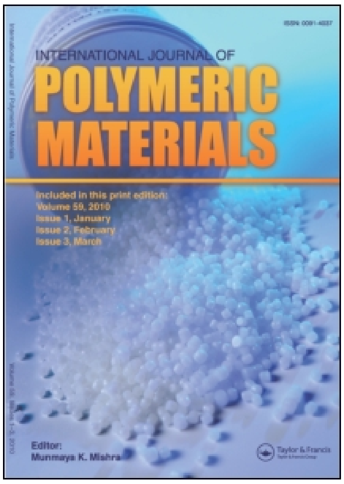

International Journal of Polymeric Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

Mechanical and dielectric properties of styrene-butadiene rubber polyester short-fiber composites, part III: Composites loaded with fast extrusion furnace carbon black

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Online publication date: 27 October 2010

To cite this Article Bishai, A. M. , Ward, A. A. M. , Ghoneim, A. M. and Younan, A. F.(2003) 'Mechanical and dielectric properties of styrene-butadiene rubber polyester short-fiber composites, part III: Composites loaded with fast extrusion furnace carbon black', International Journal of Polymeric Materials, 52: 1, 31 — 48

To link to this Article: DOI: 10.1080/714975855
URL: http://dx.doi.org/10.1080/714975855

International Journal of Polymeric Materials, 52:31–48, 2003

0091-4037/03 $12.00 + .00
DOI: 10.1080/00914030390119172

MECHANICAL AND DIELECTRIC PROPERTIES OF STYRENE-BUTADIENE RUBBER POLYESTER SHORT-FIBER COMPOSITES, PART III: COMPOSITES LOADED WITH FAST EXTRUSION FURNACE CARBON BLACK

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The present article aims to study the mechanical and dielectric properties of styrene butadiene rubber (SBR) reinforced with polyester (PE) short-fiber loaded with fast extrusion furnace carbon black (FEF) in increasing quantities. The effect of carbon particle size on the mentioned properties is also reported.

From the results obtained in this article and others, it was found that, the tensile strength and Young's modulus of FEF and HAF carbon black increase with the increase of carbon content whereas elongation at break was decreased. The retaining ability of the tensile strength and Young's modulus of the composites loaded with either FEF or HAF carbon black has been increased with the increase of aging time and decreased by increasing carbon content in the vulcanizates. At certain concentration of carbon black in the rubber matrix, there was an abrupt increase in the dielectric permittively ε' *and the dielectric loss* ε''*. The concentration at which* ε' *and* ε'' *shows a sharp increase is in line with the increase of carbon black particle size.*

The mechanical and dielectric properties for vulcanizates loaded with smaller carbon black particle size are higher than those loaded with larger ones. The dielectric permittivity ε' *has been increased by increasing the temperature whereas the dielectric loss was decreased. The results are discussed and Interpreted.*

Keywords: mechanical properties, styrene-butadiene rubber, polyester short-fiber, permittivity, dielectric loss, relaxation time

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INTRODUCTION

Fillers play an important role in improving the physical properties of elastomers. Filling rubber with various fillers is an easy way to obtain a wide spectrum of applications. The physical properties of filled rubber can be easily varied by changing the type, the quantity and mixture composition of the filler.

Fillers were used in order to extend and cheapen the compounds, since it was found that in butyl rubber quite a bit of fillers could be added without detracting too much from the final vulcanizate properties [1]. Fillers are either conductive or non conductive. Conductive fillers include carbon black and were discovered by Mote [2] in 1904. The main reason for the predominant use of fillers in elastomers, particularly carbon black, is the reinforcement they impart to the vulcanizates [2–5].

A reinforcing filler improves the mechanical properties of the final vulcanizates. This is very much dependent on the particle size of the filler; small particles have a much greater effect than coarse ones. Particle size is directly related to the reciprocal of the surface area per gram of filler, thus the effect of smaller particles actually reflect the greater extent of interface between polymer and solid material.

This work details results of the mechanical and dielectric properties already given in a previous paper [6]. It deals with investigations concerning the effect of filling SBR-PE composites with another kind of carbon black, fast extruding furnace (FEF), on the mentioned properties. Also, to point out the effect of carbon black particle size in rubber material on the mechanical and dielectric properties.

EXPERIMENTAL

The Material Used in This Study

(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) Sulfur (SP. Gr. 2.04–2.06) essential vulcanizing agent.
(8) Polyester short-fiber [64 mm] from Misr company silk, kafr El-Dawar, Egypt.
(9) Fast Extruding Furnace (FEF) carbon black N 550.

Sample Preparation

The preparation of rubber vulcaizates were carried out according to ASTM method [7]. 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 [8].

Mechanical Measurements

The apparatus used for measuring the processing and curing characteristics of the rubber compounds have been described in a previous paper [9]. The measurements were carried out at $150 \pm 1°C$. The vulcanized sheets were cut into five individual dumbell-shaped specimens in longitudinal and transversal directions by steel die of constant width (0.4 cm). The minimum thickness of test specimens was determined by gauge calibrated in hundredths of millimeter.

Tensile strength, elongation at break and Young's modulus of the samples were determined as mentioned before [9].

Dielectric Measurements

Measurements of the permittivity ε' and the dielectric loss ε'' for the samples under investigation were carried out as described in the previous paper [9]. The errors in ε' and ε'' amount to $\pm 2\%$ and $\pm 5\%$ respectively.

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 FEF carbon black to SBR-PE fiber composites on the mechanical and dielectric properties, samples loaded with different concentrations up to 70 phr FEF black were prepared as given in Table 1.

Mechanical Properties

The rheometric characteristics are given in Table 2. From this table it is clear that the maximum torque (M_H) and the optimum cure (t_{C90}) decrease with the initial loading of FEF, then they increase with the increase of FEF carbon concentration in SBR-PE vulcanizates. While the reverse is true for the cure rate index CRI.

The mechanical properties were measured for the rubber vulcanizates in the longitudinal and transversal directions. The data obtained are given in Table 3 and illustrated graphically in Figure 1. From this figure it is clear that both the tensile strength and Young's modulus increase with the

TABLE 1 Rubber formulations containing different concentrations of FEF carbon black

Ingredients	*phr*
SBR 1502	100.00
Stearic acid	2.00
Zinc Oxide	5.00
Processing oil	3.00
Hydrated silica	5.00
Hexamethylene-Tetramine	3.20
Resorcinol	5.00
CBS	1.00
Sulfur	2.00
Polyester fiber	20.00
FEF carbon black	0–70

TABLE 2 The rheometric characteristics at (152 + 1°C) of SBR-PE fiber vulcanizates containing different FEF carbon black contents

Sample / *Property*	*FEF carbon black contents (phr)*							
	0	10	20	30	40	50	60	70
Minimum torque (M_L) (dNm)	77.00	62.00	61.20	64.80	65.60	66.00	71.20	76.70
Maximum torque (M_H) (dNm)	7.00	4.30	5.00	5.20	5.50	5.95	6.31	6.51
Scorch time (t_{52}) (min)	3.60	3.50	3.75	3.25	3.26	3.27	3.21	2.96
Optimum cure time (t_{C90}) (min)	24.00	22.00	22.50	23.00	23.80	24.50	24.50	25.00
Cure rate index (CRI) $(min)^{-1}$	4.90	5.41	5.33	5.06	4.87	4.71	4.70	4.54

increase of FEF carbon content, while elongation at break is decreased in both directions indicating high stress at low extension which is very important in most rubber articles reinforced by carbon black. The tensile strength and Young's modules in the longitudinal direction (L), are higher than in the transversal (T) one, while an opposite trend is observed for elongation at break. This indicates rather well oriented fibers in rubber matrix. It is noticed also that the values of the tensile strength, elongation at break and Young's modulus for samples loaded with FEF carbon black are lower than the corresponding ones loaded with HAF carbon black [6]. This could be attributed to the smaller surface area of the FEF carbon black particles in contact with the elastomer as the particle size of FEF is larger than HAF carbon black [10].

Effect of Aging on the Mechanical Properties

Thermal Aging

The rubber vulcanizates were thermally aged at $90 \pm 1°C$ using the oven method [11], for different successive periods up to 7-days. The tensile

TABLE 3 The mechanical properties of (SBR-PE)/FEF vulcanizates in the longitudinal (*L*) and transversal (*T*) directions

Property	*Sample*	*FEF carbon black contents in (phr)*							
		0	10	20	30	40	50	60	70
(a) Before aging									
Tensile Strength	*L*	4.95	5.95	6.86	8.65	11.96	13.26	15.29	16.25
(N/mm^2)	*T*	4.45	5.46	6.21	7.45	9.36	11.26	12.47	14.00
Elongation	*L*	184	153	137	129	113	102	89	79
At break %	*T*	200	171	155	137	117	107	94	81
Young's modulus	*L*	7.23	11.56	16.51	19.87	24.51	27.96	29.98	32.68
(N/mm^2)	*T*	6.21	9.26	13.90	17.50	21.21	24.90	27.60	29.50
(b) After 7 days thermal aging at 90 ± 1°C									
Tensile Strength	*L*	5.73	7.11	8.15	10.21	13.61	14.5	16.15	16.68
(N/mm^2)	*T*	4.68	6.89	7.53	8.58	10.21	12.41	13.34	14.24
Elongation	*L*	100	95	78	75	61	49	30	20
At break %	*T*	134	87	69	56	38	30	20	18
Young's modulus	*L*	9.57	18.72	24.85	26.87	29.2	32.56	31.11	34.89
(N/mm^2)	*T*	6.53	17.5	20.50	26.11	25.89	30	31.27	32.11
(c) After 2 years natural aging									
Tensile Strength	*L*	5.00	6.01	6.95	8.97	12.05	13.67	15.89	16.46
(N/mm^2)	*T*	4.46	5.55	6.23	7.75	10.21	11.97	12.87	14.28
Elongation	*L*	186.21	154.00	139.00	131.26	114.00	105.21	92.00	80.21
At break %	*T*	202.23	171.00	156.00	136.27	118.00	108.95	96.90	86.95
Young's modulus	*L*	7.31	11.67	16.97	20.31	25.00	28.21	30.26	33.00
(N/mm^2)	*T*	6.28	9.59	14.00	17.89	21.87	25.00	27.89	30.35

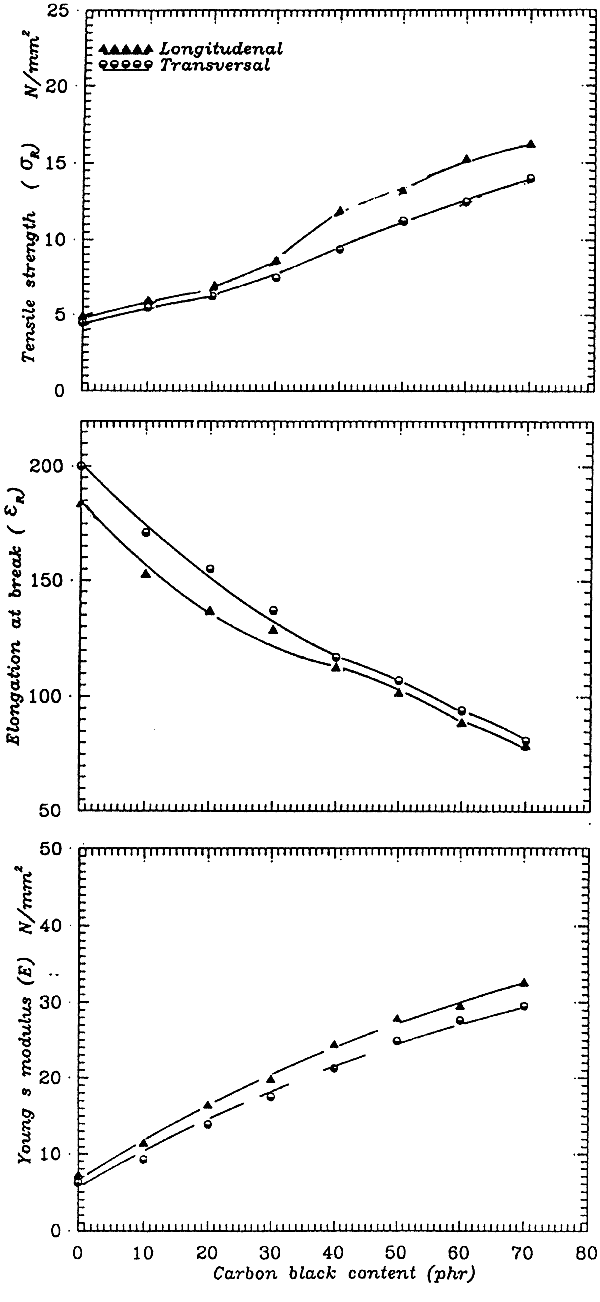


FIGURE 1 The dependence of the mechanical properties (Long-Trans) for SBR-PE short-fiber vilcanizates on FEF carbon black content.

strength, elongation at break and Young's modulus were remeasured after aging in the longitudinal and transversal directions. The data obtained after 7-days thermal aging are given in Table 3b. It is clear that the tensile strength and Young's modulus increased while elongation at break has decreased.

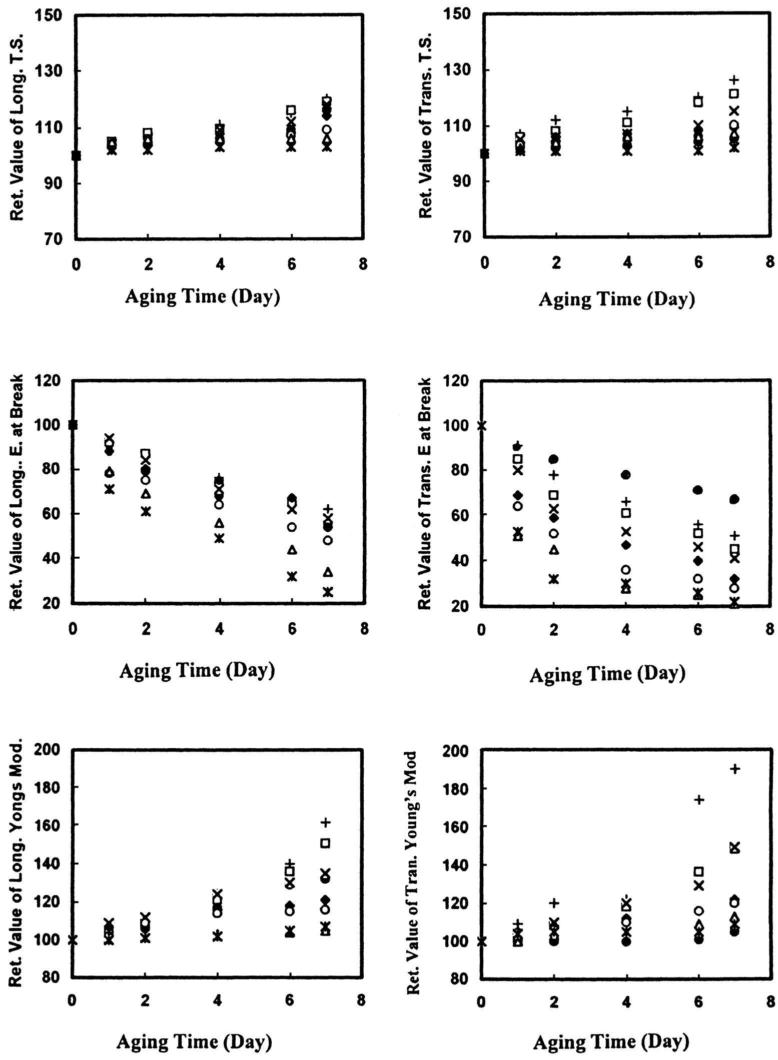


FIGURE 2 The dependence of the retained values of the tensile strength (T.S.) elongation at break (*E*) and Young's modulus on aging time for SBR-PE short-fiber vulcanizates loaded with different concentrations of FEF carbon black – 0 phr (●), 10 phr (+), 20 phr (□), 30 phr (×), 40 phr (◆), 50 phr (○), 60 phr (∇), and 70 phr (*).

It can be noticed that after thermal aging the elongation at break in the transversal direction is lower than in the longitudinal one which is the reverse before aging. This could be due to further break down of the carbon black aggregates. This behavior was seen also for SBR-PE vulcanizates loaded with HAF carbon black (6).

The retained values of the tensile strength elongation at break and Young's modulus in the longitudinal and transversal directions were calculated as before [6]. The dependence of these retained values on the aging time in both directions is shown in Figure 2. It is clear that the retained values of the tensile strength and Young's modulus have increased with the increase of aging time and decreased with increased carbon content, whereas the retained values of elongation at break have decreased with aging time and carbon content.

It is of interest to notice that the retained values of Young's modulus for sample loaded with 10 phr FEF increased up to 162% and 190% in the longitudinal and transversal direction respectively, which is about the same values as in case of HAF carbon black (6).

Natural Aging

The previously investigated samples were exposed to normal weathering circumstances for about two years. The mechanical properties of naturally aged samples were remeasured and the data obtained are given in Table 3c.

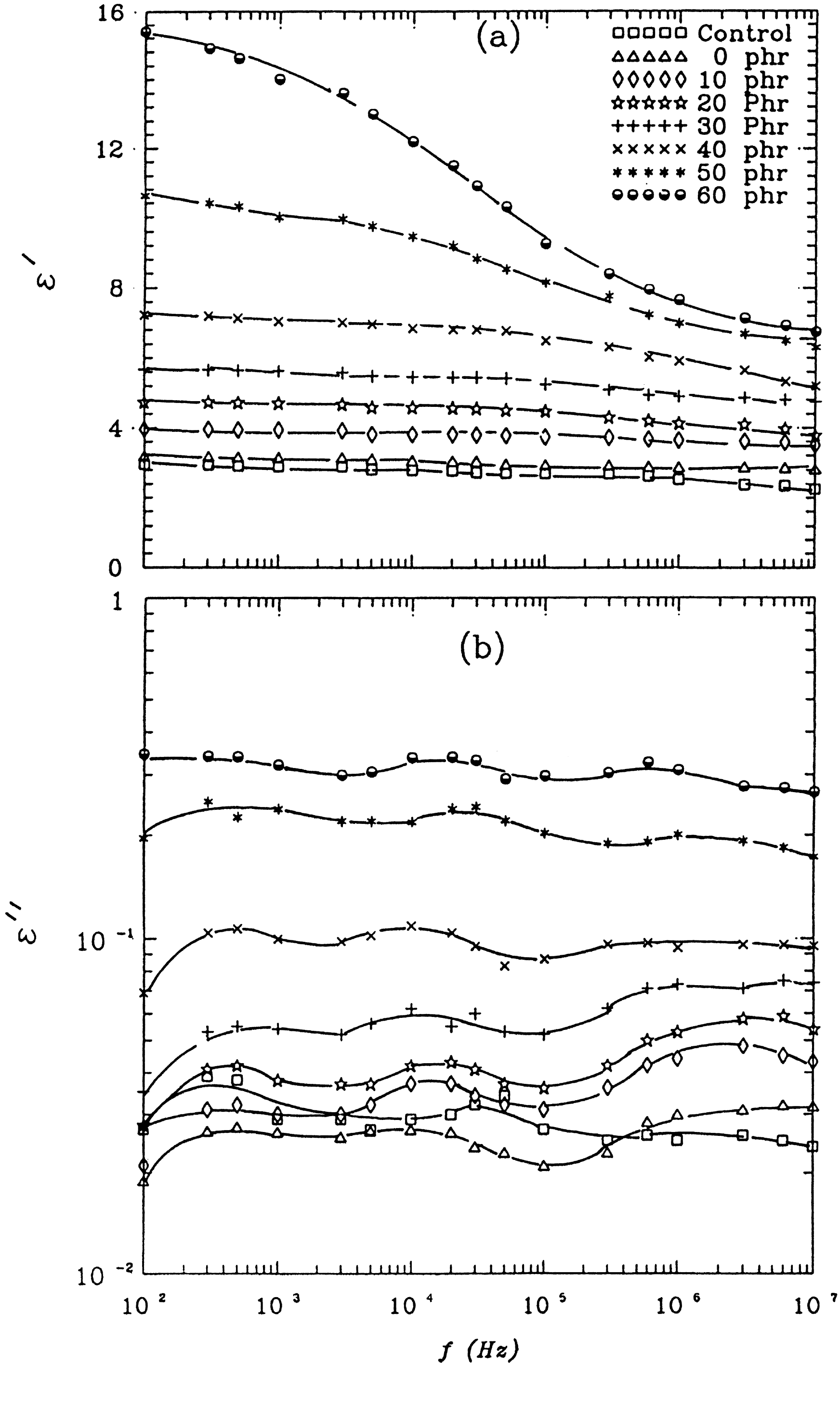
Comparing these data with those obtained before aging. Table 3a, it is clear that the tensile strength, Young's modulus and elongation at break were slightly increased after natural aging. This behavior is similar to that obtained in the case of HAF black [6]. This could indicate that these samples resist the environmental conditions.

DIELECTRIC PROPERTIES

Effect of Frequency on the Dielectric Properties

The permittivity ε' and the dielectric loss ε'' for samples loaded with concentrations from 0 up to 60 phr FEF were measured in the frequency range from 100 Hz to 10 MHz at 30°C. The data obtained are illustrated graphically in Figure 3. From Figure 3a, it is clear that ε' decreases with the increase in frequency which is more pronounced at high concentrations indicating the presence of a dispersion region. The dependence of the dielectric loss ε'' on the frequency, is shown in Figure 3b. The absorption curves obtained are broad, indicating a distribution of relaxation times. Similar behavior has been reported earlier [12–18].

FIGURE 3 (a) The permittivity ε' and (b) the dielectric loss ε'' *versus* frequency (f) for SBR-PE vulcanizate loaded with different contents of FEF black (10, 20, 30, 40, 50, and 60) at 30°C.


(a)
Control
0 phr
10 phr
20 Phr
30 Phr
40 phr
50 phr
60 phr
ε'
16
12
8
4
0
(b)
ε''
1
10 ⁻¹
10 ⁻²
10 ²
10 ³
10 ⁴
10 ⁵
10 ⁶
10 ⁷
f (Hz)

The data were analyzed using a computer program based on superimposed Fröhlich terms [19]. Three absorption regions were obtained. Figure 4 shows an example of the analysis of the sample having concentration 40 phr FEF carbon black. Results of the analysis of the data are given in Table 4. From this table it is clear that ε''_{max} increases with the increase of carbon content. While the distribution parameters *pi*'s are unchanged. No considerable change in the relaxation times $\tau_{I}.\tau_{II}$ is noticed. While the relaxation times τ_{III} have increased by the increase of carbon content. τ_1 could be due to Maxwell-wagner effect [20 – 22], τ_{II} may be due to segmental mobility in the main chain while τ_{III} could be due to cooperative motions of many polar groups in the main chain.

It was noticed that values of the dielectric loss ε'' in case of FEF black samples were much lower than the corresponding ones in the HAF black [6]. This could be attributed to the lower conductivity of FEF than HAF [23].

Effect of Frequency on the Dielectric Properties at Different Temperatures

ε' and ε'' for the different (SBR-PE)/FEF samples were measured at the frequency range from 100 Hz to 50 kHz at temperatures 30, 40 and 60°C. It was found that the changes in ε' and ε'' with temperature are detectable only at high concentrations. The variation of ε' and ε'' *versus* frequency at 30, 40 and 60°C for the sample loaded with 60 phr FEF carbon black is shown in Figure 5. It is clear that values of ε' decrease with frequency and increase with temperature while those of ε'' decrease with frequency and temperature. Two absorption regions are shown in the ε''-log frequency curves, a low frequency one whose maximum at frequencies lower than 1 kHz is due to Maxwell Wagner effect and another absorbtion region at higher frequencies whose peak is at about 15 kHz. This is comparable to the second absorption region obtained from the analysis of this sample given in Table 4 at 30°C before aging. As the temperature rises the height of this absorption region decrease it, becomes broader and shifts to lower frequencies. This could be attributed to the formation of larger aggregates, resulting from the interaction which may take place between carbon black and rubber. This supports the results obtained from thermal aging. Where the relaxation time of this sample increased from 10.98 to 13.26×10^{-6} second before and after thermal aging respectively.

As ε' increases and ε'' decreases with the rise in temperature for SBR-PE vulcanizates loaded with either HAF or FEF carbon black, so both vulcanizates give promising insulating properties at high temperatures.

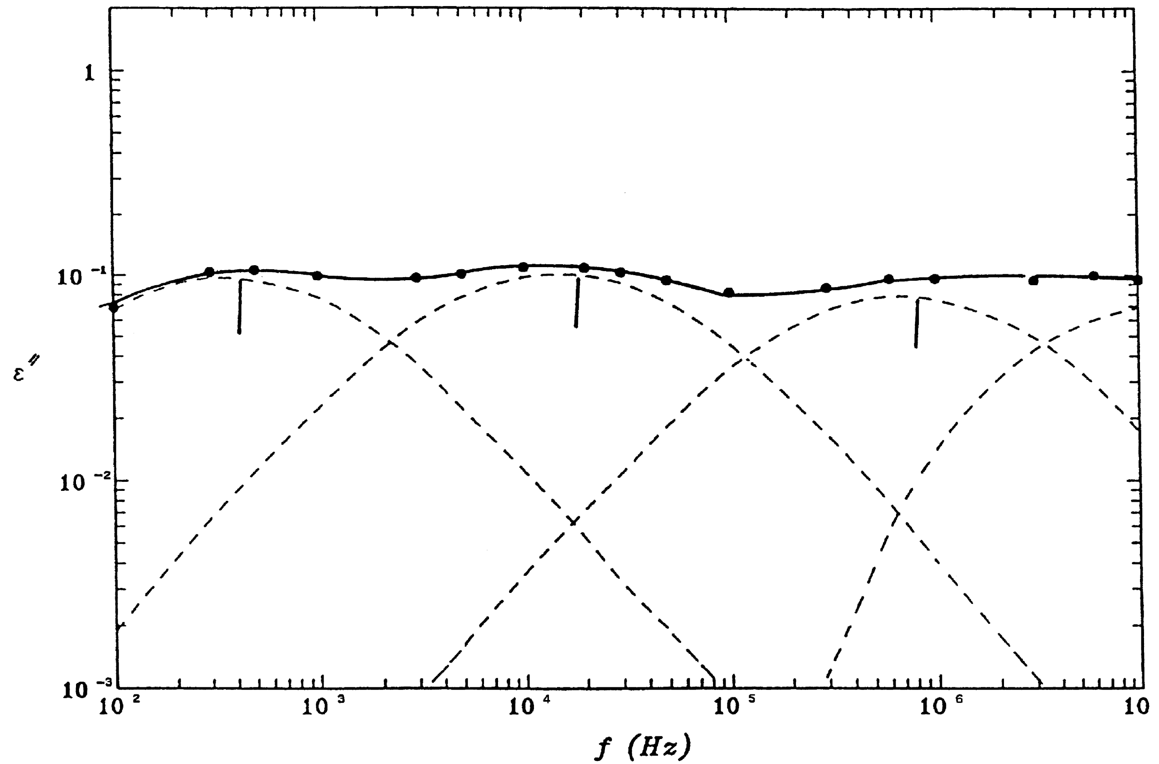


FIGURE 4 Absorption curves of SBR rubber-polyester fiber vulcanizate loaded with 40 phr FEF black. Fitting experiemental ε'' values (●) using Fröhlich terms (solid line).

TABLE 4 Results of the analysis of SBR-PE vulcanizates loaded with FEF black; p_i Fröhlich distribution parameter, $\varepsilon''_{\max}$ value of maximum absorption, and τ_i relaxation time for the first (I), second (II), and third (III) absorption regions at 30°C

Carbon black content	p_i			ε''_{max}			$\tau_i(s)$		
(*phr*)	I	II	III	I	II	III	I × 10^4	II × 10^6	III × 10^8
				Before aging					
0	2.8	2.8	2.8	0.025	0.024	0.025	3.98	9.40	16.75
10	2.8	2.8	2.8	0.029	0.034	0.036	4.19	9.90	19.89
20	2.8	2.8	2.8	0.039	0.038	0.040	4.19	10.00	19.90
30	2.8	2.8	2.8	0.051	0.055	0.060	4.19	10.60	22.74
40	2.8	2.8	2.8	0.097	0.105	0.080	4.19	10.60	23.41
50	2.8	2.8	2.8	0.238	0.215	0.180	4.24	10.60	24.49
60	2.8	2.8	2.8	0.313	0.300	0.265	4.42	10.98	28.94
				After 7-days thermal aging					
0	2.8	2.8	2.8	0.034	0.038	0.029	3.98	10.50	20.94
10	2.8	2.8	2.8	0.042	0.051	0.040	4.19	10.60	21.22
20	2.8	2.8	2.8	0.055	0.065	0.060	4.19	10.60	28.94
30	2.8	2.8	2.8	0.068	0.102	0.073	4.19	10.75	32.15
40	2.8	2.8	2.8	0.130	0.145	0.116	4.42	11.40	39.79
50	2.8	2.8	2.8	0.350	0.362	0.255	4.42	12.20	41.88
60	2.8	2.8	2.8	0.450	0.440	0.320	4.48	13.26	46.81

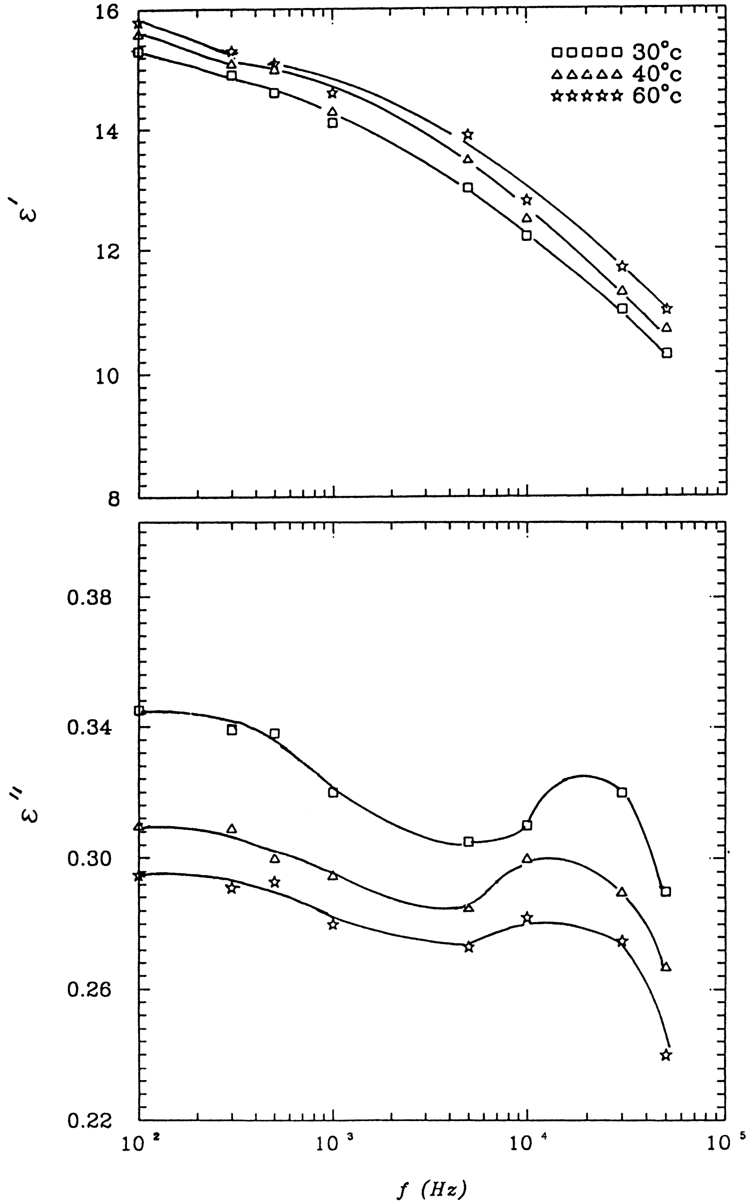


FIGURE 5 The permittivity ε' and the dielectric loss ε'' *versus* frequency (f) for SBR-PE short-fiber Vulcanizates loaded with 60 phr FEF carbon black at different temperatures.

Effect of FEF Concentration on the Dielectric Properties

Figure 6 illustrates the variation of ε' and ε'' *versus* carbon concentration for SBR-PE vulcanizates at 100 Hz and 100 kHz. It is clear that ε' and ε'' increase gradually with the increase of carbon concentration except at 50 phr FEF, where there is an abrupt increase in ε' and ε''. Similar behavior was found in the case of HAF carbon black [6], but it was at 40 phr carbon

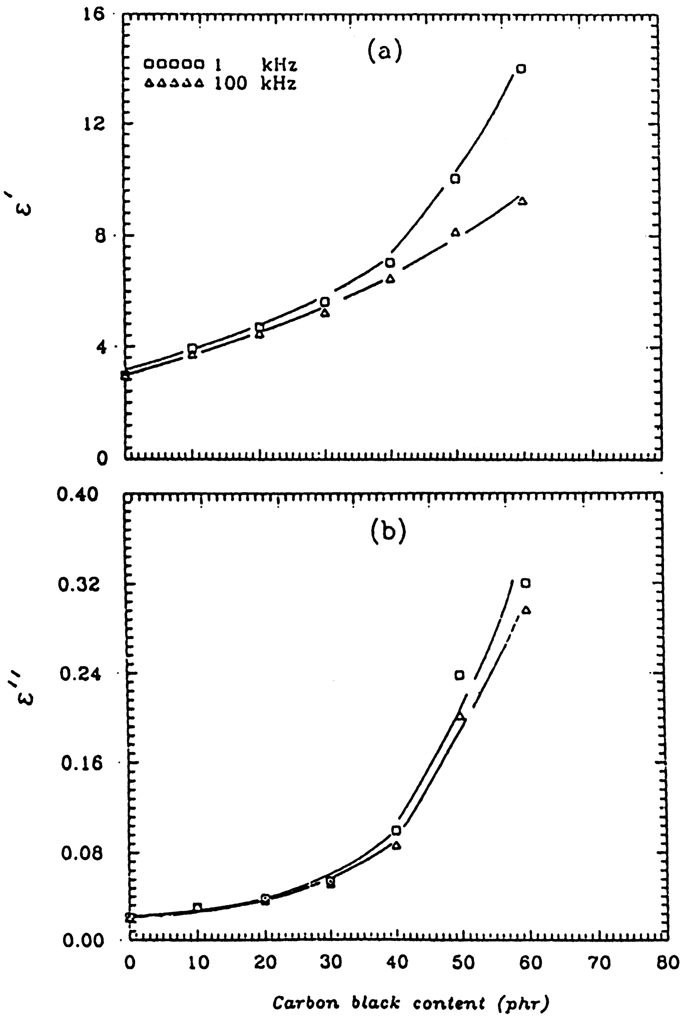


FIGURE 6 Dependence of the (a) dielectric permittivity ε' and (b) dielectric loss ε'' on the carbon block (FEF) content.

content. The concentration at which ε' and ε'' shows sharp increase, is in line with the increase in particle size of HAF and FEF carbon, it is about 28 and 40 nm respectively. This is in good agreement with the results obtained before for SAF, HAF, FEF, SRF and MT carbon black [12–15]. This could be attributed to the interaction which was suggested to take place. Smaller particle size of carbon black have a tendency to form chains [24, 25]. This leads to the onset of percolation behavior.

The logarithms of the relaxation times τ_{III} of the third absorption region were plotted against FEF carbon content, Figure 7. The relation obtained is approximately linear with a slope of about 0.35, while in the case of HAF black mixes it was 0.7. This is considered to be a good support for the suggestion that FEF carbon black has weaker interaction with rubber than HAF.

Effect of Aging on the Dielectric Properties

Thermal Aging

The samples were thermally aged for 7 days. The dielectric properties were measured at a frequency range from 100 Hz to 10 MHz at 30°C.

Comparing these results with those obtained before aging it was found that ε' and ε'' have slightly increased. The data were analyzed as mentioned above, and the results obtained are given in Table 4. No change in the values

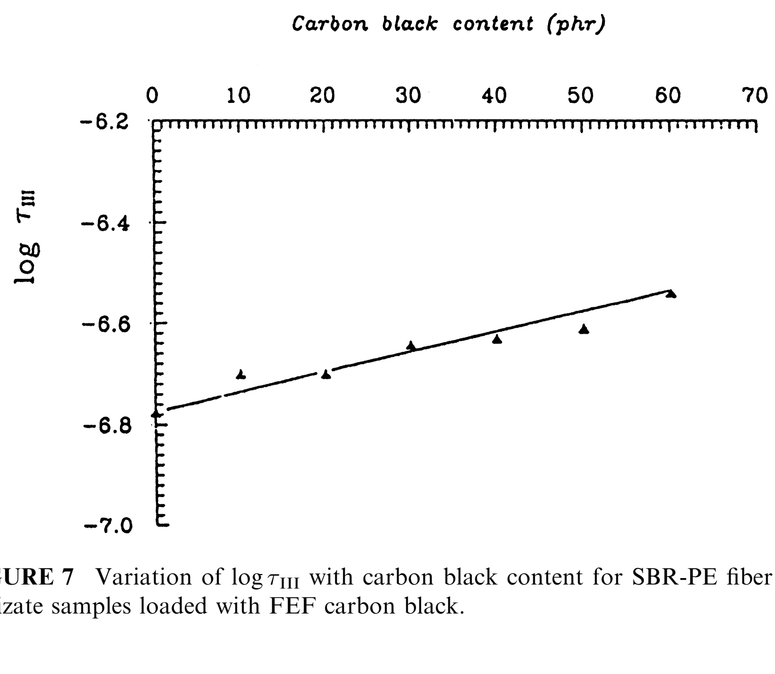


FIGURE 7 Variation of log τ_{III} with carbon black content for SBR-PE fiber vulcanizate samples loaded with FEF carbon black.

of the relaxation times for the first and the second absorption regions is noticed for low FEF concentrations, while for high concentrations the relaxation times τ_{II} have increased. On the other hand, the relaxation time of the third absorption region had increased for all concentrations. This may be due to either the formation of large aggregates caused by the interaction between carbon and elastomer or more likely, due to the formation of cross linked molecules during thermal aging.

Natural Aging

The permittivity ε' and the dielectric loss ε'' were measured after two years on naturally aged samples at a frequency range from 100 Hz to 10 MHz. It was found that no significant change upon natural aging in the values of ε' and ε'' for concentrations up to 50 phr-FEF, while for concentration of 60 phr FEF, ε' and ε'' were slightly higher. This may be due to absorption of moisture by the sample from the environment. This was confirmed by heating the sample at 70°C under vacuum for 24 hours, ε' and ε'' were measured again and the sample has regained the values of ε' and ε'' for the fresh one. Similar trend has been found previously in the case of SBR-PE/HAF composites [6] and others [12–14].

CONCLUSIONS

1. Tensile strength and Young's modulus of SBR-PE composites loaded with either FEF or HAF carbon black have been increased by increasing carbon content in rubber matrix while elongation at break was decreased.
2. Tensile strength and young's modulus in the longitudinal direction were higher than in the transversal one, while an opposite trend was found for elongation at break. This confirm that the PE fiber was fairly oriented in the longitudinal direction in rubber matrix, in both composites loaded with FEF or HAF carbon black.
3. The retaining ability of tensile strength and Young's modulus for the composites loaded with either FEF or HAF carbon is increased by increasing aging time and decreases with the increase of carbon content in the rubber matrix.
4. After 7-days thermal aging the retaining ability for the composites loaded with 10 phr of either HAF or FEF carbon black has been increased up to 162% and 190% at the longitudinal and transversal directions respectively.
5. The mechanical and dielectric properties for samples loaded with smaller particle size carbon black HAF [6] are higher than those loaded with larger particle size carbon black SRF [9]. This is attributed to a good dispersion of carbon black having smaller particle size in the polymer matrix.

6. At certain concentration of carbon black in rubber matrix there was an abrupt increase in the permittivity ε' and the dielectric loss ε''. The concentration at which ε' and ε'' shows sharp increase is in line with the increase of carbon black particle size. This could be attributed to the onset of percolative behavior of the carbon black particles.
7. The dielectric permittivity ε' has been increased by increasing the temperature, whereas the dielectric loss ε'' was decreased for composites loaded with either HAF or FEF.
8. The dielectric losses for the composites loaded with FEF carbon black were much lower than the corresponding ones loaded with HAF carbon black.

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