

Electrical Properties of Natural-Fiber-Reinforced Low Density Polyethylene Composites: A Comparison with Carbon Black and Glass-Fiber-Filled Low Density Polyethylene Composites

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SYNOPSIS

The electrical properties of sisal fiber–low density polyethylene (LDPE) and coir fiber–LDPE composites have been studied. The dielectric constant progressively increases with increase of fiber loading and decreases with increase of frequency in the case of all composites. The dielectric constant of sisal–LDPE composites has been studied as a function of fiber length. Volume resistivity values decrease with fiber content. The increase of dielectric constant with fiber loading is more predominant at low frequencies in both the sisal fiber–LDPE and coir fiber–LDPE composites. The results of the natural-fiber-filled composites were compared to those of the carbon and glass-fiber-filled LDPE composites. The dielectric constant of carbon-black-loaded LDPE composites increases with carbon content, and the increase is sharper at high carbon content. This is associated with the network formation of carbon black in LDPE matrix. © 1997 John Wiley & Sons, Inc.

INTRODUCTION

The use of cellulosic materials in thermoplastic composites is highly beneficial because the strength and toughness of the plastics can be improved. Moreover, natural fibers are very cheap, highly available, and renewable. So the natural-fiber-reinforced thermoplastic composite based products are more economical to produce than the original thermoplastics.¹ The use of composite materials in engineering as dielectric is becoming increasingly important. Therefore, studies on the electrical properties of fiber-reinforced thermoplastic composites are very important. Volume resistivity and dielectric strength of some natural fibers have been measured by Kulkarni et al.² Poulaert and Issi studied the low temperature electrical resistivity of carbon-black-loaded high density polyethylene.³ Li et al. have re-

ported the studies of electrical properties of nickel coated carbon-fiber-reinforced polyether sulfone composites.⁴ Dielectric behavior of the wood–polystyrene composite has been studied by Poliszko and Hoffman.⁵ Debnath, De, and Khastigir have studied the effect of addition of mica, silane coupling agent, and silane treated mica on the ambient dielectric properties of vulcanized styrene–butadiene rubber.⁶ Dipole relaxation dielectric loss behavior of a fiber glass–epoxy composite has been studied as a function of moisture by Reid et al.⁷ This study of the dielectric relaxation behavior of a glass–epoxy composite has suggested three modes of moisture interaction with, or adsorption by, the composite. Bhat-tacharya et al.⁸ have studied the effect of temperature on the electrical conductivity of poly(vinyl chloride) (PVC)–copper composites. Tetracyanofuorodimethane salts have been incorporated into insulating polymers to raise their dielectric constants by Kita and Okamoto.⁹ Noguchi et al.¹⁰ have investigated the melt flow behavior and electrical conductivity of carbon black–PVC systems and ex-

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Table I Physical and Mechanical Properties of Low Density Polyethylene^a

Melt Flow Index (g/10 min)	Density (kg/m ³)	Tensile Strength at Break (kg/cm ²)	Elongation at Break (%)	Modulus Elasticity (kg/cm ²)	Vicat Softening Point (°C)	Crystalline Melting Point (°C)
40	916	90	200	1400	85	104

LDPE = Indothene 16 MA 400.

plained the properties in terms of the structure of carbon black aggregates. Electrical and electromechanical measurements for rubber filled with carbon black in the 0–70 phr range were done by Burton et al.¹¹ However, very limited studies have been reported on the electrical properties of lignocellulosic fiber-reinforced polymer composites. In the present paper, the dielectric properties of sisal fiber and coir fiber-reinforced polyethylene composites have been studied with special reference to the effects of fiber loading, fiber length, and frequency. The properties of these composites have been compared to those of carbon black and glass fiber-filled low density polyethylene (LDPE) composites.

EXPERIMENTAL

The properties of LDPE, sisal fiber, and coir fiber are given Tables I, II, and III, respectively. Low

density polyethylene (LDPE, Indothene 16 MA400) was procured from M/s. Indian Petrochemical Corporation Ltd., Baroda. Sisal fibers (Agave-Vera Cruz) was provided by a local extraction unit in Marthandam, Tamil Nadu. Coir fiber was supplied by the local processing unit in Kollam, Kerala. Chopped glass fiber (E glass) was supplied by CEAT Ltd., Hyderabad. Carbon black (HAF-N 330) was supplied by Philip's Carbon Black Ltd., Durgapur, India. A solution mixing technique using toluene as the solvent at high temperature was adopted for the preparation of composites as reported by Joseph et al.¹² and George et al.¹³ The composites were prepared by injection molding. Test samples of 2 mm thickness and 10.2 mm diameter were used for testing. The samples were coated with silver paint on the two sides. Copper wires were fixed as electrodes on either sides. The measurements were done at room temperature by varying the frequencies (5 Hz–

Table II Properties of Sisal Fiber

Diameter of Fiber (μm)	Density (kg/m ³)	Cellulose Content (%)	Hemicellulose Content (%)	Lignin Content (%)	Moisture Content at 65% rh and Room Temperature (%)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)
100–300	1450	66–72	12	14–10	11	400–700	9–20	5–14

Table III Properties of Coir Fiber

Diameter of Fiber (μm)	Density (kg/m ³)	Cellulose Content (%)	Hemicellulose Content (%)	Lignin Content (%)	Moisture Content at 65% rh and Room Temperature (%)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)
100–460	1150	32–43	0.15–0.25	40–45	10–12	131–175	4–6	15–40

Table IV Dielectric Constants of LDPE Samples Prepared by Two Different Methods for Various Frequencies

Frequency (Hz)	$10^{1.5}$	10^2	$10^{2.5}$	10^3	$10^{3.5}$	10^4	$10^{4.5}$	10^5	$10^{5.5}$	10^6	$10^{6.5}$	10^7
Injection molded virgin LDPE	4.1	4.1	3.3	3.3	3.3	3.2	3.3	3.3	3.3	2.5	2.3	2.0
LDPE prepared by solution mixing followed by injection molding	4.1	3.3	3.3	3.2	3.2	3.2	3.2	3.2	3.2	2.4	2.2	2.0

13 MHz) using a 4192 Impedance Analyzer (Hewlett-Packard, U.S.A.).

Transcrystallinity studies were done as follows.

A few granules (five to seven) of LDPE were placed on a clean glass slide. One or two fibers were placed over it and then covered with a cover glass. The

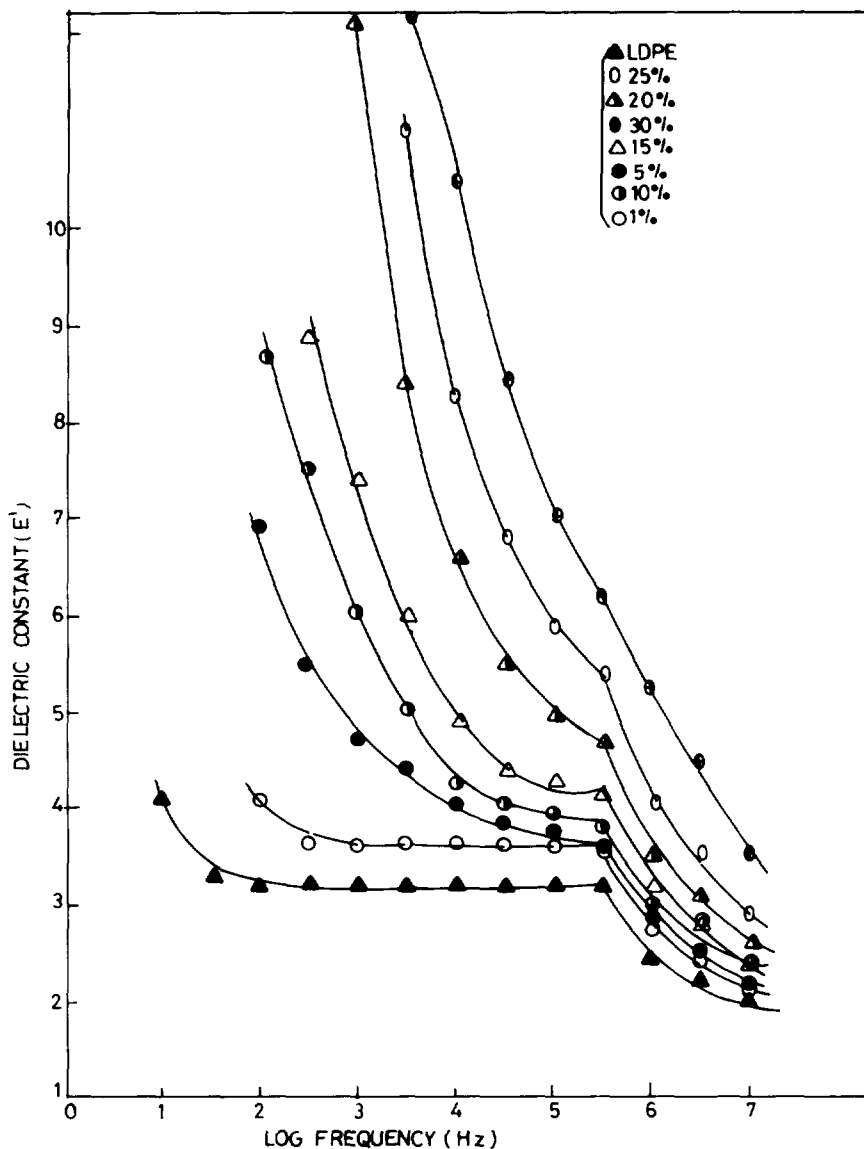


Figure 1 Ambient dielectric constant versus logarithm of frequency for different sisal fiber loadings. Fiber length = 6 mm.

total system was heated above the melting point of LDPE ($\sim 125^{\circ}\text{C}$) in a hot stage. The sample was kept at that temperature for about 10 min. The temperature was brought down to the crystallization temperature of LDPE ($\sim 100^{\circ}\text{C}$). The optical photomicrographs were taken after an annealing period of 12 h.

RESULTS AND DISCUSSION

The properties of composites usually respond to processing conditions.¹⁴ Neat LDPE samples were

prepared by direct injection molding. Samples were also prepared by solution mixing in toluene followed by injection molding. The dielectric constants of the two PE samples prepared by the two techniques are given in Table IV. It is seen that the samples prepared by the two different techniques show almost the same values of dielectric constant. In fact, the variation is less than 3%. This suggests that solution mixing does not adversely affect the dielectric properties of the system.

Figure 1 shows changes in values of dielectric constants of sisal-LDPE composites with the log-

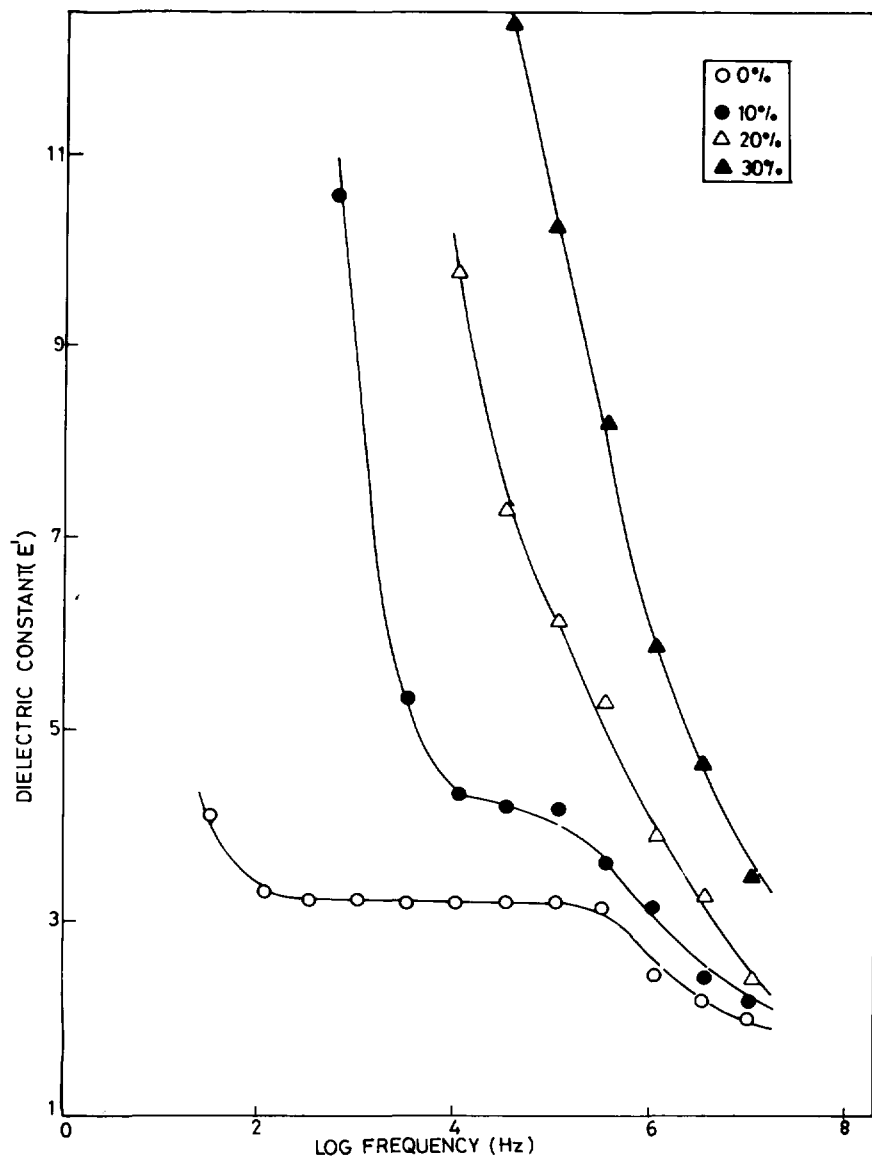


Figure 2 Ambient dielectric constant versus logarithm of frequency for different coir fiber loadings. Fiber length = 6 mm.

arithm of frequency for different fiber loading (0, 1, 5, 10, 15, 20, 25, and 30%). Figure 2 shows changes in values of dielectric constants of coir-LDPE composites with the logarithm of frequency for different fiber loading (0, 10, 20, and 30%). In both cases, the dielectric constant increases with fiber content over the entire range of frequencies. The increase is higher for low frequencies, lower for medium frequencies, and very small for high frequencies. This behavior can be further understood from Figures 3 and 4, which show the change in dielectric constant values with fiber loading for high, low, and inter-

mediate frequencies ($10^{2.5}$, 10^5 and 10^7 Hz) for sisal-LDPE and coir-LDPE composites, respectively. The slope of the straight line curves decreases with an increase in frequency. The dielectric constant values of any polymeric material can be explained by considering the contributions of interfacial, dipole, atomic, and electronic polarizations. Since LDPE is a nonpolar molecule, it has only atomic and electronic polarizations, which are instantaneous; so there should be no variation in the dielectric constants with frequency. The dielectric constants of LDPE are almost same over a wide range of fre-

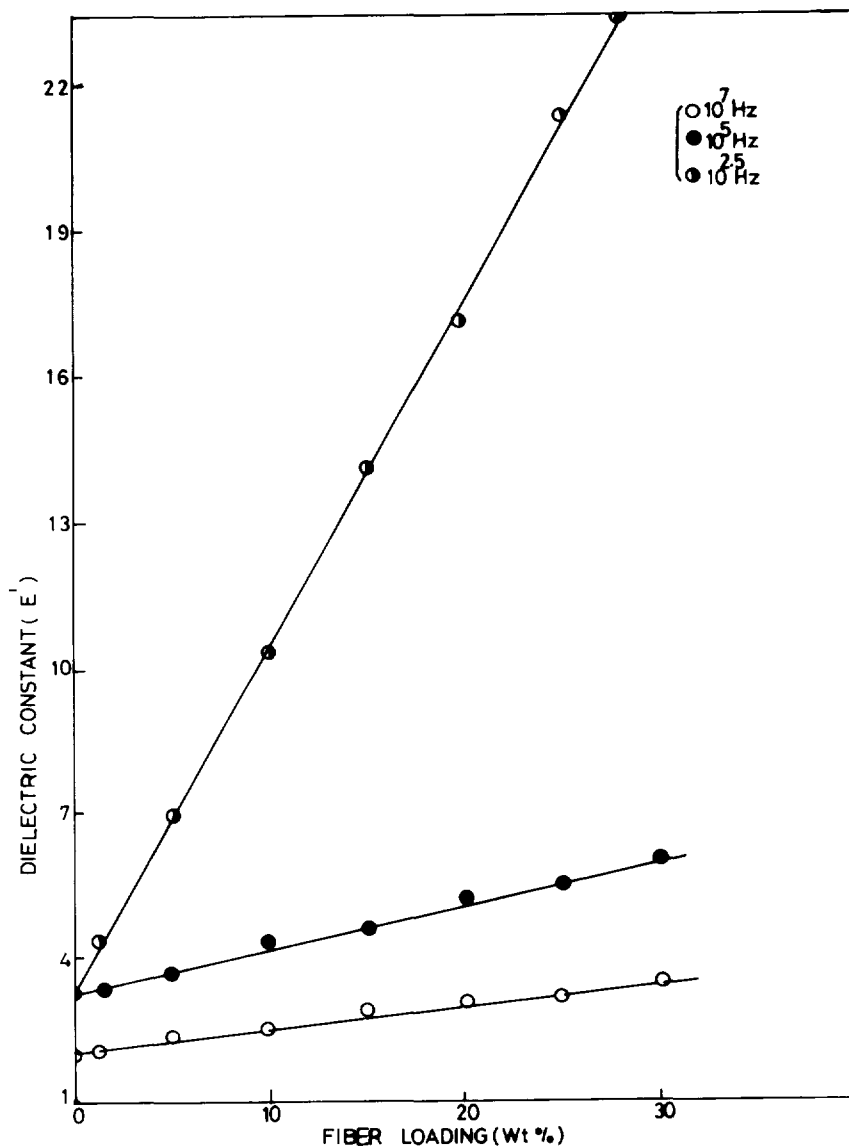


Figure 3 Ambient dielectric constant versus sisal fiber loading for different frequencies. Fiber length = 6 mm.

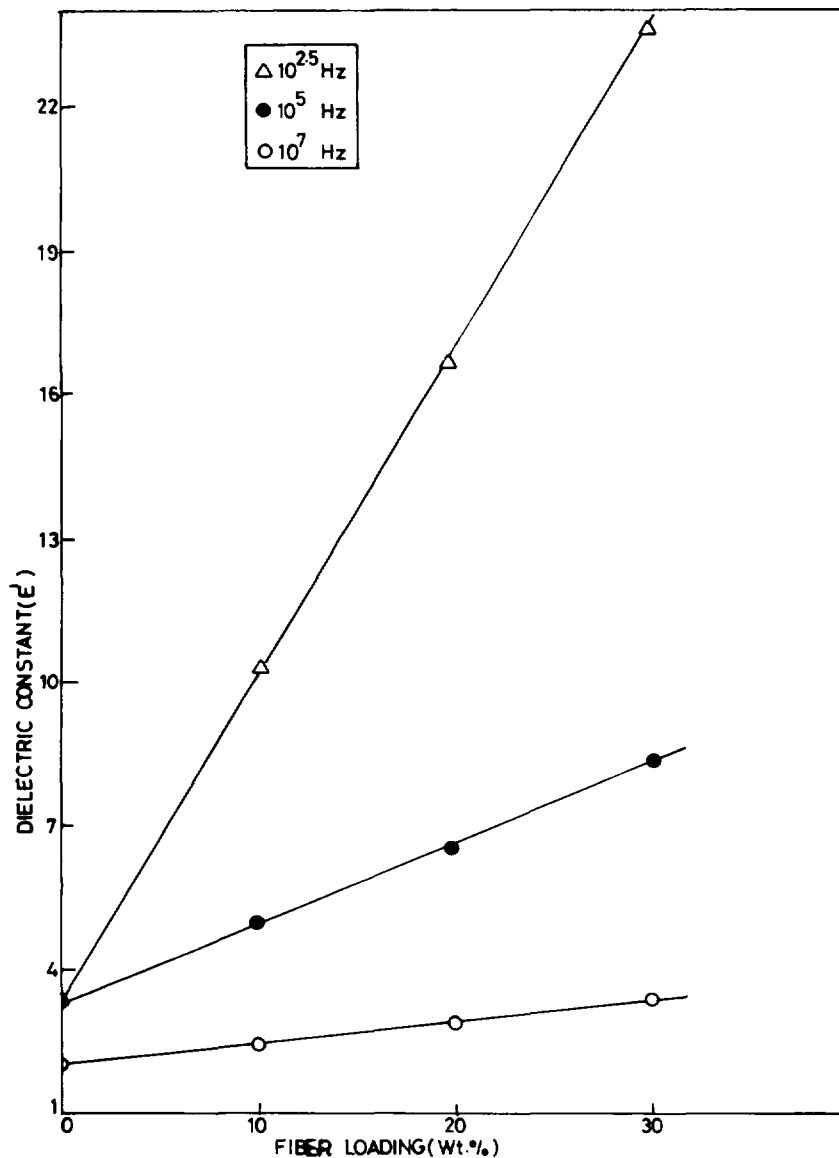


Figure 4 Ambient dielectric constant versus coir fiber loading for different frequencies. Fiber length = 6 mm.

quencies in the curve but are slightly different at very low frequencies and very high frequencies. This can be explained by assuming interfacial polarization. Interfacial polarizations are the result of heterogeneity. Traces of impurities present in LDPE are responsible for its structural inhomogeneities. The interfacial polarization usually decreases with increasing frequency. The neat LDPE curves in Figures 1 and 2 are typical of unfilled nonpolar polymers. The increase of dielectric constant with increase in fiber content can be explained by consid-

ering the chemical nature of sisal fiber and also by the heterogeneous nature of the composite (Figs. 1 and 2). Sisal fibers contain 66–72% cellulose, 12% hemicellulose, 14–10% lignin, and 11% moisture. Coir fibers contain 32–43% cellulose, 0.15–0.25% hemicellulose, 40–45% lignin, and 10–12% moisture. The dielectric constant of the polymer depends upon the polarizability of the molecules. If there is greater polarizability of the molecules, the dielectric constant of the material will be higher. The overall polarizability of a molecule is the sum of electronic,

atomic, and orientation polarizations. Of these, atomic and electronic polarizations are instantaneous polarization components. The effects of the instantaneous polarizations are seen only at very high frequencies. The dipole or orientation polarization is due to the presence of polar groups in the fibers. The degradation of cellulose during the processing affects the properties of the cellulose containing composites. Changes in the chemical structure of cellulose and possibly oxidation of the polyethylene matrix both contribute to the characteristic properties of the composites. Since the composite is heterogeneous, the interfacial polarization also

exists. Interfacial polarization influences the low frequency dielectric properties. The effect of orientation polarization and interfacial polarization will depend upon the concentration of fillers. Hence, the dielectric constant values increase with the concentration of fibers in all the frequencies, especially at low frequencies. The frequency dependence of dielectric constant is due to the fact that at low frequencies complete orientation of the molecule is possible. But at medium frequencies, there is only little time for the orientation. Orientation of the molecules is not possible at all at very high frequencies. Frequency dependence of dielectric con-

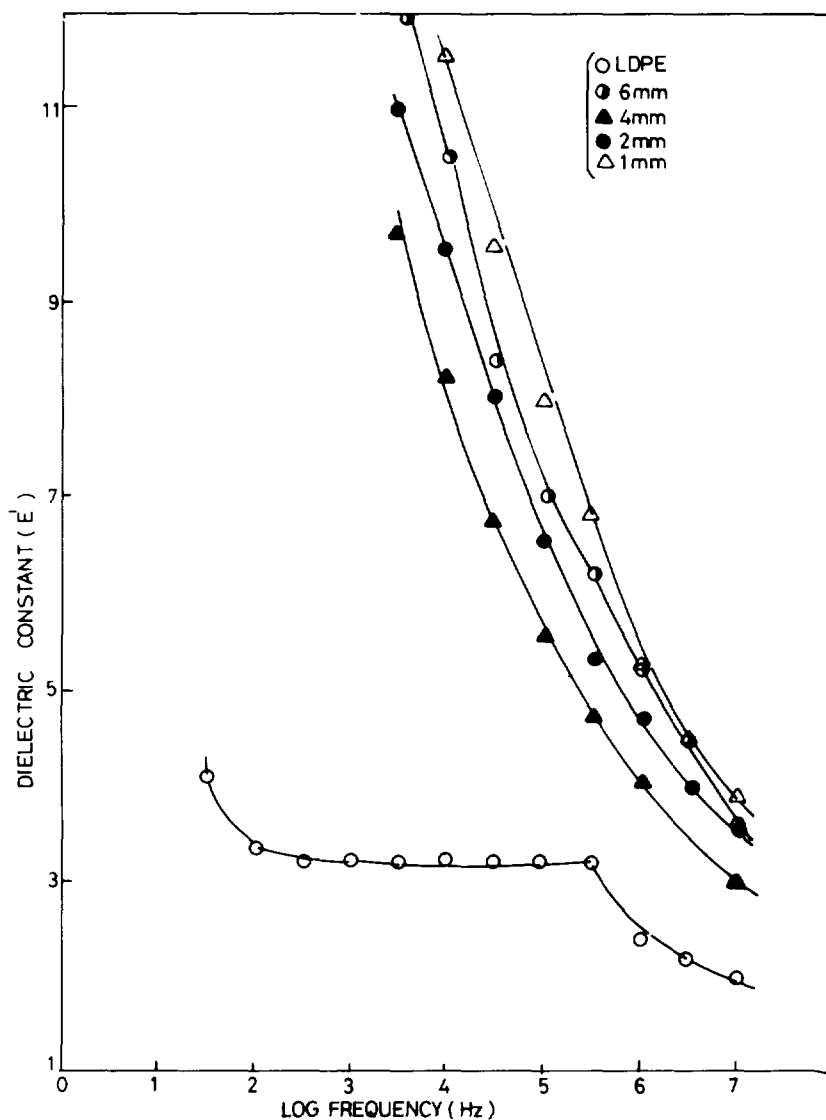


Figure 5 Ambient dielectric constant versus logarithm of frequency for different fiber lengths. Fiber loading = 30%.

stant due to interfacial polarization is similar to those corresponding to dipole polarization.

Figure 5 shows the dielectric constant values as a function of logarithm of frequency for different fiber lengths of sisal at 30% fiber loading. For 1 mm fiber length, the values are maximum; then the values decrease as the fiber length increases, showing a minimum at 4 mm fiber length. Figure 6 shows the variation of dielectric constant values with an increase of fiber length of sisal at three different frequencies (10^6 , 10^5 , and 10^4 Hz). The dielectric constant values first decrease, reach a minimum at 4 mm fiber length, and then increase. These curves can be explained in terms of interfacial polarization.

At constant fiber loading, the number of interfaces per unit volume is greater for 1 mm fiber length. Hence, the contribution of interfacial polarization is maximum, and, therefore, the dielectric constant values are maximum for 1 mm fiber length. The number of interfaces decreases with the increase of fiber length. However, when the fiber length is increased to 6 mm, the fibers undergo bending and curling. This results in an increase in the number of interfaces. Hence, the dielectric constant values again increase slightly.

Plots of reciprocal of dielectric constant ($1/E'_c$) against the volume fraction of the filler (V_1), according to Figure 7 (which describes the law of har-

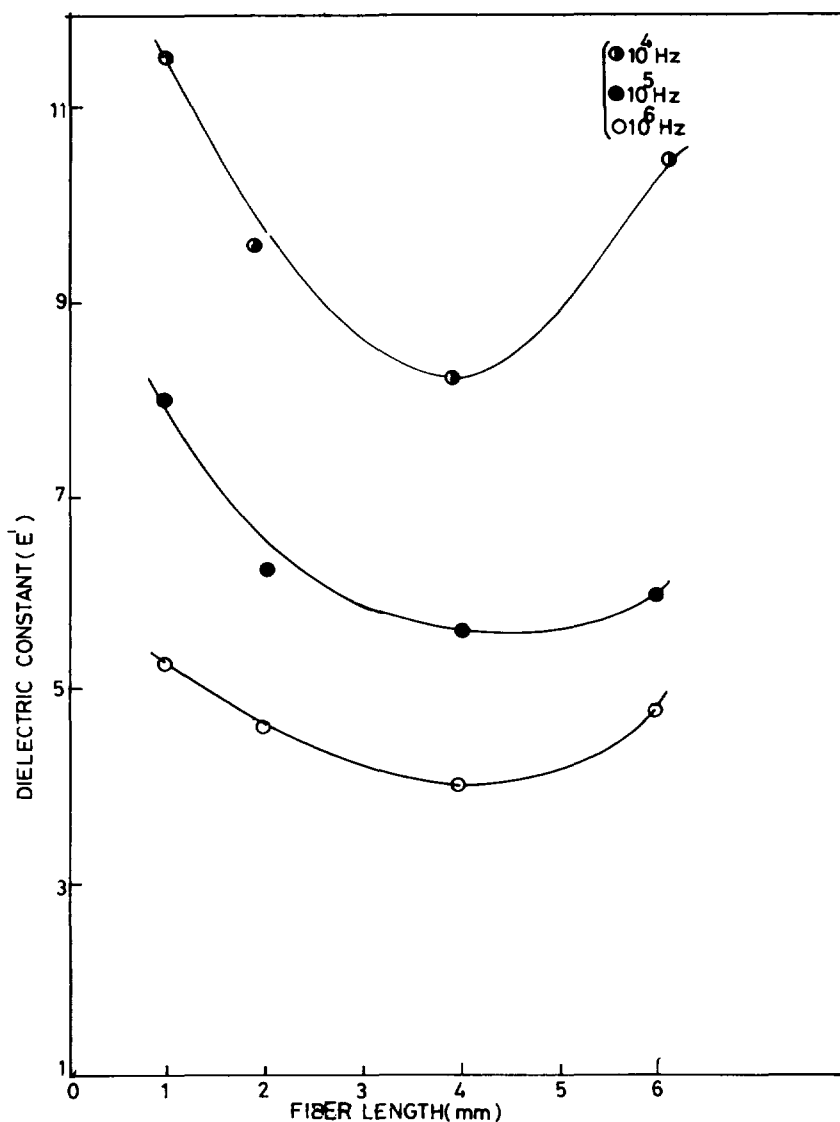


Figure 6 Ambient dielectric constant versus fiber length (mm) for different frequencies. Fiber loading = 30%.

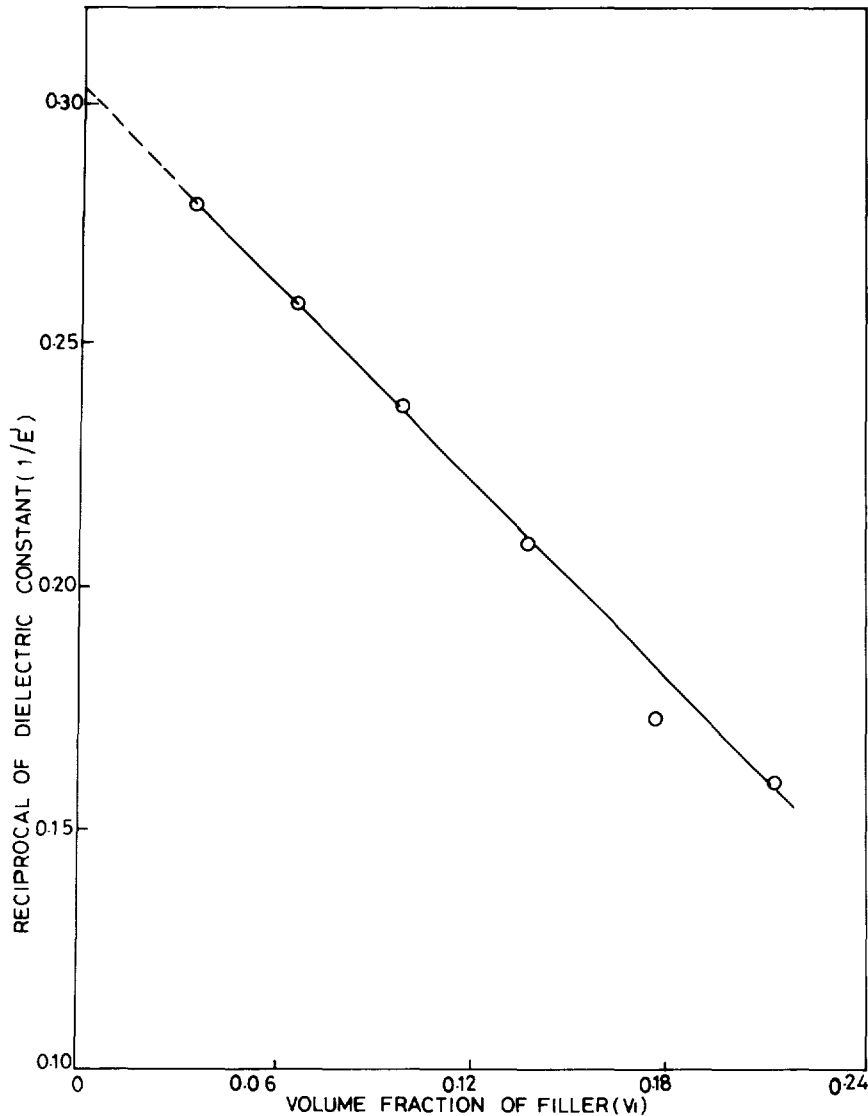


Figure 7 Plot of reciprocal of dielectric constant as a function of volume fraction of sisal fiber at a frequency of $10^{5.5}$.

monic mixture), are linear for sisal fiber-LDPE composites. Extrapolation of the plot to $V_1 = 0$ yields $1/E'_c = 1/E'_2$ where E'_2 is the dielectric constant of LDPE matrix at $f = 10^{5.5}$ Hz. The graphically obtained E'_2 value is in close agreement with the experimentally obtained value (the graphically obtained E'_2 , value = 3.3; the experimentally observed E'_2 value = 3.2).

Applicability of the Clausius-Mossotti Equation⁶ (Fig. 8) for mixture of dielectrics for sisal fiber-LDPE composites is also checked. A linear plot is obtained when specific polarization is plotted against volume fraction of the filler (V_1). The dielectric

constant E'_2 for LDPE can be theoretically obtained from the intercept of this plot at $V_1 = 0$. The graphical value for E'_2 is 3.26, while the corresponding experimental value is found to be 3.20.

Figure 9 shows changes in values of dielectric constants of glass-LDPE composites with logarithm of frequency for different fiber loading (0, 10, 20, and 30%). The dielectric constant values increase with an increase of fiber content in all the frequencies, and the increase is greater at low frequencies. Figure 10 shows the changes in dielectric constant values of the same composites with fiber loading for high, low, and intermediate frequencies (10^7 , $10^{2.5}$,

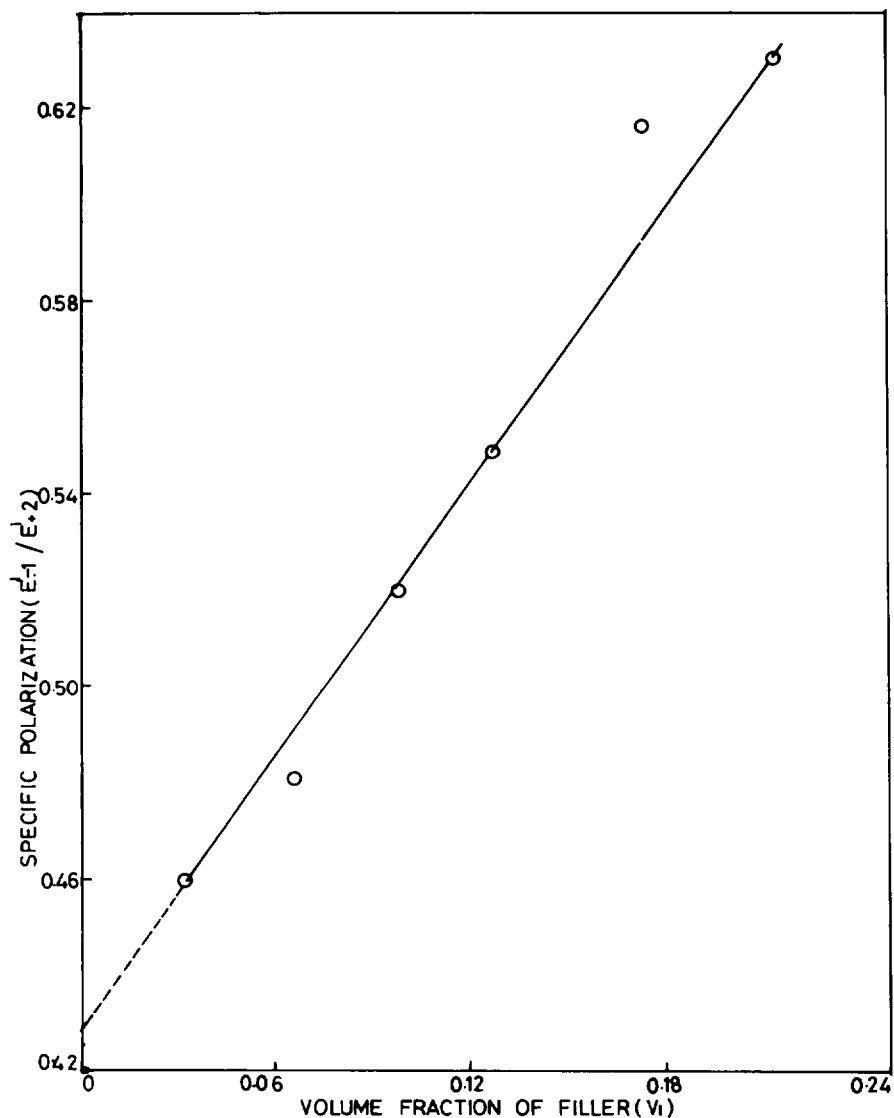


Figure 8 Plot of specific polarization versus volume fraction of sisal fiber loading at a frequency of $10^{5.5}$.

and 10^5 Hz). There is only slight difference in the dielectric constant values at high and medium frequencies, as the fiber loading increases. An appreciable increase in the dielectric constant values with increase of fiber content can be noted at lower frequencies. The above observations can be explained by considering the contributions of interfacial polarization. When structural inhomogeneities between materials of different dielectric constants and conductivities are present, interfacial polarization is expected to occur. The interfacial polarization influences the low frequency dielectric properties be-

cause it usually decreases with increasing frequency.¹⁵

The semilog plots of dielectric constant as a function of frequency for different carbon black loadings are presented in Figure 11. It also presents semilog plots of dielectric constants of 25% sisal-LDPE and 25% sisal +5% carbon black-LDPE composites as a function of frequency. The dielectric constant values increase with increase of carbon black content in all the frequencies, but the increase is lower at high frequencies compared to low and medium frequencies. There is a regular increase for

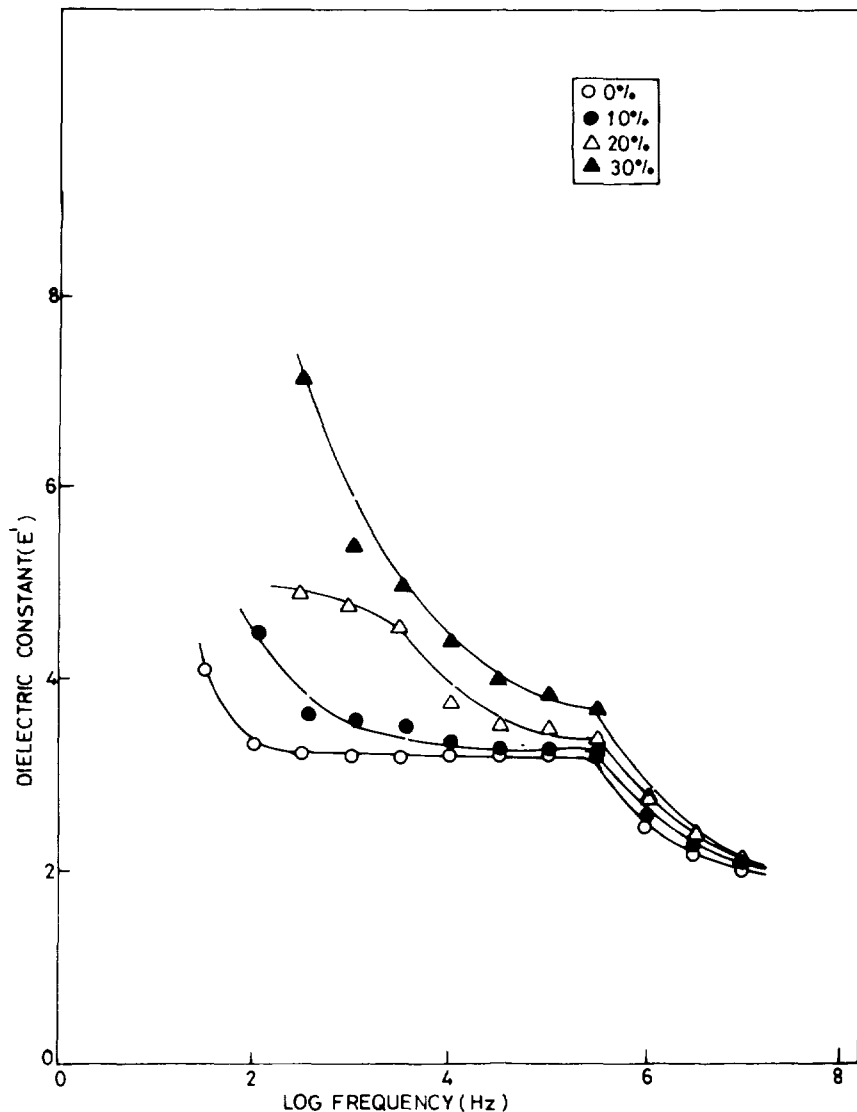


Figure 9 Ambient dielectric constant values versus logarithm of frequency for different glass fibers. Fiber length = 6 mm.

10 and 20% carbon black loaded LDPE composites. At low carbon black loading ($\leq 20\%$), the conductive carbon black particles are separated by insulating polymer layers, and the interfacial polarization becomes extremely large. A conductive network structure of carbon black in LDPE matrix is developed at 30% carbon black loading. However, such a network formation is not possible at 10 and 20% carbon black loading.¹⁶ In the case of 25% sisal with 5% carbon black composite, the high dielectric constant values at low frequencies can be explained by the large interfacial polarization and orientation polarization. The curve of this composite lies in between

20% carbon black and 30% carbon black composites at low frequencies. The dielectric constant values of the 25% sisal composite is lower than that of 25% sisal with 5% carbon black.

The change of dielectric constant with increase of carbon black content at $10^{2.5}$, 10^5 , and 10^7 Hz frequencies can be better understood from Figure 12. The increase of dielectric constant with carbon content is regular up to 20%. However, there is a sudden increase with 30% carbon black. This is evident from the change in slope of the curve beyond 20% loading. In general, polymers filled with high conductive particles can be classified into two systems. One is

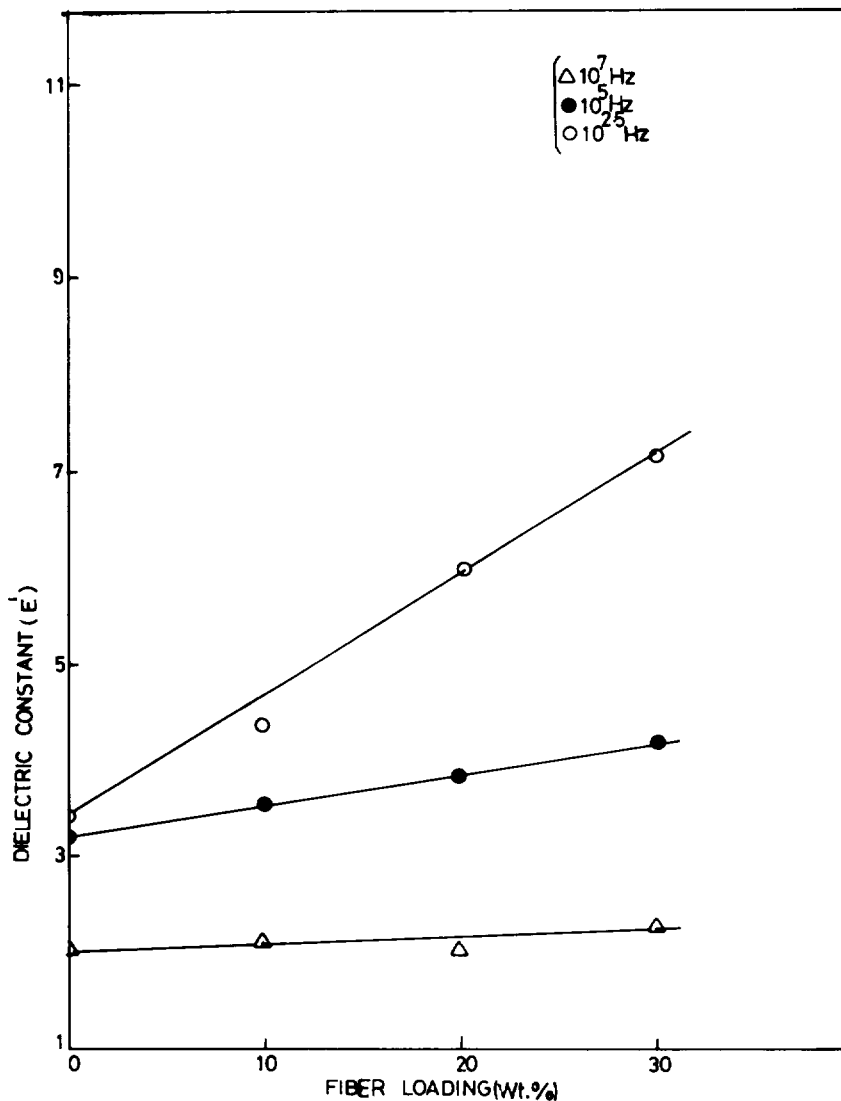


Figure 10 Ambient dielectric constant values versus glass fiber loading for different frequencies. Fiber length = 6 mm.

a system with low content of particles in which dispersed particles hardly touch each other (dispersed system). In the second case with higher filler content, conductive chains are formed by carbon black network (attached system). Electric conduction system of composites quickly changes from a dispersed system to an attached system when volume content of conductive particles increases.¹⁷

Figure 13 depicts the change of dielectric constant with frequency for neat LDPE, LDPE with 30% glass, 30% sisal, 30% coir, and 30% carbon black. At higher frequencies, the differences are marginal, except that for carbon black composite. At low frequencies, the dielectric constant varies in the fol-

lowing order: neat LDPE < glass-LDPE < sisal-LDPE < coir-LDPE < carbon black-LDPE. The high dielectric constant of carbon black composites is due to the arrangement of conductive carbon particles in the form of a conductive network structure and also due to the high interfacial polarization. The higher dielectric constants of lignocellulosic fiber composites as compared to glass fiber composites due to the presence of polar groups. Hence, orientation polarization also acts in addition to interfacial polarization. The glass-LDPE composites have a greater value of dielectric constants than that of LDPE due to small interfacial polarization.¹⁸

The semilog plots of volume resistivity as a func-

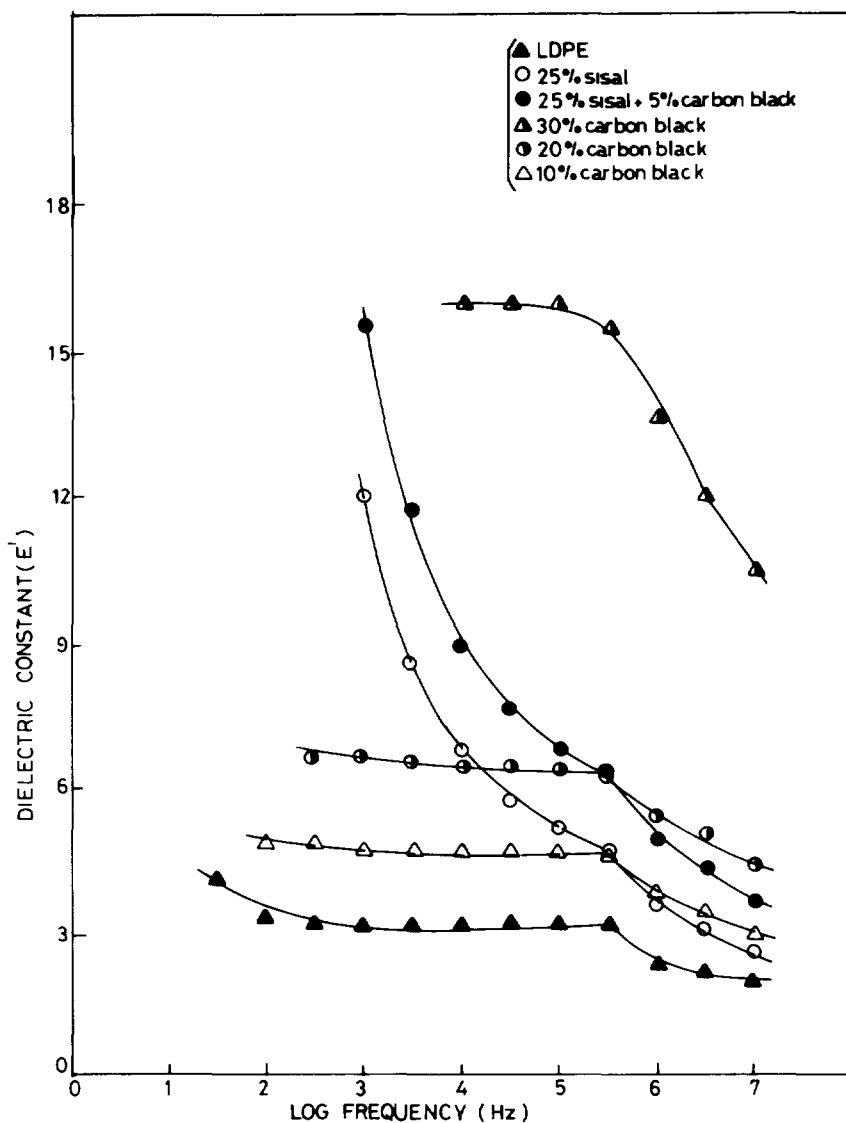


Figure 11 Ambient dielectric constant versus logarithm of frequency for different carbon black loadings.

tion of frequency for different sisal fiber loadings are presented in Figure 14 (0, 5, 15, and 30%). Volume resistivity decreases with an increase of frequency, i.e., electric conduction increases with fiber loading. It is known that in any polymeric material, the current flows mainly through the crystalline regions, and the noncrystalline regions allow current to pass through mainly due to the presence of moisture. Since sisal fiber is lignocellulosic, it absorbs moisture. The presence of moisture and impurity content increases the conduction. A fiber embedded in a thermoplastic melt can act as a nucleant for the growth of spherulites. If there are many nucleation

sites along a fiber surface, the resulting spherulite growth will be restricted in the lateral direction so that a columnar layer known as transcrystallinity will develop and enclose the fiber (Figs. 15 and 16). This also increases electrical conduction.¹⁹⁻²¹

Figure 17 indicates the variation of volume resistivity with the logarithm of frequency for different carbon black content (0, 10, 20, and 30%) and 25% sisal with 5% carbon black. Electrical conduction increases with the increase of frequency and carbon content. The volume resistivity of 25% sisal-LDPE composite containing 5% carbon black is similar to that of 10% carbon black-LDPE composite. The

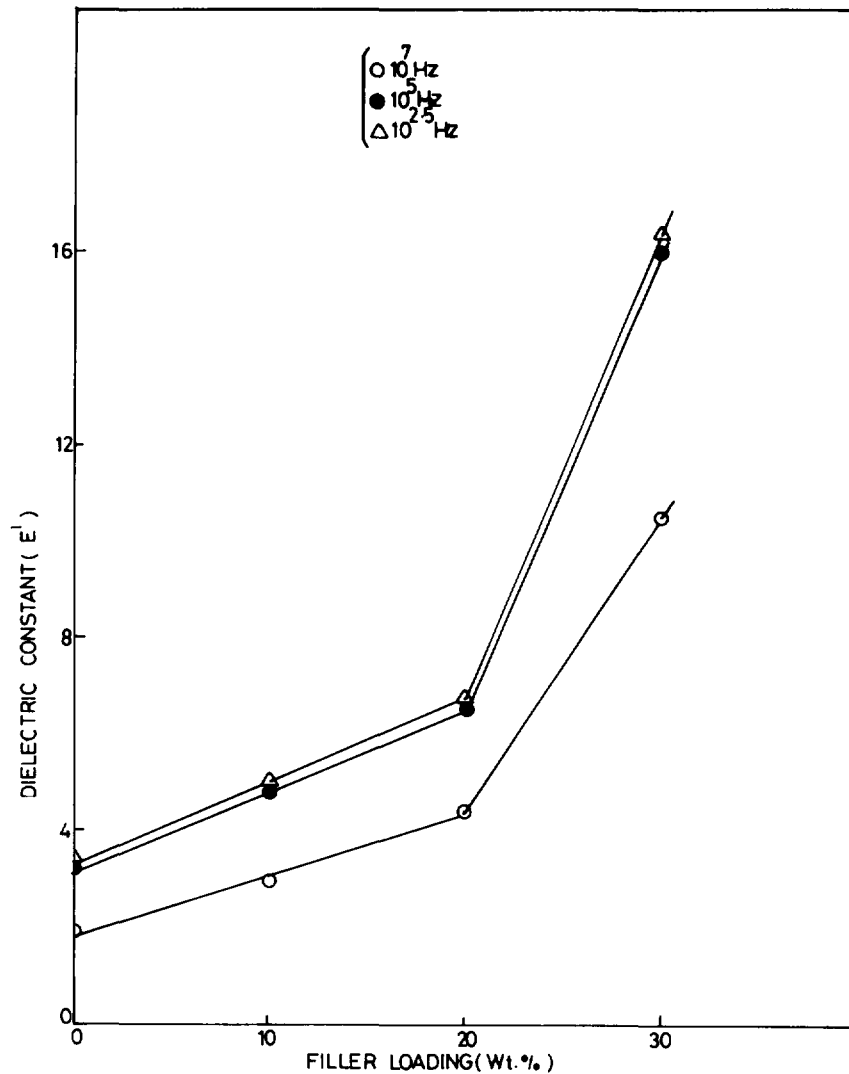


Figure 12 Ambient dielectric constant versus filler loading for different frequencies.

basic and generally accepted concepts of conductivity for polymer compositions containing carbon black are based on the fact that carbon black forms developed chain or network structures. The degree of conductivity depends on the nature of these chain structures. Volume resistivity of a carbon black depends not only as the electrical properties of the resulting chain structures, but also on the number of these within the volume.

Figure 18 shows the loss factor (E'') as a function of logarithm of frequency for different coir fiber loading. The loss factor increases with fiber content in the frequencies ranging from 10^5 to 10^7 Hz. A strong relaxation is observed with a peak at a frequency of $10^{5.5}$ Hz. The peak is found for all the

compositions at the same region. The position of peaks are found to be identical both for filled and unfilled systems. These loss peaks might be attributed to the dipolar relaxation of the polymer matrix. The addition of fiber does not affect the relaxation mechanism but enhances the relaxation magnitude at each frequency. The increase of loss factor due to increased concentration of fiber indicates increased polarization by the introduction of polar groups. LDPE got a less intense peak at the same frequency, due to slight oxidation of it forming traces of polar compounds.

Figure 19 shows the change of loss factor (E'') with the logarithm of frequency for different carbon black loading. The figure also presents the change

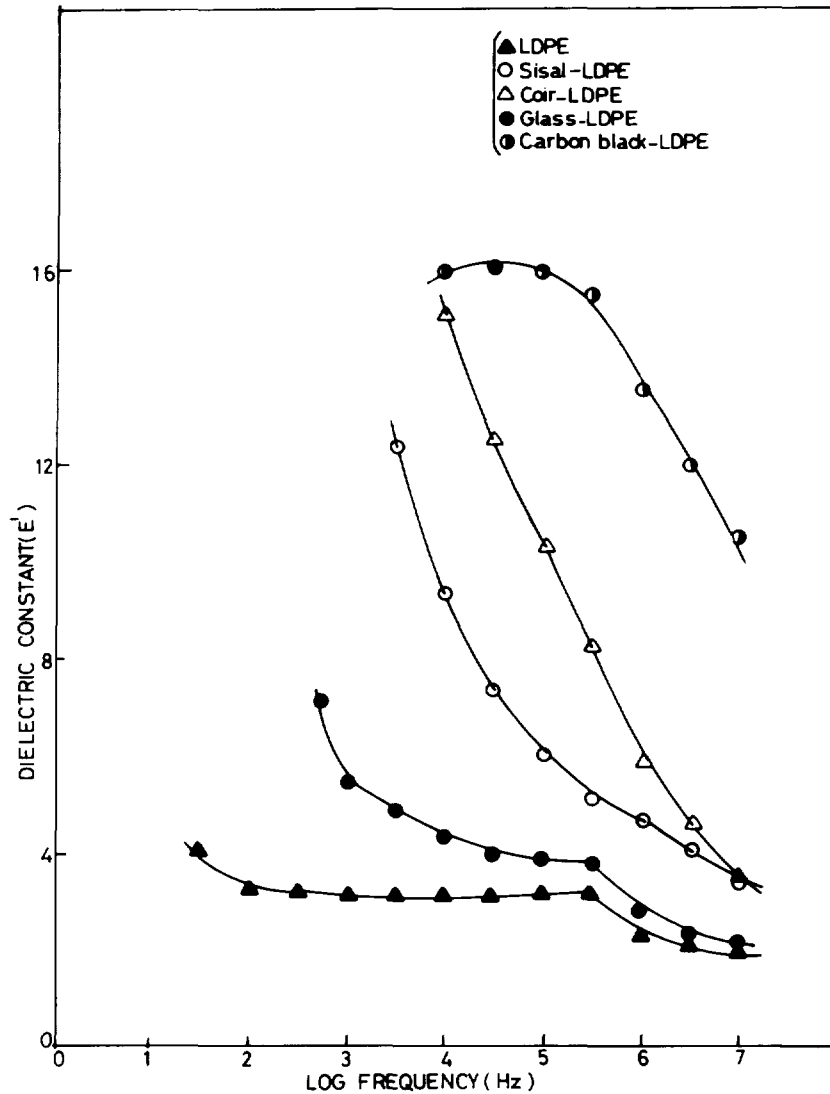


Figure 13 Ambient dielectric constant versus logarithm of frequency for different fillers. Filler loading = 30%; fiber length = 6 mm.

of loss index (E'') of 25% sisal/5% carbon black-LDPE composite with the logarithm of frequency. The loss factor increases with carbon black content in all the frequencies ranging from 10^5 to 10^7 Hz. A peak is found for all the curves at around a frequency of $10^{5.5}$ Hz due to strong relaxation.

These loss peaks might be attributed to dielectric relaxation involving interfacial polarization. The increase of loss factor due to increased concentration of carbon black indicates increased polarization. The position of the curve for 25% sisal/5% carbon black-LDPE composite is above 20% carbon black LDPE composite. This is due to relaxation involving orientation polarization and interfacial polarization.

With 30% carbon black-LDPE composite, the loss factor increases to a greater extent compared to other compositions. This is due to the formation of conductive chains and high interfacial polarization associated with 30% carbon black LDPE composite.^{6,15}

CONCLUSION

The dielectric constant, volume resistivity, and dielectric loss factor of natural-fiber-reinforced LDPE composites have been studied as a function of frequency, fiber content, and fiber length. The dielectric

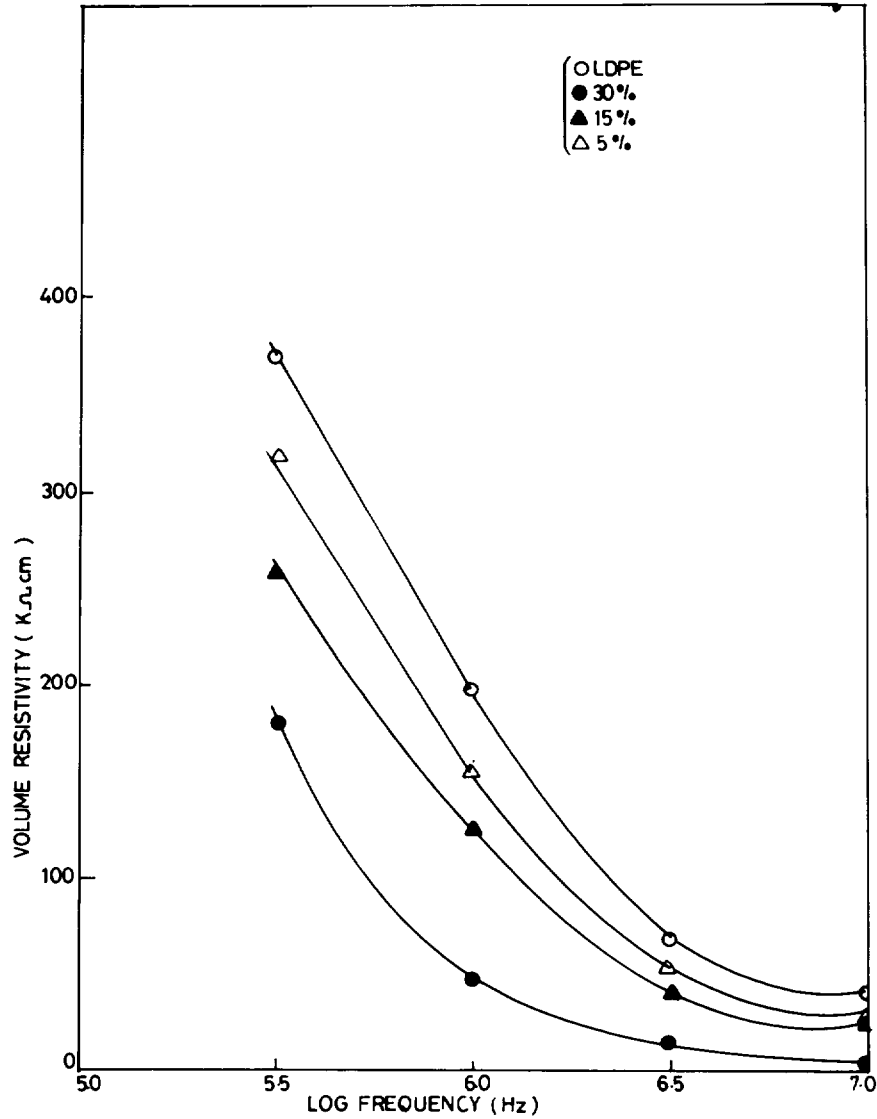


Figure 14 Ambient volume resistivity versus logarithm of frequency for different sisal fiber loadings. Fiber length = 6 mm.



Figure 15 Optical photomicrograph of sisal-LDPE system showing transcrystallinity ($\times 100$ times).



Figure 16 Optical photomicrograph of sisal-LDPE system showing a magnified view of transcrystallinity ($\times 100$ times).

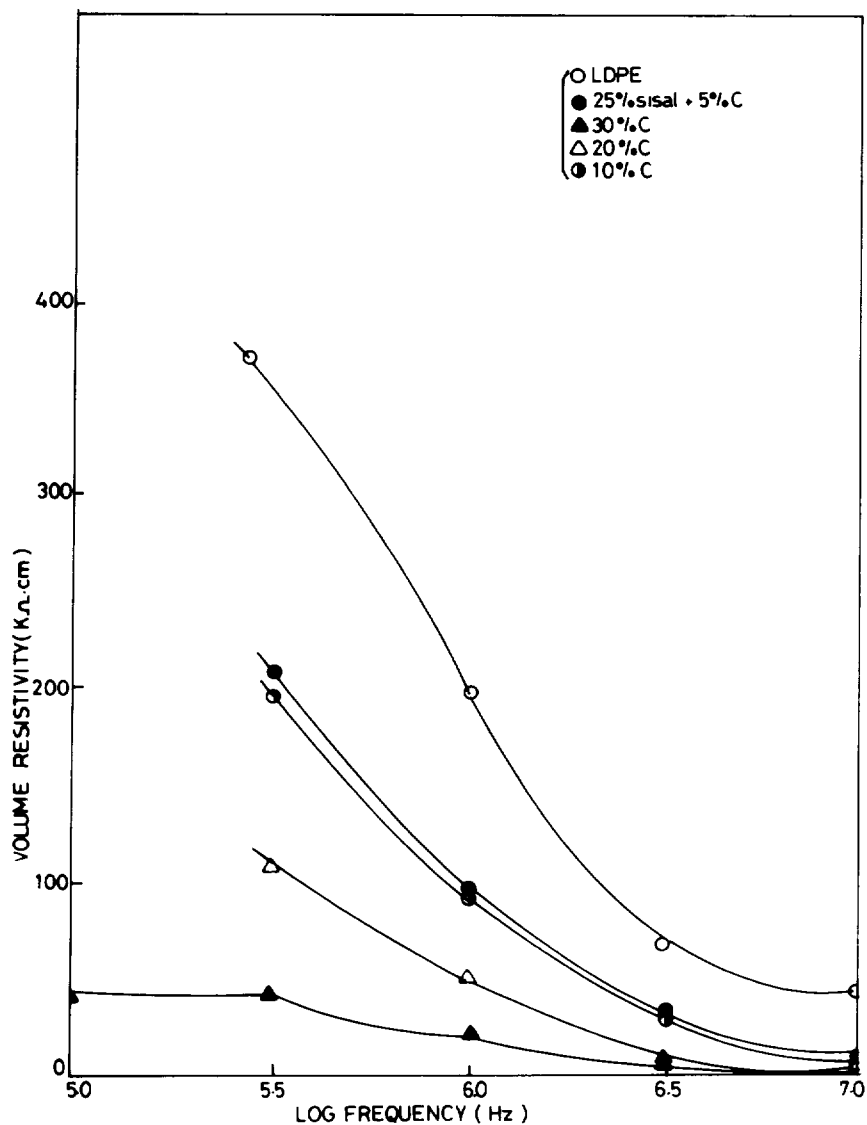


Figure 17 Ambient volume resistivity versus logarithm of frequency for different carbon black loadings.

constants of sisal-LDPE and coir-LDPE progressively increase with increase of fiber loading in all the frequencies ranging from 1 to 10^7 Hz. It is also noted that dielectric constant decreases with increase of fiber length and frequency. Maximum dielectric constant values are obtained at low frequencies. Sisal-LDPE composites of 1 mm fiber length and 30% fiber loading got highest values of dielectric constants in all the observed frequencies. The volume resistivity values were found to be decreased with increase of frequency and fiber loading;

i.e., the electric conductivity of composites were greater than that of neat LDPE. It is observed that dielectric loss factor of coir-LDPE composites increases with increase in fiber concentration. The electric properties of natural fiber-reinforced LDPE composites were compared with that of glass-LDPE and carbon black-LDPE composites. The same trend in the electrical properties were observed for glass-LDPE composites. The changes of dielectric constants of glass-LDPE composites with frequency and fiber loading were small compared to sisal and

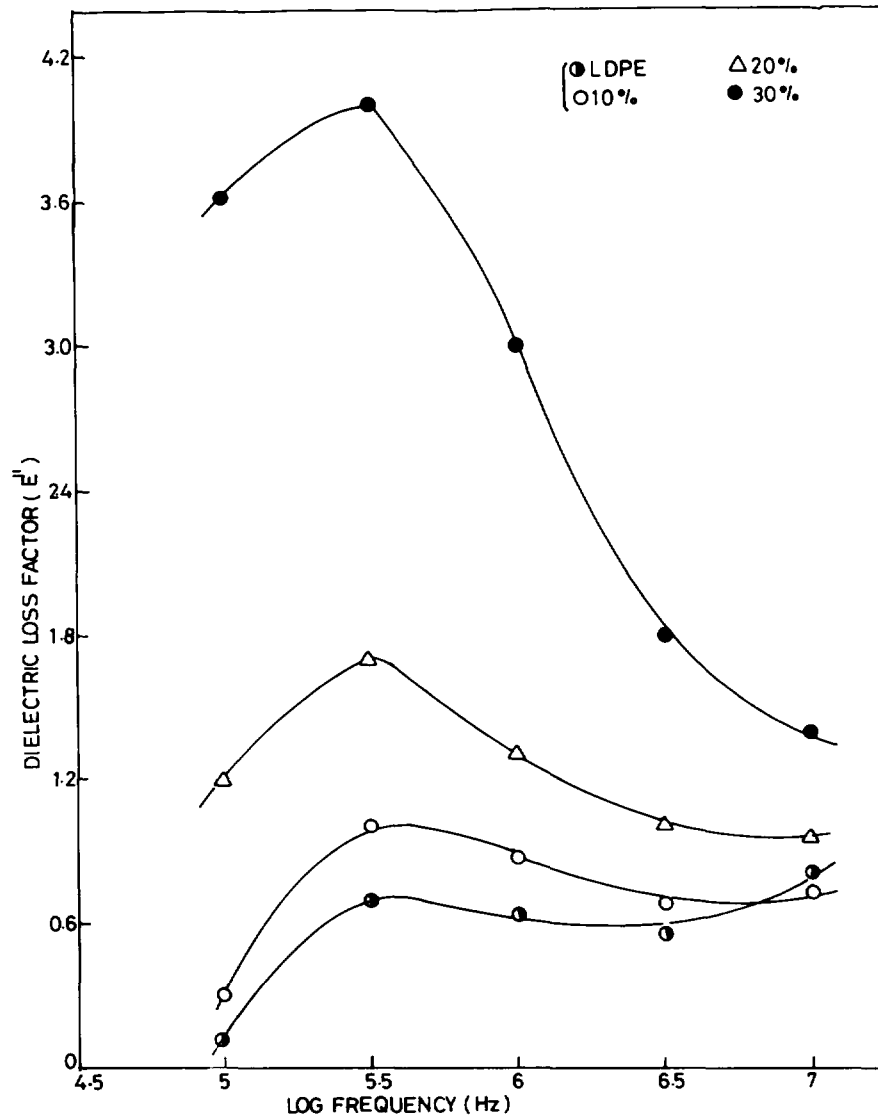


Figure 18 Ambient dielectric loss factor versus logarithm of frequency for different coir fiber loadings. Fiber length = 6 mm.

coir fiber LDPE composites due to low interfacial polarization. The dielectric constant, dielectric loss index, and electrical conductivity were found to increase with carbon black content. There is a sudden increase in the conductivity of carbon black-LDPE composites with a 30% carbon black content. This is due to the formation of conductive network of carbon particles. At low frequencies, the dielectric constant varies in the following order: neat LDPE < glass-LDPE < sisal-LDPE < coir-LDPE < car-

bon black-LDPE. A 25% sisal-LDPE composite containing 5% carbon black was found to have high dielectric constant, high dielectric loss factor, and low volume resistivity. The electrical conductivity of hydrophobic LDPE can be improved by mixing it with hydrophilic lignocellulosic fibers and conductive carbon black. It is also proven that the solution mixing technique has no adverse effects. Finally, it is important to mention that a 25% sisal-LDPE composite containing 5% carbon black can

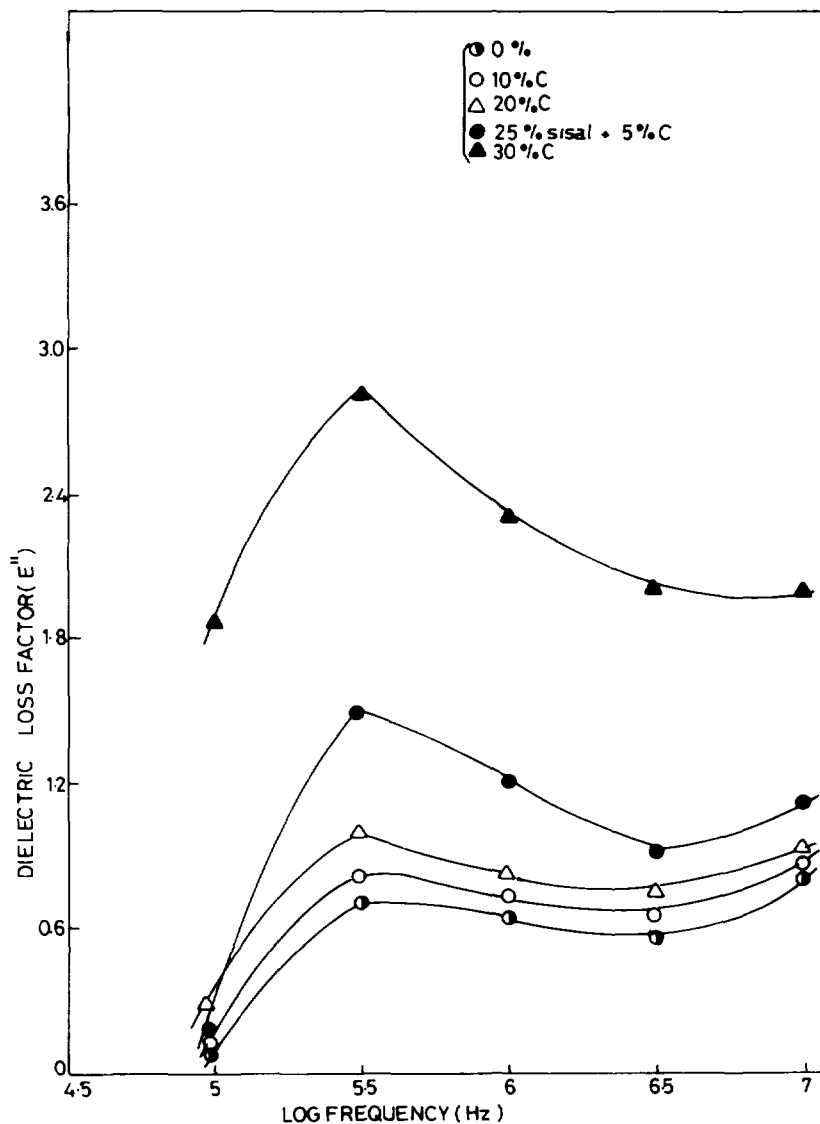


Figure 19 Ambient dielectric loss factor versus logarithm of frequency for different carbon black loadings.

be used in antistatic applications to dissipate static charges. Studies in these directions are in progress in this laboratory.

We thank Dr. A. D. Damodaran, Director, and Dr. Gopakumar Warriar, Scientist, Regional Research Laboratory, Thiruvananthapuram, for their valuable help in measuring the electrical properties of composites using the LF-Impedance Analyzer. Augustine Paul is thankful to U.G.C. for providing teacher fellowship.

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Received July 26, 1995

Accepted June 10, 1996

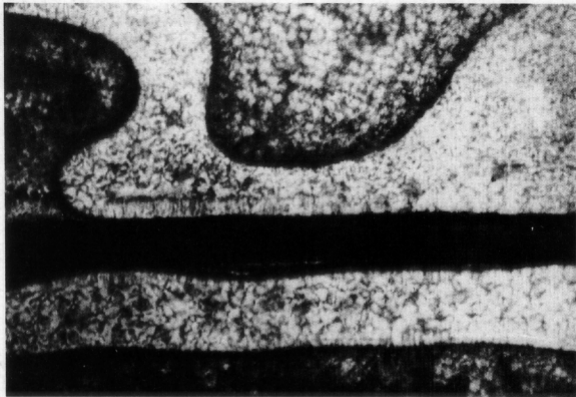


Figure 15 Optical photomicrograph of sisal-LDPE system showing transcrystallinity ($\times 100$ times).

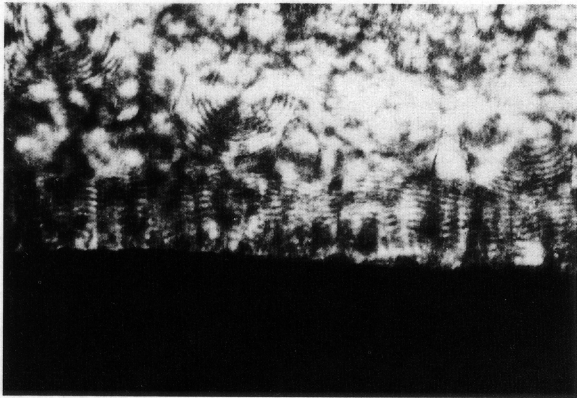


Figure 16 Optical photomicrograph of sisal-LDPE system showing a magnified view of transcrySTALLINITY ($\times 100$ times).