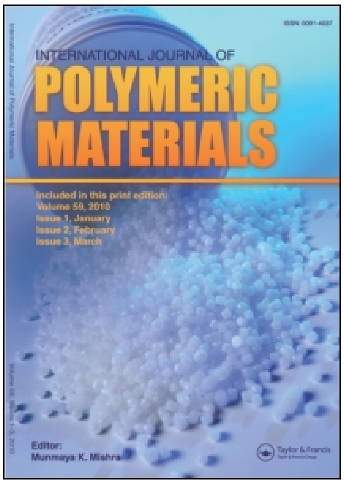

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Mechanical and Dielectric Properties of Styrene-butadiene Rubber Polyester Short-fiber Composites Part 1: Composites Loaded with Semirein Forcing Furnace Carbon Black (SRF)

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Mechanical and Dielectric Properties of Styrene-butadiene Rubber Polyester Short-fiber Composites

Part 1: Composites Loaded with Semirein Forcing Furnace Carbon Black (SRF)

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In a systematic study on the mechanical and dielectric properties of styrene-butadiene rubber (SBR) reinforced with polyester short fiber (PE), the effects were studied of adhesion system HRH (hydrated silica, resorcinol and hexamethylene-tetramine), fiber concentration, semireinforcing furnace carbon black (SRF) and thermal aging on these properties.

The permittivity ε' and the dielectric loss ε'' for the samples under test were determined in the frequency range 100 Hz to 10 MHz. It was found that the addition of the tricomponent adhesion system HRH to SBR-PE vulcanizates improves the mechanical and dielectric properties. Also, the increase of short fiber concentration in the SBR vulcanizates in presence of the adhesion system improves the mechanical properties. The addition of SRF carbon black facilitates the compounding of PE with SBR; moreover the tensile strength, Young's modulus and the permittivity were increased while the elongation at break was decreased.

The samples under test were subjected to thermal aging. The mechanical and dielectric properties were then reinvestigated and the data obtained interpreted.

Keywords: Styrene-butadiene rubber; Polyester short-fiber; Adhesion, Permittivity; Dielectric loss; Mechanical properties

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INTRODUCTION

Recently, short fibers attracted the attention of several researchers due to their good dispersion and good adhesion to rubber matrix [1, 2], as well as their advantages on the physico-mechanical and dielectric properties. The adhesion between many types of short fibers and most elastomers have been achieved by the discovery of the tricomponent system HRH (Hydrated silica, Resorcinol, Hexamethylene tetramine) [3–6]. In this case silica is believed to act as a controller for resin formation, as the reaction of resorcinol and hexamethylene tetramine helps in developing adhesion between rubber and fiber [5, 7].

Compounding of rubber with short, synthetic type fibers has been studied by O'Connor [8]. De and co-workers have reported their results on jute fiber reinforced natural and synthetic rubber. Satua and De [9] have studied short-silk fiber reinforced natural rubber composites. Murty has studied glass fiber reinforced natural rubber composites.

The effect of adhesion system HRH (hydrated silica, resorcinol and hexamethylene-tetramine) between styrene-butadiene rubber and polyester short fiber on the physico-mechanical and dielectric properties have been studied [10]. It was found that the presence of adhesion system together with polyester short fiber improves the physico-mechanical and dielectric properties.

The effect on the physico-mechanical and or dielectric properties [11–19] of aging of natural and synthetic rubber loaded with either black or white fillers have been studied. Few Investigations have been devoted to aging of SBR-PE fiber vulcanizates [10, 20–22].

The aim of this work is to point out the influence of the different constituents composing the HRH system, the polyester short-fiber concentration, the addition of SRF carbon black and the thermal aging on the mechanical and dielectric properties of styrene-butadiene rubber.

EXPERIMENTAL

The materials used in this study were:

1. Styrene-butadiene rubber (SBR) 1502 supplied by Esso Chemie.
2. Zinc oxide to activate the action of accelerator.

3. *N*-cyclohexyl-*z*-benzothiazole sulfonamide (CBS) acts as an accelerator to reduce the time required for cure.
4. Stearic acid acts as a softener to facilitate the dispersion of material added to rubber.
5. Naphthenic processing oil (sp.gr. 0.96 and viscosity at 100°C = 80 - 90 CP).
6. Colloidal Hydrated silica (Hisil), Resorcinol and Hexamethylenetetramine (HMTA) as tricomponent adhesive system (HRH).
7. Sulphur (SP. Gr. 2.04 - 2.06) essential vulcanizing agent.
8. Polyester short-fiber [64 mm] from Misr company silk, kafr El-Dawar Egypt.

The preparation of rubber vulcanizates were carried out according to ASTM method [23]. All ingredients were accurately weighed. Mixing was carried out on a laboratory controlled temperature two roll mill of the following dimensions: outside diameter 470 mm, working distance = 300 mm, speed of slow roll = 17 r.p.m and friction ratio 1 : 1.4. Care was taken to ensure fiber orientation in the mill direction [24].

Mechanical Measurements

A Mansanto oscillating disc rheometer model 100 was used for measuring the processing and curing characteristics of the rubber compounds [25, 26]. The measurements were carried out at 152 ± 1°C. The vulcanized sheets were cut into five individual dumbell-shaped specimens in both longitudinal and transversal directions by a steel die of constant width (0.4 cm). The minimum thickness of test specimens was determined by gauge calibrated in hundredth of millimeter.

The tensile, strength, elongation at break and Young's modulus were determined using an electronic Zwick testing machine (1425) from Germany. Tensile strength is defined as "The applied force per unit area of the original cross-sectional area at the moment of rupture of the specimen."

$$\text{Tensile strength} = K/TW(N/\text{mm}^2)$$

K = strength in Newton necessary to cause rupture, T = thickness in mm, W = width of the specimen in mm.

Ultimate elongation $E\%$ (elongation at break) is expressed as the percentage of the original length attained at rupture:

$$E\% = \left(\frac{L - Lo}{Lo}\right) \times 100$$

where L is the length of the specimen at the moment of rupture and Lo the original length.

In the common practice of rubber technology, the stress regarded for a given elongation is used to represent the material stiffness. This quantity is defined as the ratio of stress to strain. If this ratio is constant, the material is said to obey Hook's low, and the constant is called Young's modulus which is given by:

$$\frac{\text{Stress}}{\text{Strain}} = \frac{F/A}{\Delta L/L}\left(\frac{N}{\text{mm}^2}\right)$$

Dielectric Measurements

Measurements of the permittivity ε' and the dielectric loss ε'' for the different vulcanizates of SBR were carried out at the frequency range 100 Hz up to 10 MHz. A NF-Dekameter (DK 05) of the Schering bridge type (wissenschaftlich- Technische – werkstatten WTW Germany) was used for the frequency range 60 Hz to 100 KHz. A circuit magnification meter (Q-meter) type 1245 with an oscillator type TF 1246 from Marconi Instrument Ltd. (England) were used for frequencies between 50 KHz and 10 MHz.

For dielectric measurements, a guard-ring capacitor type NFM $5/T$ from (WTW) was used. The Thickness and capacitance of the sample can be measured in a single compression. The cell temperature can be controlled using an ultrathermostat.

The cell was calibrated using plates of known permittivity values, such as air, trolitul, and glass with different thicknesses ranging from 2 up to 7 mm. The errors in ε' and ε'' amounts to $\pm 2\%$ and $\pm 5\%$, respectively [10]. The samples were prepared in the form of discs 50 mm in diameter and 3 mm thick.

RESULTS AND DISCUSSION

To study the effect of adding the different constituents composing the tricomponent adhesion system HRH on the mechanical and dielectric properties of SBR, Ten rubber formulations were prepared as given in Table I.

Mechanical Properties

The rheometric characteristics of the mixes were measured at $152 \pm 1°C$ and listed in Table I. The results for samples 3 and 5 are not included in the table, as they were uncured because resorcinol has a retarding effect on the vulcanizates, due to its oxidizing (acidic) nature which prevents the vulcanization in the absence of HMTA. From Table I, it is clear that the addition of one or two or all of the ingredients of HRH system decreases the cure time (t_{c90}) while the maximum torque (M_H) and the cure rate index (CRI) are increased, with respect to sample 1.

The tensile strength, elongation at break and Young's modulus of the mixes vulcanized at the same temperature, were measured and the data obtained are given in Table I. From these data, it is clear that the addition of Hisil (sample 2) increases both the tensile strength and elongation at break, while they are decreased by the addition of HMTA (sample 4). Adding polyester (PE) fibers (sample 9) decreases the tensile strength, whereas a marked increase in tensile strength and Young's modulus is obtained by addition of the tricomponent adhesion system HRH together with polyester fiber (sample 10). This indicates that the presence of adhesion system between fiber and SBR is necessary to obtain high tensile strength and low elongation, which is important for most rubber articles reinforced by textile.

The rubber samples were subjected to thermal aging at $90 \pm 1°C$ for two days. The mechanical properties were remeasured after aging and the results are listed in Table I. Comparing the results obtained before and after aging, it is clear that after aging the tensile strength and Young's modulus for sample 10 which contains the tricomponent adhesion system HRH are increased whereas the elongation at break is highly decreased. This can be attributed to further curing of the

TABLE I Rubber formulations containing different ingredients of the tricomponent adhesion system (HRH) in (Phr), the rheometric characteristics and mechanical properties (Phr): Parts per hundred parts rubber by weight

Sample / *Ingredients in phr*	1	2	3	4	5	6	7	8	9	10
SBR 1502	100	100	100	100	100	100	100	100	100	100
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Zinc oxide	5	5	5	5	5	5	5	5	5	5
SRF Carbon black	10	10	10	10	10	10	10	10	10	10
Processing oil	1	1	1	1	1	1	1	1	1	1
Hydrated silica (Hisil)	–	5	–	–	5	5	–	5	–	5
Hexamethylene-tetramine (HMTA)	–	–	–	3.2	–	3.2	3.2	3.2	–	3.2
Resorcinol	–	–	5	–	5	–	5	5	–	5
CBS	1	1	1	1	1	1	1	1	1	1
Sulphur	2	2	2	2	2	2	2	2	2	2
Polyester fiber	–	–	–	–	–	–	–	–	20	20
Rheometric characteristics at $152 \pm 1°C$										
Minimum torque (M_L) (dNm)	7.0	8.0		6.0		6.0	6.5	8.0	7.5	8.0
Maximum torque (M_H) (dNm)	35.5	38		55.5		58.5	47.0	51.0	51.0	55.5
Scorch time t_{s2} (min)	15.5	13		8.5		9.0	2.5	3.0	13.0	3.0
Optimum cure time (t_{c90}) (min)	52.0	43		16.5		18.0	20.0	25.0	33.0	21.5
Cure rate index (CRI) (min^{-1})	2.74	3.35		12.5		11.11	5.56	4.55	5.0	5.4
Mechanical properties										
Tensile Strength (N/mm^2)	2.73	5.66		2.42		3.07	7.57	8.62	1.89	4.51
Elongation %	594.6	785.4		509.6		514.5	681.5	566.6	364.2	167.6
Young's modulus (N/mm^2)	0.64	0.84		1.61		0.89	1.22	1.05	8.58	15.3
Mechanical properties after 2-days thermal aging ($90 \pm 1°C$)										
Tensile Strength (N/mm^2)	2.77	5.66		3.28		3.50	9.23	8.15	1.85	5.46
Elongation %	532.7	608.2		438.4		444.6	354.0	497.9	94.84	59.7
Young's modulus (N/mm^2)	0.72	1.08		2.53		1.18	2.86	1.13	32.15	52.0

*Samples 3 and 5 are uncured.

resin which leads to further formation of cross-linked resin which balance the degradation occurs during aging.

Dielectric Properties

The permittivity ε' and the dielectric loss ε'' of the samples under test were measured in the frequency range from 100 Hz to 50 KHz at 30°C. and illustrated graphically in Figure 1. Figure 1a, shows the dependence of the permittivity ε' on frequency for the investigated samples. From this figure it is clear that ε' increases at the whole frequency range by the addition of either Hisil (sample 2) or HMTA (sample 4) or both (sample 6) with respect to sample 1. Also the addition of HRH system (sample 8) increases ε'. On the other hand, the addition of 20 phr polyester (PE) fiber (sample 9) leads to a decrease in ε' but it is higher than sample 1, while the addition of 20 phr polyester fiber together with HRH system (sample 10) increases ε' at the whole frequency range.

Figure 1b shows the frequency dependence of the dielectric loss ε'' for the samples under investigations. It is clear from this figure that the presence of either Hisil (sample 2), HMTA (sample 4) or the adhesion system HRH (sample 8) leads to a slight increase in ε'' with respect to sample 1. While the addition of HMTA and Hisil (sample 6), or polyester (PE) fiber (samples 9 and 10) leads to a decrease in ε''.

The rubber samples were subjected to thermal aging for 2-days at temperature 90 ± 1°C. The permittivity ε' and the dielectric loss ε'' were remeasured after aging and the data obtained are shown in Figure 2. It is found that the dielectric loss for sample 6 is increased while no significant change in ε' and ε'' for the other samples is noticed.

From this study it is concluded that the addition of tricomponent adhesion system HRH to SBR-PE fiber vulcanizates improves the mechanical and dielectric properties.

Effect of Polyester Short-fiber Concentration

To study the effect of polyester short fiber concentration on SBR, seven rubber formulations were prepared as given in Table II.

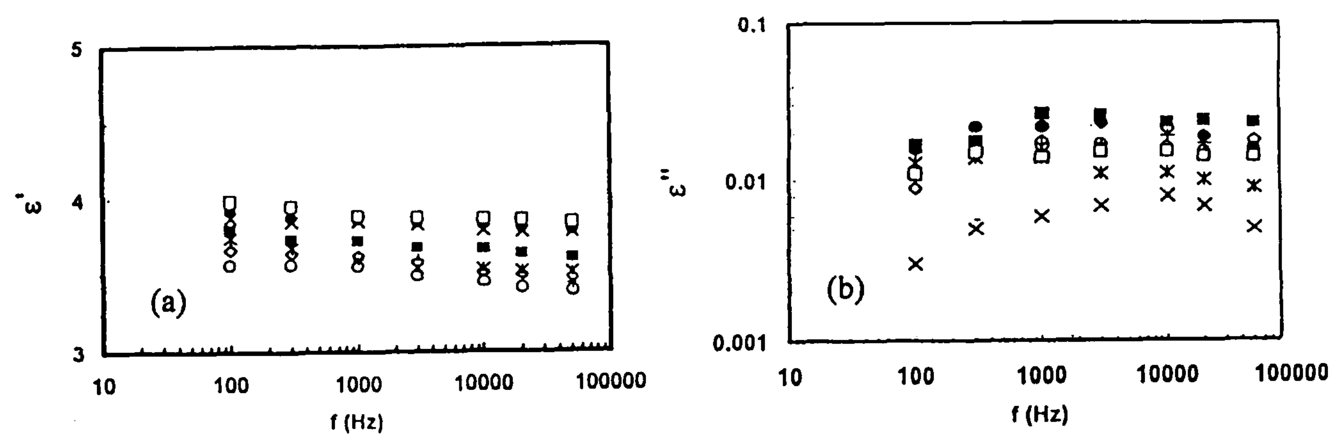
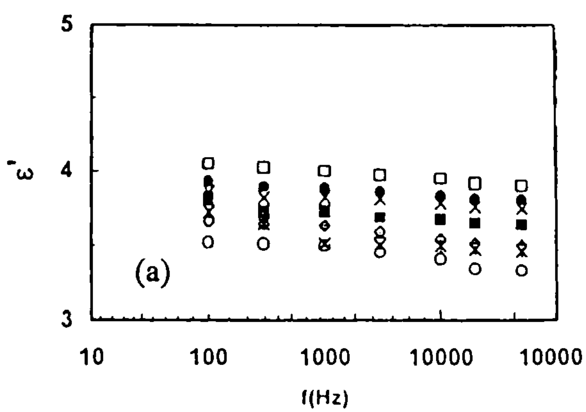


FIGURE 1 The frequency dependence at 30°C of the permittivity ε′ (a) and the dielectric loss ε″ (b) for the different formulations of SBR vulcanizates: ○1, ■ 2, ◇ 4, × 6, ∗ 7, ● 8, + 9, □ 10.



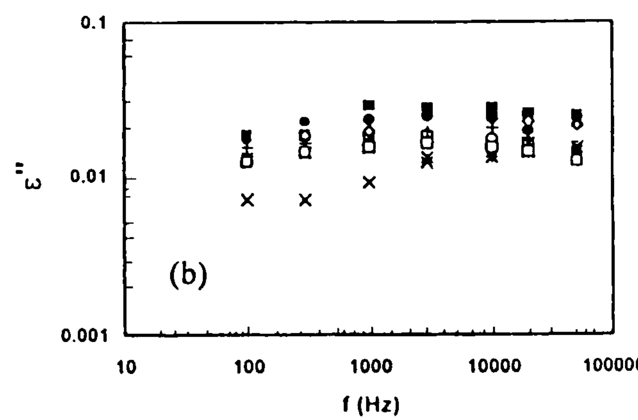


FIGURE 2 The frequency dependence at 30°C of the permittivity ε' (a) and the dielectric loss ε'' (b) for the different formulations of SBR vulcanizates after being thermally aged at 90°C for 2-days. Same notations as in Figure 1.

TABLE II Rubber formulations containing different concentrations of polyester-short fiber, the rheometric characteristics and the mechanical properties

Sample / Ingredients		PE fiber content in (phr)							
		Cont.	0	5	7.5	10	12.5	15	20
SBR 1502		100	100	100	100	100	100	100	100
Stearic acid		2	2	2	2	2	2	2	2
Zinc oxide		5	5	5	5	5	5	5	5
Processing oil		–	–	–	–	–	–	–	–
Hydrated silica (Hisil)		–	5	5	5	5	5	5	5
Hexamethylene-tetramine (HMT)		–	3.2	3.2	3.2	3.2	3.2	3.2	3.2
Resorcinol		–	5	5	5	5	5	5	5
CBS		1	1	1	1	1	1	1	1
Sulphur		2	2	2	2	2	2	2	2
Polyester fiber		–	–	5	7.5	10	12.5	15	20
Rheometric characteristics at 152 ± 1°C									
Minimum torque (M_L) (dNm)		8.5	10.0	10.0	9.3	10.8	9.0	9.5	10.5
Maximum torque (M_H) (dNm)		60.0	73.0	74.0	79.0	84.5	80.0	82.0	98.0
Scorch time t_{s2} (min)		7.5	2.3	2.3	1.8	2.3	2.5	2.5	2.5
Optimum cure time (t_{c90}) (min)		18.5	16.5	18.8	19.0	19.0	20.0	20.8	20.5
Cure rate index (CRI) (min^{-1})		9.1	6.9	6.1	5.8	6.0	5.7	5.8	5.6
Mechanical properties in the longitudinal (L) and transversal (T) directions									
Tensile Strength	L	3.87	3.93	3.95	4.08	4.16	4.28	4.67	5.00
(N/mm^2)	T	3.87	3.95	3.92	3.95	4.12	4.26	4.53	4.46
Elongation	L	543.00	560.00	510.00	480.00	420.98	378.00	306.75	186.21
%	T	552.00	563.00	516.00	498.11	424.49	389.00	316.78	202.23
Young's modulus	L	1.79	2.49	3.96	4.78	4.96	5.08	5.95	7.23
(N/mm^2)	T	1.28	2.37	3.75	3.99	4.48	4.95	5.46	6.28

*Cont. (Control Sample) Without adhesion system.

Mechanical Properties

The rheometric characteristics of the investigated samples are given in Table II. The given data indicate that the increase of PE-fiber concentration in SBR mixtures containing the HRH system increase both the maximum torque (M_H) and the optimum cure time (t_{c90}) while it decreases the cure rate index (CRI).

The tensile strength, elongation at break and Young's modulus were measured for the rubber vulcanizates in both the longitudinal (L) and transversal (T) directions and the data are given in Table II. From the obtained results it is clear that both the tensile strength and Young's modulus are increased with the increase of fiber concentration, while the elongation at break decreases in both directions. This indicates that the mechanical properties are improved by the increase of fiber concentration. Also it can be noticed that the tensile strength and Young's modulus in the longitudinal direction are higher than in the transversal direction, and the elongation at break in the longitudinal directions is lower than in the transversal one. This indicates that polyester fibers were oriented to some extent in the longitudinal direction in the rubber matrix which may lead to the increase in the stiffness of the compound.

In compounding it was difficult to increase the PE fiber concentration more than 20 phr. To increase PE-fiber content in the SBR mixtures, a trial was made by adding another reinforcing material. 20 phr Semi-Reinforcing Furnace black (SRF) was added to the SBR vulcanizates, so mixtures of (SBR-PE)- SRF carbon black containing up to 30 phr PE fiber could be obtained as given in Table III. The rheometric characteristics and the mechanical properties for the different concentrations of PE short fiber in the presence of 20 phr SRF carbon black were measured and the data obtained are given in Table III. From Table III, it is clear that the maximum torque (M_H), and the cure rate index (CRI) are increased while the optimum cure time (t_{c90}) is decreased. Also, Table III shows that after the addition of 20 phr SRF, the tensile strength and Young's modulus were increased, while the elongation at break was decreased by increasing the fiber content. This could be attributed to the reinforcement action of SRF carbon black which may lead to the increase in the stiffness of these samples [27].

TABLE III Rubber formulations containing different concentration of the polyester-short fiber after addition of 20 phr SRF carbon black, the rheometric characteristics and the mechanical properties

Ingredients \ *Sample*		*PE fiber content in (phr)* 0	5	10	15	20	25	30
SBR 1502		100	100	100	100	100	100	100
Stearic acid		2	2	2	2	2	2	2
Zinc oxide		5	5	5	5	5	5	5
SRF Carbon black		20	20	20	20	20	20	20
Processing oil		2	2	2	2	2	2	2
Hydrated silica (Hisil)		5	5	5	5	5	5	5
Hexamethylene-tetramine (HMT)		3.2	3.2	3.2	3.2	3.2	3.2	3.2
Resorcinol		5	5	5	5	5	5	5
CBS		1	1	1	1	1	1	1
Sulphur		2	2	2	2	2	2	2
Rheometric characteristics at 152 ± 1°C								
Minimum torque (M_L) (dNm)		6.0	5.0	5.0	5.0	5.0	4.0	5.0
Maximum torque (M_H) (dNm)		59.8	62.0	62.0	74.5	67.5	78.5	80.0
Scorch time t_{s2} (min)		4.1	4.0	4.0	3.5	4.0	3.3	3.5
Optimum cure time (t_{c90}) (min)		31.0	29.0	25.0	27.5	30.8	30.5	25.5
Cure rate index (CRI) (min^{-1})		3.7	4.0	4.8	4.2	3.7	3.7	4.5
Mechanical properties in the longitudinal (*L*) and transversal (*T*) directions								
Tensile Strength	*L*	3.96	5.07	5.26	5.68	6.69	7.29	8.89
(N/mm^2)	*T*	3.96	5.12	5.14	5.39	5.58	6.23	7.89
Elongation	*L*	535.56	450.67	417.00	300.09	170.33	166.00	143.27
%	*T*	537.94	469.00	440.00	315.24	193.26	185.26	159.26
Young's modulus	*L*	2.00	3.95	4.66	6.25	7.98	8.28	8.99
(N/mm^2)	*T*	1.95	3.75	4.60	6.00	6.24	7.26	7.85

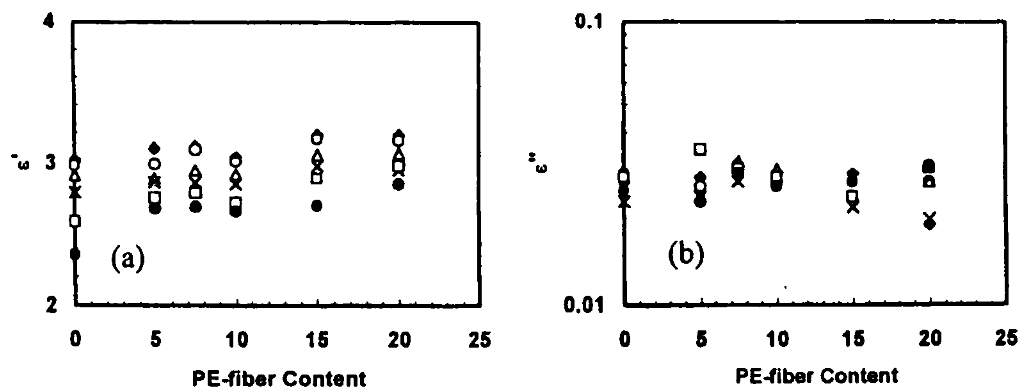


FIGURE 3 The permittivity ε' (a) and the dielectric loss ε'' (b) as a function of fiber concentrations for SBR vulcanizates at different frequencies, ◇ 0.1 KHz, ○ 1 KHz, △ 10 KHz, × 100 KHz, □ 1000 KHz, ● 10000 KHz.

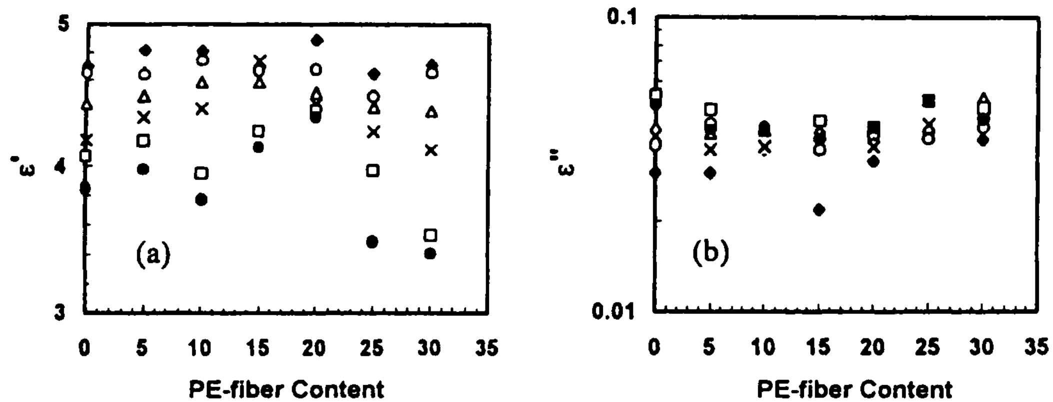


FIGURE 4 The permittivity ε' (a) and the dielectric loss ε'' (b) as a function of PE fiber content for SBR vulcanizates at different frequencies, after addition of 20 phr SRF carbon black. Same notations as in Figure 3.

Dielectric Properties

The permittivity ε' and the dielectric loss ε'' for the samples loaded with different PE-fiber concentrations in the SBR were measured at a frequency range from 100 Hz up to 10 MHz at 30°C. Their dependence on fiber concentration at different frequencies is illustrated graphically in Figure 3. From this figure it is clear that a slight increase in ε' is noticed with the increase of PE-fiber content while ε'' is about the same. Adding 20 phr SRF carbon black to SBR-PE fiber mixtures, their dependence on PE-fiber concentrations at different frequencies is illustrated graphically in Figure 4. From Figure 4. It is clear that addition of 20 phr SRF carbon black leads to an increase in ε' while ε'' is slightly increased for all samples under test. Figures 3a and 4a shows that ε' decrease with the increase of frequency at all concentrations, while ε'' is about the same in Figures 3b and 4b.

From this study it could be concluded that the mechanical as well as the dielectric properties of SBR-PE short fiber containing an adhesion system HRH were improved either by the increase of PE-short fiber concentration or addition of another reinforcing material. Moreover, the addition of another reinforcing material facilitates the compounding of polyester (PE) fiber with SBR.

CONCLUSIONS

1. Addition of the tricomponent adhesion system HRH to SBR-PE Short fiber vulcanizates improves the mechanical as well as the dielectric properties.
2. The presence of the tricomponent adhesion system HRH between PE short fiber and SBR helps the composite to resist aging, as the tensile strength and Young's modulus were increased and elongation at break was decreased after aging.
3. The increase of PE short fiber concentration in SBR leads to the improvement of the mechanical and dielectric properties.
4. The addition of another reinforcing agent like SRF to the SBR-PE fiber vulcanizates, facilitates the compounding. So, more PE fiber content could be added to the vulcanizates. Moreover the tensile strength and Young's modulus were increased and elongation at

break was decreased. Also the permittivity was increased but the dielectric loss was only slightly increased especially at the high frequency region.

5. The tensile strength and Young's modulus in the longitudinal direction were higher than in the transversal direction, and the elongation at break in the longitudinal direction was lower than in the transversal one.
6. The permittivity ε' decreases with the increase of frequency for all concentration either for carbon free samples or loaded with carbon whereas the dielectric loss slightly changed.

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