

# Preparation And Modelling Of Ac Properties For Surface-Modified Haf Loaded Sbr Matrix

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**Abstract**—In this work, two groups of carbon black / styrene butadiene rubber composites were prepared. In the first, carbon black CB was added directly to the SBR while in the second, it was immersed in a butadiene solution before it was added to the SBR matrix. Dielectric measurements show a tremendous increase in the dielectric constant for the modified group than the neat sample. Several models were then used to account for the experimental data. Also the conduction mechanism could be determined.

**Keywords**—Carbon Black, Polymer, Dielectric Constant.

## Introduction

Conducting polymer composites (CPCs) are traditionally prepared by blending electrically conductive filler with the polymer matrix[1-6]. Several types of fillers are widely used such as carbon black, graphite, silica, Al<sub>2</sub>O<sub>3</sub>, etc. Among those, carbon black has been extensively employed because it renders the polymer matrix conductive at relatively low filler content [1]. In addition it enhances the mechanical properties of the matrix. However, increasing the CB content to achieve a certain level of conductivity may cause a deterioration of the material. That is it is one of the technical and economic importance is to achieve a satisfactory level of conductivity at relatively low filler loading [2].

However, the conductivity of CPCs filled with CB does not depend only on the filler content but also on the surface properties of the two phases at the interface separating them and on the continuity of the filler phase inside the matrix. CB is well known to have a tendency to form aggregates and therefore at normal conditions, it is not distributed uniformly inside the matrix and therefore charge carriers faces a large energy gap at the interface separating the two phases. One of the methods used to reduce the gap other than increasing the filler content is to increase the surface area of the interface [3-6].

This work focuses on exploring and testing the validity of modifying the surface area of the interface through immersing the CB into an active solution of the same type as the used matrix, on the dielectric properties of the prepared composites

## Experimental work

### 1. Materials and Sample preparation

Bulk styrene butadiene rubber (SBR) was supplied by

Ingredients	Phr*
SBR	100
Stearic acid	2
Zinc oxide	5
Processing oil	10
HAF (before treatment)	(0, 10, 20, 30, 50, 60, 70, 90)
HAF (after treatment)	(10, 30, 50, 70)
MBTS <sup>a</sup>	2
PBN <sup>b</sup>	1
Sulfur	2

**Table (1)** Composition of the composite

\* Parts per hundred parts by weight of rubber.

a Dibenzthiazyle disuiphide

b Phenyl - β - naphthylamine

TRENCO, Alexandria, Egypt. While polybutadiene solution supplied by Merck, Germany was used in this study. High abrasion furnace (HAF N330) black was also supplied by TRENCO.

The calculation of the true graphite volume fraction was performed through the following relationship

$$\varphi_{\text{true graphite}} = \frac{w_{\text{true graphite}} / \rho_{\text{true graphite}}}{\sum_i w_i / \rho_i}$$

(1)

Where  $w_i$  and  $\rho_i$  are the weight fraction and the density of the  $i^{\text{th}}$  phase, respectively.

Samples of SBR were prepared according to standard methods with the compositions shown in **Table (1)**. The test samples were dumbbell shaped with dimensions  $2 \times 3 \times 10 \text{ cm}^3$ . All samples were vulcanized at  $(153 \pm 2) \text{ }^\circ\text{C}$  under a pressure of  $40 \text{ kg/mm}^2$  for 20 min. For CB modified samples, CB was immersed in the polybutadiene solution for 30 min to allow the mixture to become homogeneous, and then was added according to the same recipe in **Table (1)** (taking into account the mass of the styrene solution that was used) as in the untreated samples. They were then thermally aged at  $90 \text{ }^\circ\text{C}$  for 35 days to attain reasonable stability and reproducibility of measured quantities.

### 2. Measurements

Conducting polymer composites possess a frequency ( $\omega$ ) dependent, complex dielectric constant  $\varepsilon^*(\omega) = \varepsilon'(\omega) - i\varepsilon''(\omega)$ . The real part  $\varepsilon'(\omega)$  represents

the dielectric constant and the imaginary part  $\epsilon''(\omega)$  accounts for the dielectric loss. The ratio of the imaginary to the real part ( $\epsilon''/\epsilon'$ ) is the "dissipation factor" which is denoted by  $\tan \delta$ , where  $\delta$  is called as the "loss angle" denoting the phase angle between the voltage and the charging current. The AC conductivity can be deduced from the equation:  $\sigma_{AC} = 2\pi f \epsilon_0 \epsilon''$ , where  $f$  is the frequency, and  $\epsilon_0$  is the permittivity of free space.

The dielectric properties were measured using a bridge (GM Instek LCR – 821 meter) in the frequency range  $10^2 - 10^5$  Hz. Both samples capacitance and  $\tan \delta$  values were measured for all samples at different temperatures and frequencies. The dielectric constant  $\epsilon'$  (real part of the complex dielectric constant) of the samples was calculated by using the relation.

$$\epsilon' = \frac{d}{\epsilon_0 A} C$$

(2)  
 Where  $C$  is the capacitance of the sample,  $d$  is the thickness of the sample,  $A$  is the cross-sectional area of each of the parallel surfaces of the sample.

## Results and Discussion

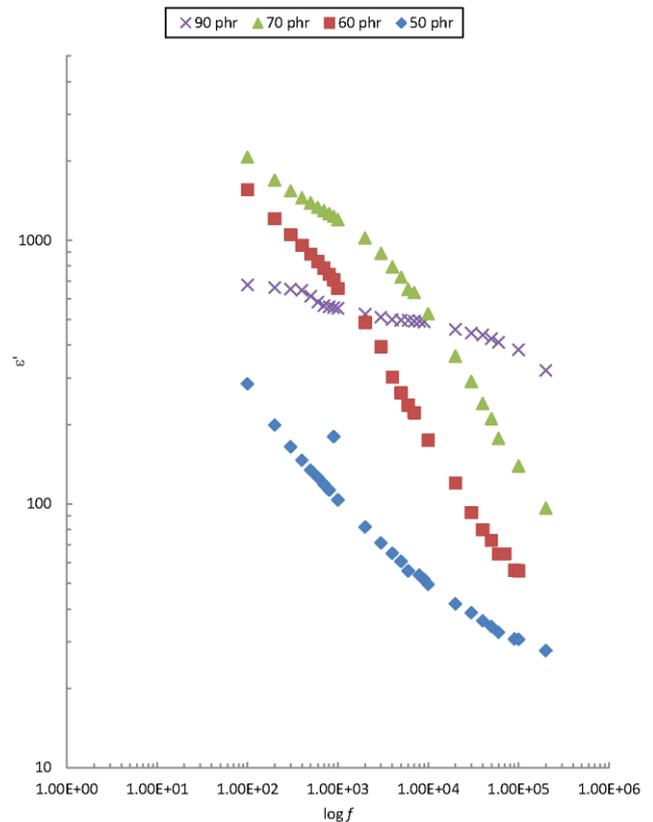
### Un treated HAF black

#### (a) Frequency Dependence

The frequency dependence of the dielectric constant  $\epsilon'$  at room temperature ( $\sim 300$  K) of SBR loaded with different HAF content is shown in Figure (1). The plots in **Fig. (1)** show that  $\log \epsilon'$  decreases almost linearly with  $\log f$  in the frequency range from  $10^2$  Hz to  $10^5$  Hz, and remains at almost constant for higher frequencies. The addition of HAF black clearly increases the value of the dielectric constant  $\epsilon'$  [**Fig.(2)**]. Upon adding untreated-HAF black to SBR matrix, the dielectric constant increases moderately until the percolation value is attained at which the dielectric constant increases abruptly and reaches a value of 1200 for the value 0.3 of CB volume fraction. This increase in the value of the dielectric constant may be attributed to the buildup of space charges at the interface separating the conducting phase and the insulating phase due to the difference in the conductivities of the two phases. This buildup process is referred to as Maxwell-Wagner polarization originating in the insulator - conductor interfaces. Space charge polarization occurs when more than one component is present or when segregation occurs in a material that contains incompatible chemical species and mobile charge carriers are trapped at the interfaces of these heterogeneous systems. The electric field distortion caused by these trapped charges increases the overall capacitance as well as the dielectric constant of the system [7]

#### (b) Effect of HAF black contents

The dielectric constant of a composite dielectric containing more than one component may be expressed in the general form



**Figure (1)** show the variation of the dielectric constant with the frequency for un-treated HAF loaded SBR samples

$$F(\epsilon'_c) = \sum_{i=1}^m V_i F(\epsilon'_i)$$

(3)

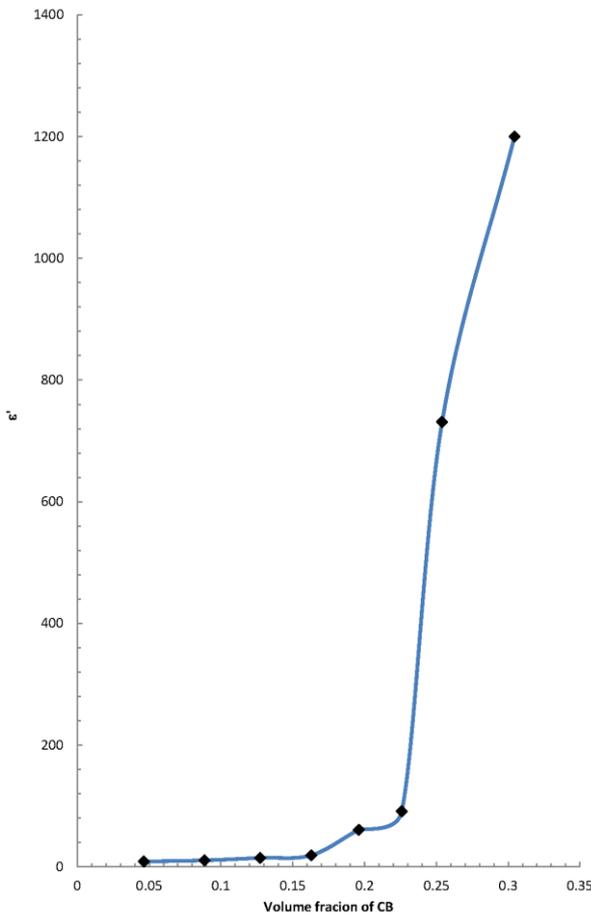
Where  $F(\epsilon'_c)$  is a function of the composite dielectric constant,  $V_i$  and  $\epsilon'_i$  are the volume fraction and the dielectric constant for the  $i^{\text{th}}$  component of the composite containing " $m$ " number of components. For two components system, **Eq.(3)** takes the form

$$F(\epsilon'_c) = \epsilon'_1 V_1 + \epsilon'_2 (1-V_1)$$

(4)

Where  $\epsilon'_1$  and  $\epsilon'_2$  are the dielectric constants of components 1 and 2 respectively and  $V_1$  is the volume fraction of component 1.

**Figures (2–4)** illustrate the dielectric interaction pattern of mixed systems, with respect to HAF contents. The dielectric constant  $\epsilon'_c$  in these plots is calculated at a frequency of  $10^4$  Hz; where both dispersion and relaxation phenomena are most operative. The plot of  $\epsilon'_c$  versus  $V_2$ , the volume fraction of the HAF carbon black, shows clear non-linear behavior [Figure (2)].



**Figure (2)** shows the variation of the dielectric constant with the volume fraction of carbon black at a frequency  $10^4$  Hz.

However, plot of  $(1/\epsilon'_c)$  versus  $V_2$ , which describes the law of harmonic mixture is linear [Figure (3)]. This would imply a series combination of the blend constituents. Extrapolation of the plots to  $V_2 = 0$  yields  $1/\epsilon'_c = 1/\epsilon'_1$ , where  $\epsilon'_1$  is the dielectric constant of SBR matrix at  $f = 10^4$  Hz. Graphically-obtained value of  $\epsilon'_1$  disagrees with the experimentally obtained value [Table (2)].

A trial was made to test the applicability of the Clausius-Mossotti equation for a mixture of dielectrics in the present systems. Slopes and intercepts are obtained when the specific polarization  $(\epsilon'_c - 1/\epsilon'_c + 2)$  is plotted against  $V_2$ . [Figure(4)]

The dielectric constant  $\epsilon'_c$  can be theoretically obtained from the intercepts of these plots at  $V_2 = 0$ . The calculated value of  $\epsilon'_c$  shows large deviation from experimental one as shown in Table (2).

(exp)	(logarithmic law)	(Clausius-Mossoti)
2.56	7.69	5.89

**Table (2)** The experimentally obtained value for the dielectric constant and its calculated values using the logarithmic law and the clausius- Mossotti equation.

One of the successful models is that due to Bruggman [8], who considered spherical conductive inclusions. Bruggman's equation can be simplified by assuming that the dielectric constant of the conductive inclusions, tends to infinity, yielding Eq. (5), which is often used, provided that the volume fraction of the inclusion is considerably less than unity ( $V_2 \ll 1$ )

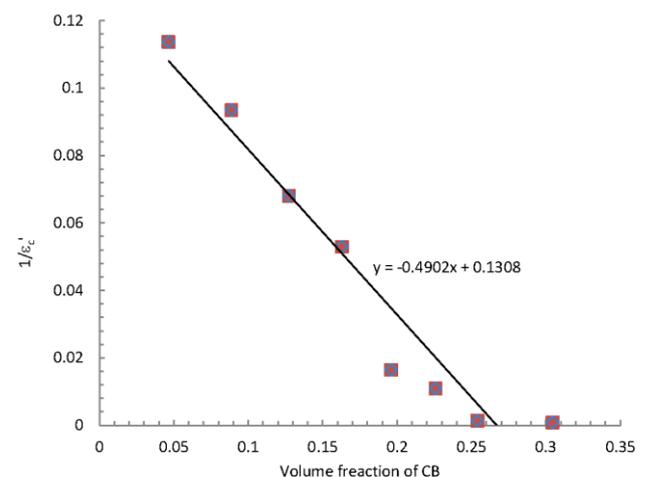
$$\epsilon'_c = \epsilon'_1 (1 + 3 V_2) \quad (5)$$

Subscript (1) refers to the SBR matrix, and subscript (2) refers to the filler. The calculated values of  $\epsilon'_c$  for SBR loaded with HAF contents are given in Table (3). A disagreement between the calculated and the experimental values was observed especially for higher volume fractions.

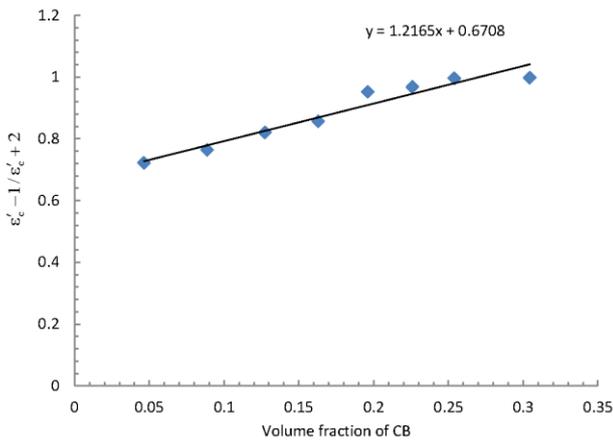
Volume fraction	$\epsilon'_c$	
	(Bruggman)	(Experimental Values)
0.0464	9.1136	9.34
0.08865	10.1276	15.7
0.1273	11.0552	18
0.16286	11.90864	21.1
0.196	12.704	49.5
0.2259	13.4216	174
0.254	14.096	526
0.3044	15.3056	483

**Table (3)** The calculated values (from Bruggman model) and the experimentally obtained values of the dielectric constant as a function of the volume fraction of carbon black.

In all the above formulae, the dielectric constant is supposed to be independent of both the applied field and the components characteristics. This was taken care of by Tsangaris *et al* [9], who proposed a new model with suitable equations formulated to express the dielectric constant  $\epsilon'_c$  as a function of the applied frequency and the components characteristics. This equation is as follows



**Figure (3)** Plot of  $(1/\epsilon'_c)$  versus  $V_2$ , which describes the law of harmonic mixture.



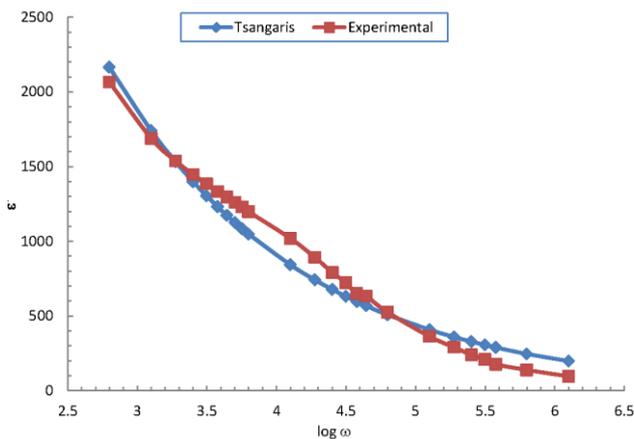
**Figure (4)** The specific polarization  $(\epsilon'_c - 1 / \epsilon'_c + 2)$  is plotted against  $V_2$ , which describes Clasius- Mossotti equation

$$\epsilon'_c = \frac{\epsilon'_1}{\left[ (\epsilon'_1 - 1)^Y + 1 \right]} \left\{ \left[ \left( \frac{\sigma}{\omega \epsilon_o} \right)^{Y_2} (\epsilon'_1 - 1)^{1-Y_2} \cos \left( \frac{\pi V_2}{2} \right) \right]^Y + 1 \right\} \quad (6)$$

Where  $Y$  is the depolarizing factor given by

$$Y = \frac{1}{1 - (a/b)^2} - \frac{a/b}{\left[ 1 - (a/b)^2 \right]^{3/2}} \cos^{-1} \left( \frac{a}{b} \right) \quad (7)$$

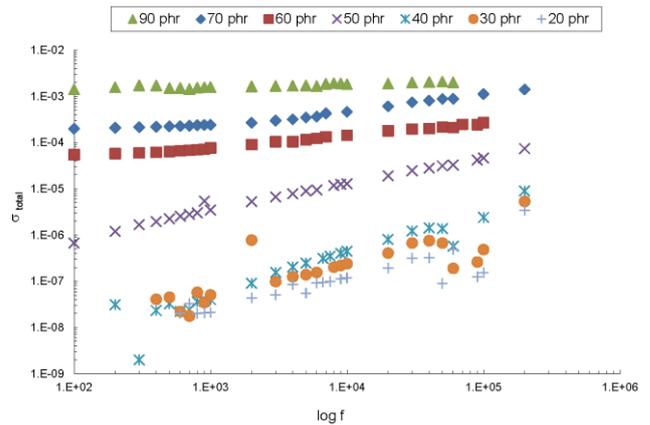
Where  $\omega$  is the frequency,  $\sigma$  is the conductivity of the filler,  $\epsilon'_1$  is the dielectric constant of the SBR matrix, and  $a/b$  is the aspect ratio of the filler ( $a$  and  $b$  are the semi-major and semi-minor axes respectively).



**Figure (5)** shows the fitting of the experimental data with Tsangaris model for 70 phr sample

**Fig.(5)** represents the good fitting of the calculated values of dielectric constant using **Eq.(6)** and the experimental data for the sample (70 phr) as an example. The shape of HAF – black may be transformed from spherical to ellipsoidal or even a long rod shape according to the volume fraction of HAF-black particles and thus the depolarizing factor  $Y$

is assumed to vary with the variation of the HAF contents within the SBR matrix.

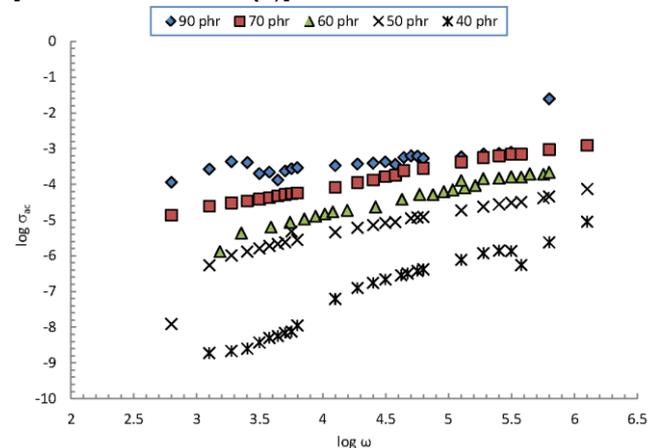


**Figure (6)** shows the variation of the total conductivity as a function of frequency for all samples at room temperature

In **Fig.(6)**, the variation of the measured conductivity as a function of the frequency is shown, at room temperature, for all HAF loaded SBR samples. From this figure, it is evident that the DC contribution is significant at low frequencies, whereas the frequency-dependent term is significant at high frequencies. The total conductivity  $\sigma_{tot}$  can be represented by the following equation [10]

$$\sigma_{tot}(f) = \sigma_{dc} + A \omega^s \quad (8)$$

Where  $A$  is a constant and the superscript  $s$  is a frequency exponent with values lying between 0 and 1. From **Eq.(8)**, it is evident that the values of  $\sigma_{dc}$  can be estimated from the plateau values of conductivity in the  $\sigma_{tot} - f$  plots. Such values of  $\sigma_{dc}$  are in satisfactory agreement with the measured  $\sigma_{dc}$  values [As shown in **Table (4)**].



**Figure (7)** Curves resulting from subtracting  $\sigma_{dc}$  from  $\sigma_{tot}$  data, to obtain the a.c. for different samples at 300 K

On subtracting  $\sigma_{dc}$  from  $\sigma_{tot}$  data, we obtain the AC conductivity at different frequencies. Curves resulting from this procedure are presented in **Fig.(7)** for different samples at 300 K.

Sample (phr)	$\sigma_{dc}$ (calculated) ( $\Omega^{-1} m^{-1}$ )	$\sigma_{dc}$ (measured) ( $\Omega^{-1} m^{-1}$ )
10	$0.92 \times 10^{-12}$	$1.12 \times 10^{-12}$
20	$4.40 \times 10^{-12}$	$5.35 \times 10^{-12}$
30	$1.93 \times 10^{-11}$	$2.19 \times 10^{-11}$
40	$5.22 \times 10^{-11}$	$5.00 \times 10^{-11}$
50	$7.51 \times 10^{-7}$	$7.27 \times 10^{-7}$
60	$1.14 \times 10^{-4}$	$1.23 \times 10^{-4}$
70	$6.12 \times 10^{-4}$	$6.30 \times 10^{-4}$
90	$4.01 \times 10^{-3}$	$3.90 \times 10^{-3}$

**Table (4)** gives the calculated and experimental values of  $\sigma_{dc}$  for all samples

It is observed that  $\sigma_{ac}$  has a frequency dependence given by

$$\sigma_{ac} \propto f^s \quad (9)$$

Where the exponent  $s$  is seen to be a function of the frequency  $f$ . The  $s$  - values could be determined from the slope of the  $\log \sigma_{ac} - \log f$  curve. For all loaded SBR samples, one could fit the plotted data by a polynomial of the form

$$\log \sigma_{ac}(f) = a + b (\log f) + c (\log f)^2 \quad (10)$$

The slope at any frequency  $f$  was obtained from the equation [10]

$$s = \frac{d(\log \sigma)}{d(\log f)} \quad (11)$$

By using Eqs. (8) & (9), we obtain

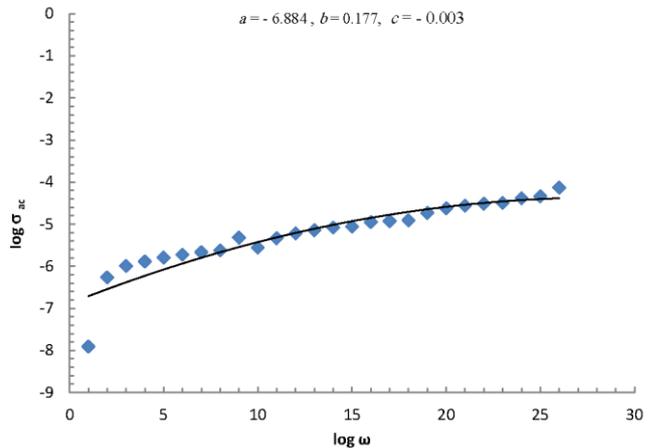
$$s = b + 2c(\log f) \quad (12)$$

In **Fig.(8)**; we get a typical fitting data for the sample 50 phr. The solid line represents the calculated data which fits well the experimental points. **Fig.(9)** shows the dependence of  $s$  on  $\log \omega$  for all samples. The implications of these data are explained on the basis of different models proposed for amorphous semiconductors [11].

Several theoretical models have been proposed for AC conduction in amorphous semiconductors. Two distinct mechanisms have been developed for the relaxation phenomena:

- [1] Quantum Mechanical Tunneling (QMT) of electrons or polarons through the barrier separating localized states and;
- [2] Classical hopping over the same barrier (CBH)

For QMT of electrons, the frequency exponent  $s$  is deduced to be



**Figure (8)** A typical fitting data for 50 phr sample, to find the constants  $a$ ,  $b$ ,  $c$ .

$$s = 1 - \frac{4}{\ln\left(\frac{1}{\omega\tau_o}\right)} \quad (13)$$

i.e.,  $s$  is temperature independent, while, for CBH model that was proposed by Pike [12], the frequency exponent  $s$  is evaluated to be

$$s = 1 - \frac{6kT}{\left[W_M - kT \ln\left(\frac{1}{\omega\tau_o}\right)\right]} \quad (14)$$

Where  $W_M$  is the maximum barrier height. It is evident from **Figure (9)** that the frequency exponent  $s$  decreases as a function of frequency for samples 30 phr and 50 phr. This suggests that the variation follows **Eq. (13)**, indicating a QMT mechanism is operative in these samples. Meanwhile, for samples 40 phr, 60 phr, 70 phr, 90 phr, the frequency exponent  $s$  increases as a function of frequency indicating the existence of CBH mechanism. The extracted values of  $\tau_o$  for 30 phr and 50 phr loaded SBR samples with the QMT model are summarized in **Table (5)**.

Sample (phr)	$\tau_o$ (s)
30	$1 \times 10^{-5}$
50	$4.95 \times 10^{-9}$

**Table (5)** shows the value of  $\tau_o$  for the samples 30 phr and 50 phr, which exhibit QMT.

### 3.2.2 Treated Surface HAF black

It is a well-established fact that, as two surfaces becomes identical, the interfacial adhesion tension between them becomes zero leading to a maximum in adhesion energy. Obviously, the interface in any multiphase polymeric system should be modified properly to approach this optimum condition [13]. HAF carbon black was coated mechanically by polystyrene. Previously weighted amounts of HAF –black were poured into the beaker which filled with polystyrene solution. System was mixed well and allowed to dry slowly in vacuum oven at room temperature to the constant weight. The modified coated HAF black is

with the same concentrations of untreated HAF black this could be attributed to the fact that the HAF particles are coated with a thicker layer of polystyrene (all prepared by solution casting method from their polymer solutions) and as the coat becomes thicker; the degree of reinforcement gets weaker as expected as pores of the filler were blocked more and fewer available sites for wetting results.

#### (b) HAF Black contents

Figures (11–13) illustrate the dielectric interaction pattern of mixed systems, with respect to the modified HAF black contents. The dielectric constant  $\epsilon_c'$  in the plots is observed at frequency  $10^4$  Hz (as those calculated previously in Figures (2 - 4)).

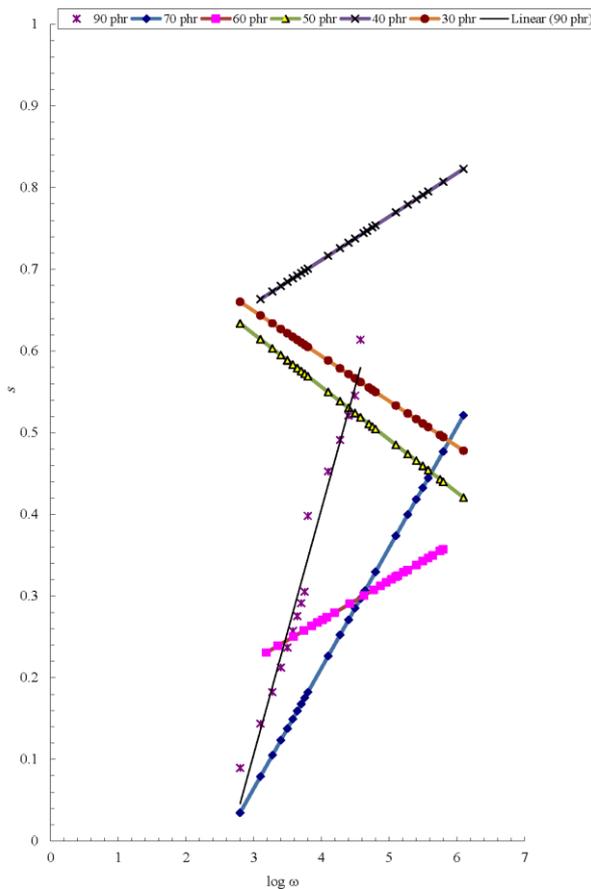


Figure (9) The dependence of  $s$  on  $\log \omega$  for all samples.

then added to the SBR matrix in different ratios (10, 30, 50, 70 phr).

#### (a) Frequency Dependence

The frequency dependence, of the dielectric constant  $\epsilon_c'$  at room temperature (300 K) of SBR loaded with treated HAF carbon black is shown in Fig.(10). The plots in Fig.(10) also show that  $\log \epsilon_c'$  decreases almost linearly with  $\log f$  in the frequency range 100 Hz to 5 kHz and remains at almost constant value at higher frequencies. But the recorded values of  $\epsilon_c'$  for SBR loaded with treated HAF black, are lower than that recorded for SBR samples loaded

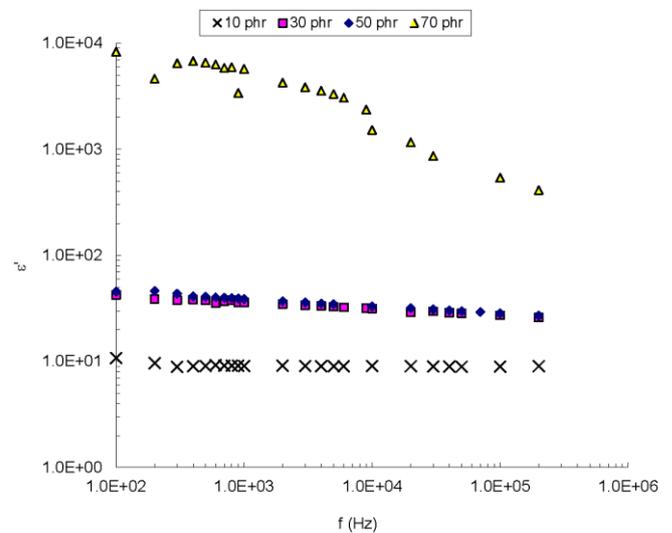
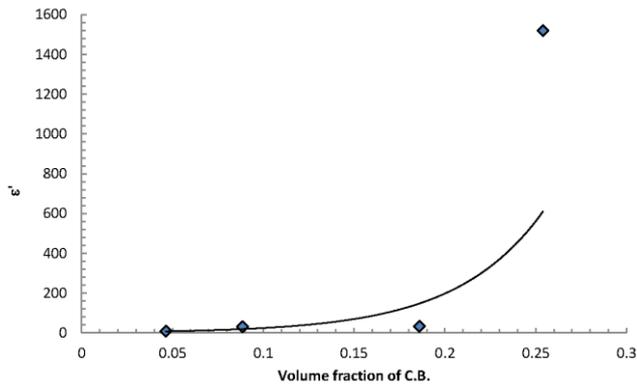


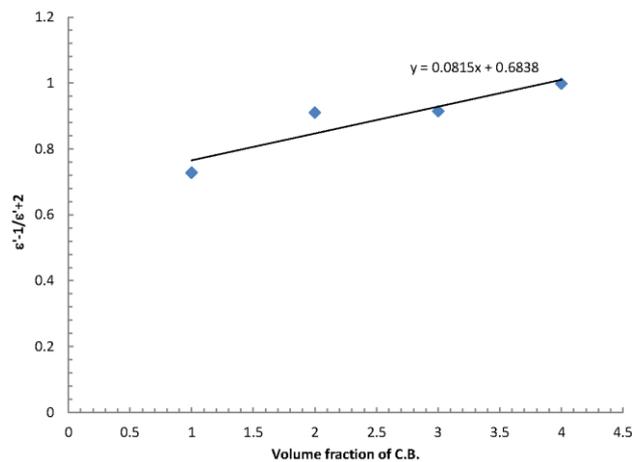
Figure (10) The frequency dependence of the dielectric constant for modified samples containing 10, 30 phr, 50 phr, 70 phr.

The plot of  $\epsilon_c'$  versus the volume fraction of HAF-black  $V_2$  [Figure ( 11)], still shows clear non-linear behavior as detected for samples loaded with untreated HAF black. However, for higher HAF loading (70 phr), the  $\epsilon_c'$  value for treated HAF black loaded SBR sample is greater than that for untreated sample.

Plots of  $(1/\epsilon_c')$  versus  $V_2$ , which describes the law of harmonic mixtures are still linear [Figure (12)]. Extrapolation of the plot to  $V_2 = 0$ , yields  $1/\epsilon_c' = 1/\epsilon_1'$ , where  $\epsilon_1'$  is the dielectric constant of SBR matrix at a frequency  $10^4$  Hz. Graphically obtained value of  $\epsilon_1'$  still disagrees with the experimentally obtained value as shown in Table (6).



**Figure (11)** The variation of the dielectric constant with the volume fraction of CB.



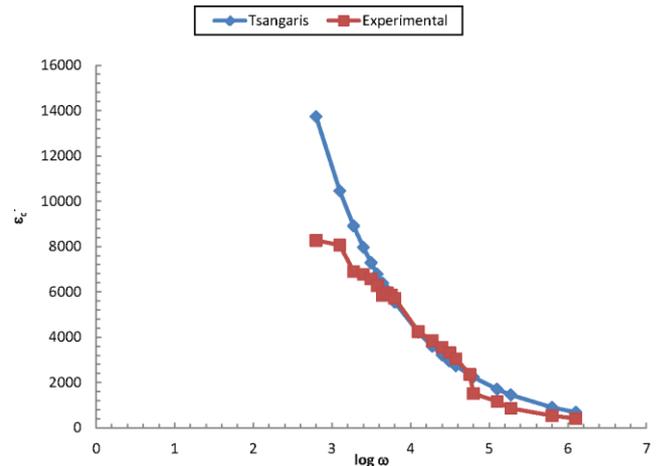
**Figure (13)** The specific polarization ( $\epsilon_c' - 1 / \epsilon_c' + 2$ ) is plotted against  $V_2$ , which describes Clasius- Mossotti equation

For the case of Clasius – Mosotti equation, for the treated samples, slopes and intercepts are obtained when the specific polarization  $[(\epsilon_c' - 1) / (\epsilon_c' + 2)]$  is plotted versus  $V_2$  (Figure 13). The calculated value of  $\epsilon_c'$  obtained from the intercept of the plot at  $V_2 = 0$  still shows some deviation from the experimental one as shown in **Table (6)**

$\epsilon_c'$		
(exp)	(logarithmic law)	(Clausius-Mossotti)
2.56	9.62	7.46

**Table (6)** The experimental value of the dielectric constant as compared to it's value obtained using the logarithmic law and the Clausius- Mosotti equation.

The calculated values of  $\epsilon_c'$  for treated SBR samples using Bruggmann's **Eq.(7)** are also given in **Table (7)** and a disagreement between the values with respect to the experimental one is observed.

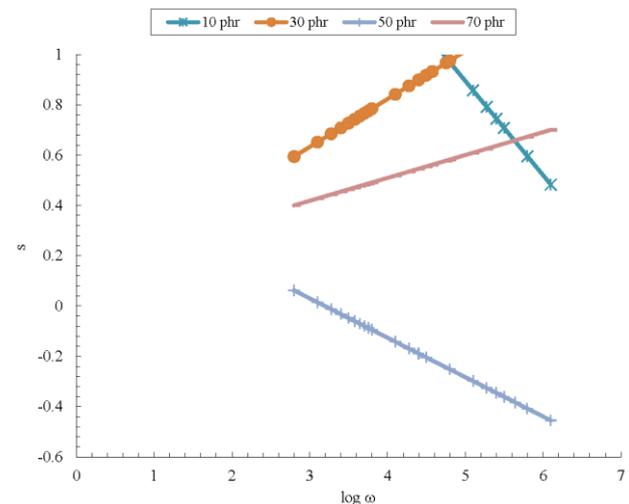


**Figure (14)** The fitting of the experimental data with Tsangaris model for treated 70 phr sample

Volume fraction	$\epsilon_c'$	
	(Bruggman)	(Experimental)
0.0464	9.1136	9.02
0.1273	11.0552	31.4
0.196	12.704	33.1
0.254	14.096	1520

**Table (7)** The calculated values (from Bruggman model) and the experimentally obtained values of the dielectric constant as a function of the volume fraction of carbon black.

Fig.(14) represents the good fitting of the calculated values of  $\epsilon_c'$  according to Tsangaris **Eq.(6)** and experimental data for 70 phr treated HAF/SBR samples as an example. The shape of the HAF – black and the depolarizing factor varied appreciably with the process of treatment of HAF- black as observed from **Table (7)**. The AC conduction mechanisms were also elucidated for treated samples using Eqs.(13) and (14). one could found that SBR samples loaded with 10, 50 phr treated HAF – black follows a QMT mechanism, meanwhile, SBR samples loaded with 30, 70 phr treated HAF-black follow the CBH mechanism as indicated in **Fig.(15)**



**Figure (15)** The dependence of s on log ω for treated samples.

## Conclusions

Surface-modified carbon black when used as reinforcing filler causes enhancement in the value of the dielectric constant as well as the ac conductivity of the composite as compared to ordinary mixed composites, and this could be attributed explained as a result of increasing the effective surface area of the interface separating the two phases of the composite.

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