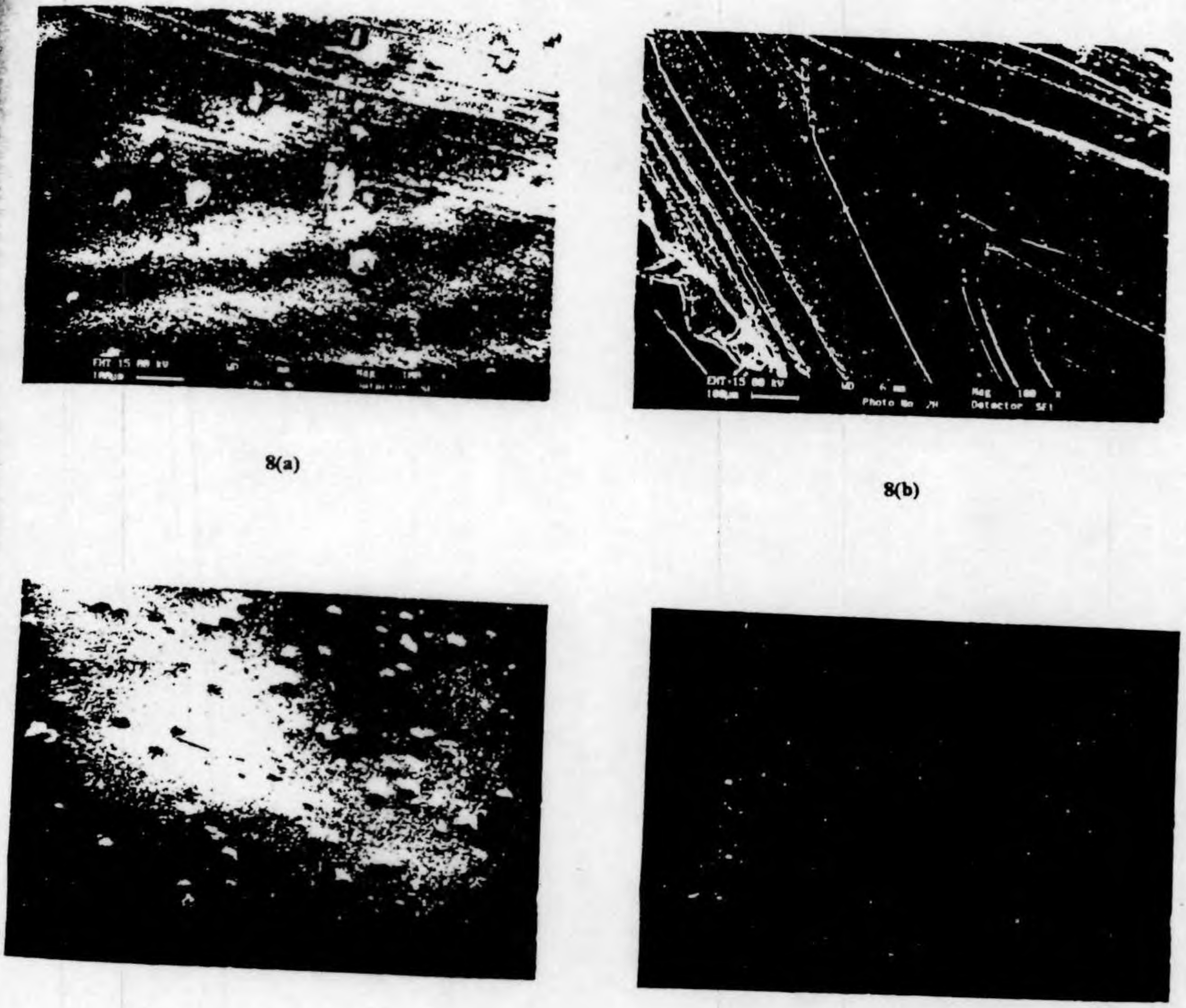


Fig.7 Variations in resilience and hardness of silica filled NR with ENR incorporation



8(a)

8(b)

8(c)

8(d)

Figure.8 SEM Photographs showing the silica dispersion of different samples (CBS-DPG cure) .
8(a) NR-Silica, 8(b) NR-8% Si 69-Silica,
8(c) NR/ENR(95/5)-Silica 8(d) NR/ENR(85/15)-Silica