

# Studies of Electrical and Mechanical Properties of Poly(vinyl chloride) Mixed with Electrically Conductive Additives

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**ABSTRACT:** Different samples of poly(vinyl chloride) (PVC) compositions were formulated from PVC, a polar plasticizer such as dioctylphthalate (DOP), and variable proportions of electrically conductive additives such as fast extrusion furnace (FEF) carbon black (CB), poly(vinylpyridine) (PVP), or polyacrylonitrile (PAN). Epoxidized soybean oil was added as a heat stabilizer. Samples of the PVC–CB system were also prepared by dispersing different concentrations of CB into the PVC matrix. The electrical studies showed that the addition of CB to the PVC–DOP system produces a plasticized PVC with high electrical conductivity

whereas the compounding of PVC with CB produces a sample with much higher electrical conductivity. The effect of the structure of PVP and PAN on the electrical and mechanical properties of the PVC–DOP system was also studied to obtain a semiconductive plasticized PVC with good mechanical properties. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 91: 1590–1598, 2004

**Key words:** poly(vinyl chloride); electrical and mechanical properties; activation energy

## INTRODUCTION

Many polymers in their pure state are good electrical insulators ( $<10^{-12} \Omega^{-1} \text{cm}^{-1}$ ). However, in some cases there is a demand for a conducting or semiconducting polymer ( $>10^{-1} \Omega^{-1} \text{cm}^{-1}$ ). Conductive polymers offer many advantages compared to classic materials (metals, wood, etc.) such as facility of processing, cost effectiveness, low density compared to metals, preservation from corrosion, low hardness, and varied mechanical properties.

Electrical conductivity in plastics can be provided by incorporation of conductive materials.<sup>1</sup> In practice, electrically conductive fillers such as carbon black (CB), graphite, metallic powders, some metal oxides, and aluminum flakes are of current commercial interest.<sup>2,3</sup> However, CB is the most commonly used filler, because it is much cheaper and lighter than metallic particles.<sup>4,5</sup> Naturally, the introduction of metallic materials allows us to envisage the elaboration of polymers possessing the conductive properties of metals and the varied properties of polymers.

Polymers such as polyaniline, polythiophene, and polypyrrole have attracted considerable attention because of their high electrical conductivity. For exam-

ple, conducting polythiophene and polypyrrole blends with polystyrene were prepared and studied by Wan et al.<sup>6</sup> Moreover, Seanor<sup>7</sup> found that when polystyrene is nitrated or sulfonated its conductivity rises from  $10^{-19}$  to  $10^{-5} \Omega^{-1} \text{cm}^{-1}$ .

Conjugated polymers have also been studied with the idea that  $\pi$  electrons that are delocalized along the chains of these polymers could give conductive properties to these systems similar to those of semiconductors. The first conductive polymer was thus realized<sup>8</sup> after success in doping polyacetylene. Since this discovery, many conductive polymers have been developed from several monomers that produce conjugated systems after polymerization. Conductive polymers are mainly synthesized by chemical or electrochemical oxidation of monomers in solutions containing a supporting electrolyte.

The aim of this study was to systematically investigate the effect of adding increasing quantities of fast extrusion furnace (FEF) CB, poly(vinylpyridine) (PVP), or polyacrylonitrile (PAN) on the electrical and mechanical properties of poly(vinyl chloride) (PVC). We attempt to improve these properties and obtain a good electrical conducting sample for electronic and microwave device fabrications and battery electrode applications.

## EXPERIMENTAL

### Preparation of PVP

The PVP was prepared by adding benzoyl peroxide (0.5 g) to VP (monomer, 0.5 mol) dissolved in chloro-

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**TABLE I**  
**GPC Results**

Polymer	$M_n$	$M_w$	Polydispersity
PVP	5829	15,722	2.70
PAN	5639	22,362	3.97

form (200 mL). The reaction mixture was refluxed for 4 h at 60°C. The resulting polymer was separated by adding *n*-hexane, washed with alcohol, and dried in an air oven at 100°C.

**Preparation of PAN**

The PAN was prepared by adding benzoyl peroxide (1.0 g) to AN (monomer, 0.5 mol) dissolved in chloroform (50 mL). The reaction mixture was refluxed for 0.75 h at 60°C. The resulting polymer was precipitated, filtered, washed with alcohol, and then dried in an air oven at 100°C.

**Characterization of PVP and PAN**

PVP and PAN were characterized by determining their number-average molecular weights ( $M_n$ ), weight-average molecular weights ( $M_w$ ), and polydispersity index ( $M_w/M_n$ ) through gel-permeation chromatography (GPC). The high pressure GPC system consisted of a high pressure pump (type 600E multi-solvent delivery system) and four ultra styragel HR chromatographic columns (7.8 mm; i.d. = 300 mm; pore diameters =  $10^5$ ,  $10^4$ , 500, and 100 Å) connected in series and a differential refractometer (type M2410) as the detector. The GPC measurements were carried out in HPLC grade toluene as the mobile phase with a rate of elution of 0.7 mL/min at 40°C. The molecular weights were determined relative to monodisperse polystyrene standards. Calculations were performed

using the Millennium 32 chromatography manager with gel-permeation application software. The results are shown in Table I.

**Formulations of samples**

In the present study, several formulations were obtained by mixing the components in the proportions shown in Table II. The general procedure for the formulation is described as follows. Homogeneous samples were prepared by adding variable proportions of FEF CB or either the prepared PVP (good thermal stability due to nitrogen, which is covalently bonded to the neighboring carbons to form heterocycles) or PAN portionwise at room temperature to a composition containing 100 parts PVC (*K* value = 70, apparent bulk density = 0.3 g/mL, viscosity number = 125 mL/g) and 40 parts dioctylphthalate (DOP) under stirring. The mixtures were then mixed with epoxidized soybean oil as a heat stabilizer to give the required samples (gelation temperature at 175°C for 100 s). The samples thus obtained are denoted by  $X_0, X_1, X_2, \dots, X_{12}$ . These formulations are shown in Table II. Samples of the PVC–CB system were also prepared by dispersing variable proportions of CB into the PVC matrix. The compounding was done by adding the CB to the polymer, mixing at temperatures above the glass-transition temperature ( $T_g$ ), and subjecting the mixture to high shear.<sup>9</sup> The prepared samples are denoted by  $X_0, X_{13}, X_{14}, X_{15}$ , and  $X_{16}$  (Table III).

**Mechanical measurements**

The tensile strength and percent elongation were determined on an Instron testing machine (model 1026) at  $23 \pm 2^\circ\text{C}$  with a crosshead speed of 100 mm/min according to ASTM D 638-77a.<sup>10</sup> The results are given in Tables II and III.

**TABLE II**  
**Formulations and Mechanical Properties of PVC–DOP System Mixed with FEF CB, PVP, and PAN**

	Sample Notation												
	$X_0$	$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	$X_6$	$X_7$	$X_8$	$X_9$	$X_{10}$	$X_{11}$	$X_{12}$
Constituents parts by weight (g)													
PVC	100	100	100	100	100	100	100	100	100	100	100	100	100
DOP	40	40	40	40	40	40	40	40	40	40	40	40	40
Epoxidized soybean oil	3	3	3	3	3	3	3	3	3	3	3	3	3
CB	—	10	20	30	40	—	—	—	—	—	—	—	—
PVP	—	—	—	—	—	10	20	30	40	—	—	—	—
PAN	—	—	—	—	—	—	—	—	—	10	20	30	40
Mechanical properties													
Tensile strength (N/mm <sup>2</sup> )	12.71	13.95	10.18	9.01	6.92	11.10	8.98	8.03	5.01	12.27	10.63	9.52	7.99
Elongation (%)	590	515	350	194	119	455	292	201	158	502	400	285	220

TABLE III  
Formulations and Mechanical Properties of PVC-CB System

	Sample notation				
	X <sub>0</sub>	X <sub>13</sub>	X <sub>14</sub>	X <sub>15</sub>	X <sub>16</sub>
Constituents parts by weight (g)					
PVC	100	100	100	100	100
FEF CB	—	10	20	30	40
Mechanical properties					
Tensile strength (N/mm <sup>2</sup> )	20.67	45.37	26.25	4.95	—
Elongation (%)	10	15	10	5	—

### Dielectric measurements

The permittivity ( $\epsilon'$ ) and dielectric loss ( $\epsilon''$ ) for the denoted samples were measured at frequencies ranging from 100 Hz to 100 kHz. An LCR meter (type AG-4311B) Ando electric LTD with an NFM/5T test cell was used. The capacitance ( $C$ ) and the loss tangent ( $\tan \delta$ ) were obtained directly from the bridge from which  $\epsilon'$  and  $\epsilon''$  were calculated. The samples were in the form of disks with a 58-mm diameter and 3-mm thickness. Calibration of the apparatus was carried out using standard samples (Trolitul, glass, and air) of 3-mm thickness; the accuracy for  $\epsilon'$  was  $\pm 1\%$  and for  $\epsilon''$  was  $\pm 2\%$ . The measurements were carried out at temperatures from 20 to 60°C using an ultrathermostat.

### Electrical conductivity measurements

The electrical conductivity ( $\sigma$ ) of the investigated samples was measured by the application of Ohm's law using the NFM/5T test cell. A power supply unit (GM45161/01) from Philips was used. The potential difference ( $V$ ) between the plates holding the sample and the current ( $I$ ) flowing through it was measured by a multimeter (type URI 1050) from Rohde and Schwarz. The electrical conductivity is calculated using the equation

$$\sigma = \frac{dI}{AV} \text{ (mho m}^{-1}\text{)}$$

where  $d$  is the thickness of the sample (m) and  $A$  is its surface area (m<sup>2</sup>).

## RESULTS AND DISCUSSION

Polymers such as PVC are known to have kinetically rigid chains. On the addition of polar plasticizers such as DOP, they are present as clusters of molecules between clusters or bundles of polymer segments or molecules. As energy is applied to the system, the permittivity rises significantly, indicating that the PVC molecules are no longer held together rigidly but are free to move as if the polymer is rubbery rather than

glassy. Thus, the plasticizer molecules have penetrated the clusters or bundles of PVC molecules and separated the polymeric chains. After heating this mixture, a toughening structure forms on cooling and may result from van der Waals forces, hydrogen bonding, or crystallization of polymer segments. Moreover, with the addition of an electrically conductive additive to a polymer matrix, the specific electrical conductivity of the latter will be increased because of the formation of ionic charges with excitation by heat or light. These charged species become mobile when an electric field is applied; hence, the electrical conductivity in the host polymer increases according to the structure of the investigated additive.

In this investigation, plasticized PVC has been incorporated with FEF CB, PVP, or PAN in different ratios up to 40 parts per hundred parts of resin (phr) PVC in an attempt to improve its electrical properties and make it into a semiconductor with good mechanical properties. The effects of these additives on the

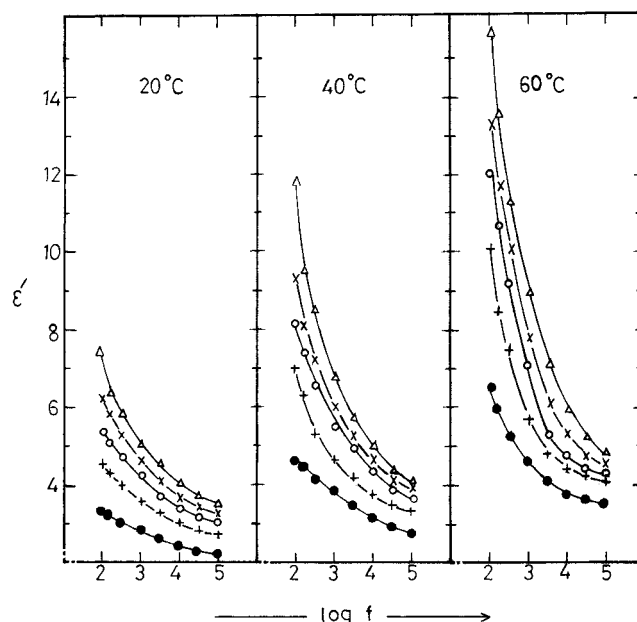
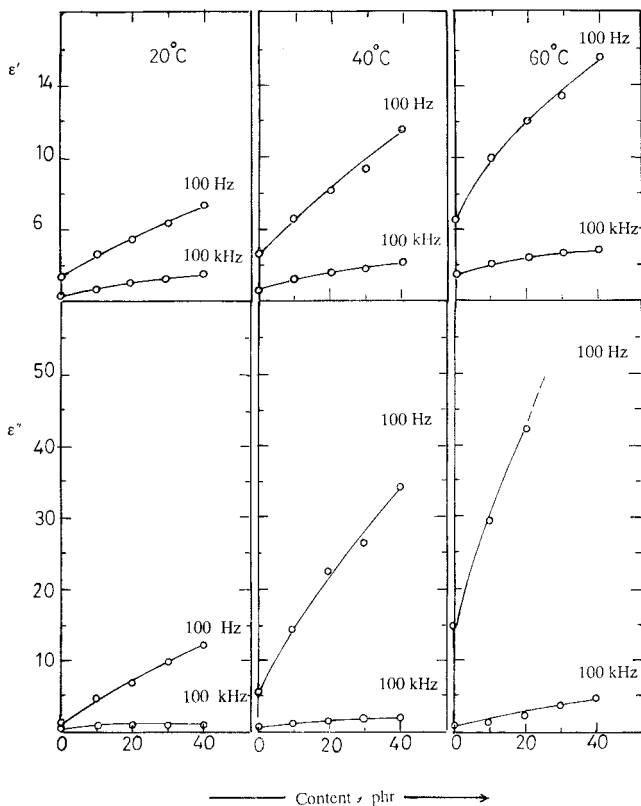


Figure 1 The permittivity ( $\epsilon'$ ) versus the frequency ( $f$ ) at different temperatures for the PVC-DOP system mixed with FEF CB at (•) 0, (+) 10, (○) 20, (×) 30, and (△) 40 phr.



**Figure 2** The variation of the permittivity ( $\epsilon'$ ) and dielectric loss ( $\epsilon''$ ) with the content of FEF CB at different temperatures and frequencies.

properties of the PVC-DOP system have been discussed. A comparative study of PVC-DOP-CB and PVC-CB systems has also been made.

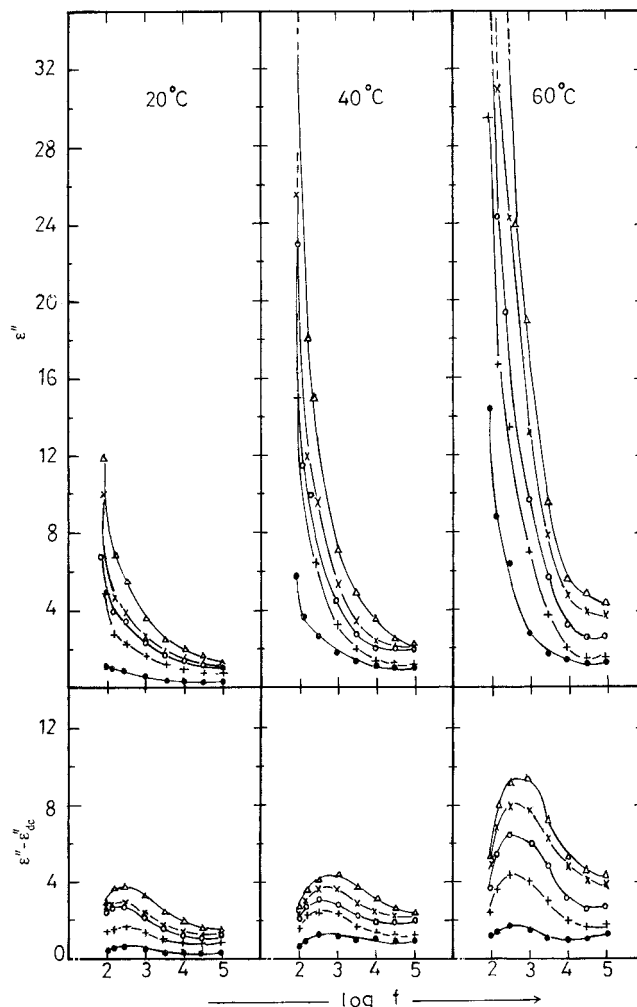
**Effect of electrically conductive FEF CB on properties of PVC-DOP system and PVC matrix**

**Electrical properties**

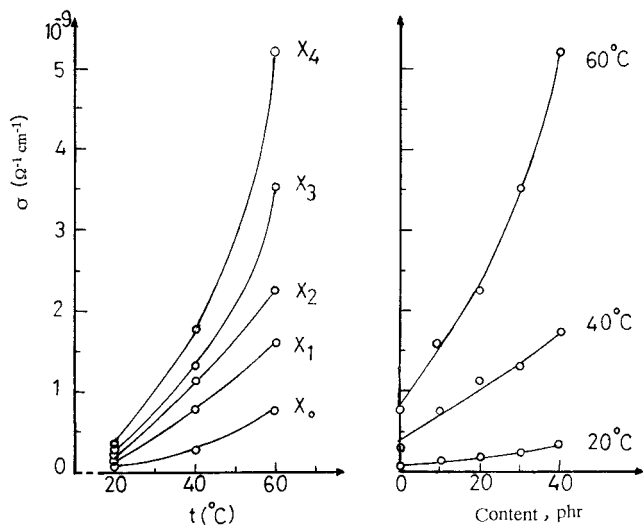
The  $\epsilon'$  and  $\epsilon''$  for samples  $X_0, X_1, X_2, X_3,$  and  $X_4$ , which correspond to the PVC-DOP-CB system, were studied over the frequency region from 100 Hz to 100 kHz at temperatures from 20 to 60°C. It is evident from Figure 1, which represents the variation of  $\epsilon'$  with the frequency ( $f$ ) at different temperatures, that the value of  $\epsilon'$  increases with increasing temperature and decreases with increasing frequency. Similar behavior was noticed before in the literature.<sup>11-14</sup> The increase of  $\epsilon'$  with the temperature can be explained by the increase in the mobility of polar groups, the decrease in density, and hence a decrease in the effect of the environment that facilitates the orientation of the mobile groups. The decrease of  $\epsilon'$  with the frequency may be caused by dielectric dispersion. Moreover, Figure 1 as well as Figure 2, which represents the relation between  $\epsilon'$  and the content of CB at frequencies of 100 Hz and 100 kHz and different temperatures, show that

the value of  $\epsilon'$  increases with the increase in carbon content, especially in the very low frequency region.

It is apparent from Figure 3, which represents the variation of  $\epsilon''$  with the frequency at different temperatures, that the value of  $\epsilon''$  is greatly increased at higher temperatures, especially in the lower frequency region. Moreover, Figure 3 reveals that, at each temperature,  $\epsilon''$  in the lower frequency region increases with increasing content of CB. Figure 2 also shows the relation between  $\epsilon''$  and the carbon content at frequencies of 100 Hz and 100 kHz and temperatures from 20 to 60°C. The increase in  $\epsilon''$  in the very low frequency region with increasing carbon concentration may be caused by the Maxwell-Wagner effect<sup>15</sup> as a result of an alternating current in phase with the applied potential, the direct current (dc) conductivity<sup>16,17</sup> resulting from the increase of ion mobility, or both. To confirm this, the dc conductivities of the investigated samples were measured by application of Ohm's law



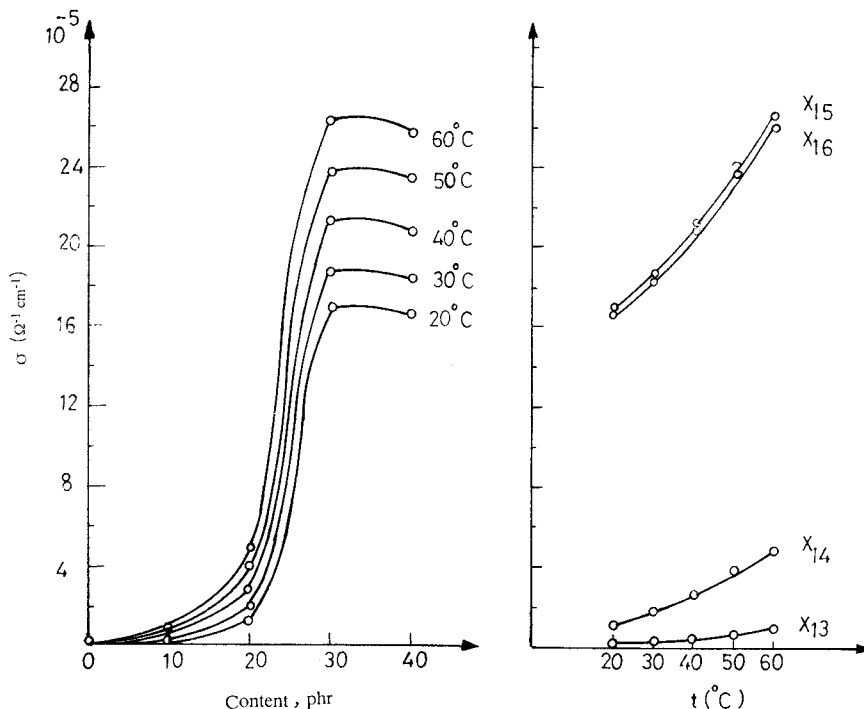
**Figure 3** The dielectric loss ( $\epsilon''$ ) and ( $\epsilon'' - \epsilon''_{dc}$ ) versus the frequency ( $f$ ) at different temperatures for the PVC-DOP system mixed with FEF CB at (•) 0, (+) 10, (○) 20, (×) 30, and (△) 40 phr.



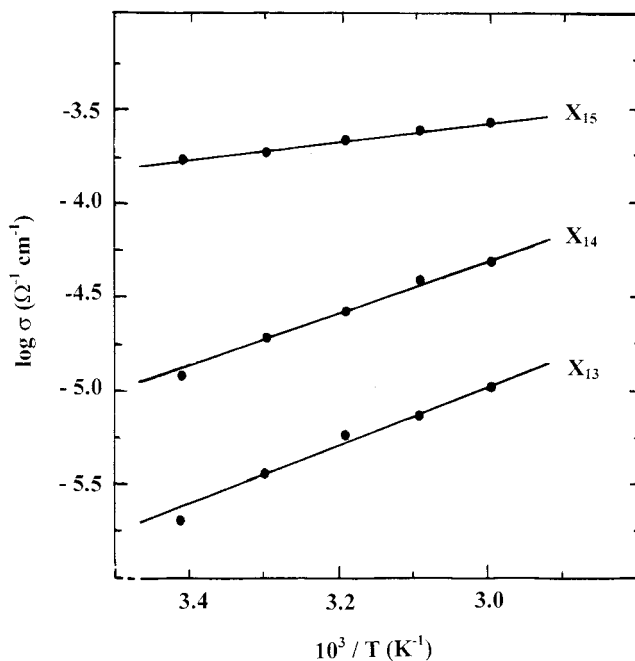
**Figure 4** The relation between the conductivity ( $\sigma$ ) and temperature ( $t$ ) or carbon content for the PVC-DOP system mixed with FEF CB.

to the dc flowing through the samples at 200 V and at temperatures of 20–60°C. All samples showed appreciable dc conductivity. The dielectric losses caused by the dc conductivity ( $\sigma$ ) at the different frequencies ( $\omega$ ) are calculated using the equation<sup>18</sup>

$$\epsilon''_{dc} = \frac{9 \times 10^{11} 4\pi\sigma}{\omega}$$



**Figure 5** The relation between the conductivity ( $\sigma$ ) and carbon content or temperature ( $t$ ) for the PVC matrix mixed with FEF CB.



**Figure 6** The relation between the  $\log \sigma$  and  $10^3/T$  for samples of the PVC matrix mixed with FEF CB.

and subtracted from the values of  $\epsilon''$  in the low-frequency region. The data of  $\epsilon''$  after subtracting  $\epsilon''_{dc}$  are plotted against  $\log f$  and are represented in Figure 3. From this figure it is clear that for all samples there is an absorption region at different temperatures. The

**TABLE IV**  
**Activation Energy of Samples of PVC-CB System**

PVC-CB System	Activation Energy
100 g PVC + 10 g CB	3.36 kcal/mol
100 g PVC + 20 g CB	2.86 kcal/mol
100 g PVC + 30 g CB	0.93 kcal/mol

increase in the maximum loss peak is noticed with the increase of carbon content. As the temperature increases, the maximum loss peak also increases, becomes clear, and is shifted to higher frequency.

It may be concluded that the low-frequency losses are not totally dc losses and that they may comprise Maxwell-Wagner losses, because the differences between the permittivities and conductivities of the different ingredients in the samples are relatively large.

On the other hand, the change of the  $\sigma$  of samples  $X_0, X_1, X_2, X_3,$  and  $X_4$  with the temperature is shown in Figure 4. It is evident from this figure that at lower temperatures the  $\sigma$  of these samples is low and it increases as the temperature is increased. This may have been caused by the increase of the mobility of the ionic bodies that takes place as a result of the excitation by heat, leading to an increase in the conductivity of the samples. Figure 4 also shows that the value of  $\sigma$

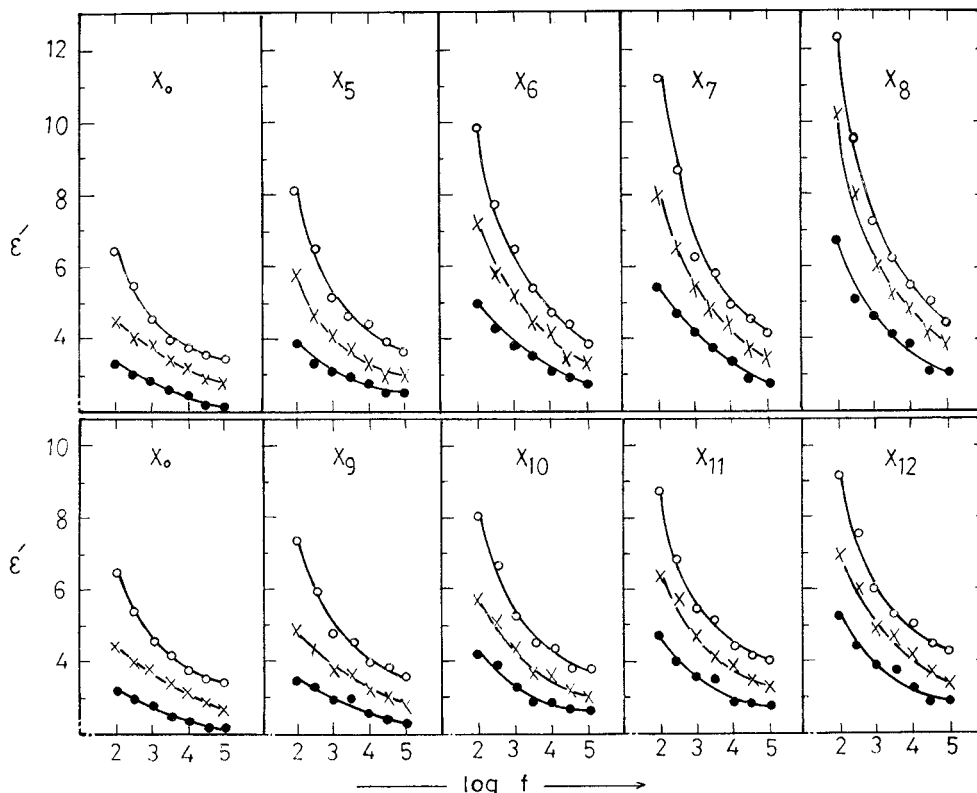
increases with the increase in the concentration of CB, which is an electrically conductive material. The figure shows the relation between the  $\sigma$  and the carbon content for the PVC-DOP-CB system at temperatures from 20 to 60°C.

For samples of the PVC-CB system (samples  $X_0, X_{13}, X_{14}, X_{15},$  and  $X_{16}$ ), the  $\sigma$  shows a much higher increase with the increase in the concentration of CB. The highest values for  $\sigma$  have been found for sample  $X_{15}$  containing 30 phr of CB (Fig. 5). where

$$\sigma = 26.3 \times 10^{-5} \Omega^{-1}\text{cm}^{-1} \text{ at } 60^\circ\text{C}$$

$$\sigma = 17.0 \times 10^{-5} \Omega^{-1}\text{cm}^{-1} \text{ at } 20^\circ\text{C}$$

These values are at the extremities of those of semi-conductors. At still higher concentrations of CB, the values of  $\sigma$  are approximately the same. The great increase in electrical conductivity may be attributed to the percolation phenomenon,<sup>19</sup> which is based on the fact that the conductive fillers are sufficient to come into contact with one another as the filler loading exceeds the threshold concentration. This means that when the CB is present in abundance in the PVC matrix, the PVC will be saturated with CB, which forms tunnels of electrons between adjacent carbon



**Figure 7** The permittivity ( $\epsilon'$ ) versus the frequency ( $f$ ) at ( $\bullet$ ) 20, ( $\times$ ) 40, and ( $\circ$ ) 60°C for the PVC-DOP system mixed with variable proportions of PVP and PAN ( $X_0, X_5, X_6, \dots, X_{12}$ ); see Table II.

clusters and through the PVC matrix.<sup>9</sup> When the electric current is applied, it is easier for the current to pass through these tunnels. Accordingly, there will be a high increase in the electrical conductivity.

The activation energy ( $E$ ) for electrical conductivity was calculated by plotting the logarithm of the  $\sigma$  against the reciprocal of the absolute temperature where a straight line was obtained for samples of the PVC-CB system (Fig. 6), showing that they tend to behave as semiconductors. From the slope of the straight line,  $E$  was calculated using the equation<sup>20</sup>

$$\sigma = \sigma_0 e^{-E/RT}$$

It is evident from the data in Table IV that the value of  $E$  decreases with the increase in the concentration of CB. The period of excitation depends on the activation energy needed to make the substance conducting. If the activation energy is low, as shown in Table IV, the PVC-CB systems, especially containing a higher concentration of CB, becomes semiconducting at room temperature or in the presence of indirect light. Thus, it can be better employed for electronic and microwave devices working at normal temperatures.

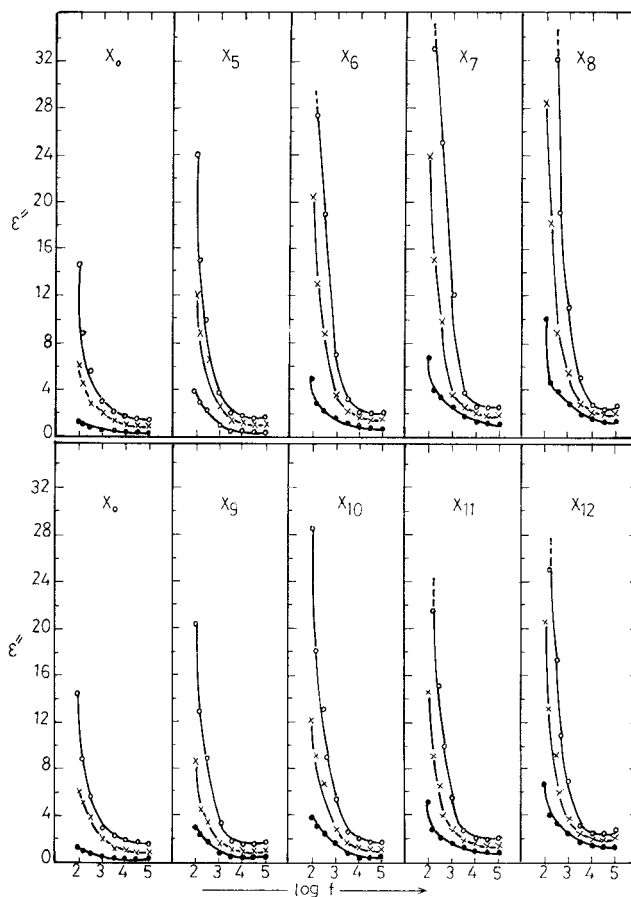
#### Mechanical properties

The variation of the tensile strength and percent elongation versus the concentration of FEF CB for PVC-DOP-CB and PVC-CB systems at  $23 \pm 2^\circ\text{C}$  is given in Tables II and III, respectively. The data show that increasing the content of CB to 10 phr increases the value of the tensile strength and then it decreases with a further increase in the CB content to 40 phr. This decrease of the tensile strength may be caused by the poor adhesion between the PVC and CB phases at a higher degree of loading, leading to the retardation of crosslinks across the interfaces. However, the data in Tables II and III show that the percent elongation decreases when increasing the content of CB up to 40 phr. The decrease of the percent elongation may be due to an interstructural process in which the CB molecules are distributed in the interaggregate space.

#### Effects of PVP and PAN on properties of PVC-DOP system

##### Electrical properties

The  $\epsilon'$  and  $\epsilon''$  for samples  $X_0, X_5, X_6 \dots X_{12}$ , which correspond to PVC-DOP-PVP and PVC-DOP-PAN systems, were studied over the frequency region from 100 Hz to 100 kHz at temperatures from 20 to  $60^\circ\text{C}$ . It is evident from Figure 7, which represents the variation of  $\epsilon'$  with the frequency at different temperatures, that the value of  $\epsilon'$  increases with increasing temperature and decreases with increasing frequency. More-



**Figure 8** The dielectric loss ( $\epsilon''$ ) versus frequency ( $f$ ) at ( $\bullet$ ) 20, ( $\times$ ) 40, and ( $\circ$ )  $60^\circ\text{C}$  for the PVC-DOP system mixed with variable proportions of PVP and PAN ( $X_0, X_5, X_6, \dots, X_{12}$ ); see Table II.

over, Figure 7 shows that the value of  $\epsilon'$  increases with an increase in the PAN content and increases to a greater extent with the increase in the content of PVP, especially at the very low frequency region.

It is apparent from Figure 8, which represents the variation of  $\epsilon''$  with the frequency at different temperatures, that the value of  $\epsilon''$  is increased at higher temperatures, especially in the lower frequency region. Moreover, Figure 8 reveals that, at each temperature, the  $\epsilon''$  in the lower frequency region increases with increasing PAN content and increases to a greater extent with the increase in the content of PVP; it is also slightly changed at frequencies higher than 10 kHz, where it becomes approximately the same for all samples and reaches very small values. This increase in the value of  $\epsilon''$  is the result of the high electrical conductivity of these samples in the same sequence.

The change of the  $\sigma$  of samples  $X_0, X_5, X_6, \dots, X_{12}$  with temperature is shown in Figure 9. It is evident from this figure that the value of  $\sigma$  increases with the increase in temperature from 20 to  $60^\circ\text{C}$ . With respect to sample  $X_0$  mixed with PVP ( $X_5, X_6, X_7$ , and  $X_8$ ) or PAN ( $X_9, X_{10}, X_{11}$ , and  $X_{12}$ ), Figure 9 also shows that

$\sigma$  is increased with the increase in PAN content and increases to a greater extent with an increase of the content of PVP, especially at the lower frequency region and higher temperatures. The increase in the value of  $\sigma$  with increasing the concentration of PVP may be attributed to the presence of the lone pair of electrons on the ring nitrogen atom in VP units that are mobile under the influence of an electric field, together with the presence of double bonds in the molecule that can be easily excited and the  $\pi$  bond is opened. Accordingly, the degree of electrical conductivity is greatly increased in these samples, despite the steric hindrance due to the presence of pyridine rings incorporated into the polymer backbone as repeat units of VP that play an important role in decreasing the mobility of the electric charges. In addition, the increase in the value of  $\sigma$  with the increased content of PAN may have been attributable to the presence of a number of polar groups in PAN that are capable of orientation in an electric field. Figure 10 shows the relation between the  $\sigma$  and the content of PVP or PAN at temperatures from 20 to 60°C.

Mechanical properties

The variation of the tensile strength and percent elongation versus the concentration of PVP or PAN at  $23 \pm 2^\circ\text{C}$  is given in Table II. The data show that the tensile strength and percent elongation are decreased with the increase of the concentration of PVP or PAN up to 40 phr in the sequence PVP < PAN, but this

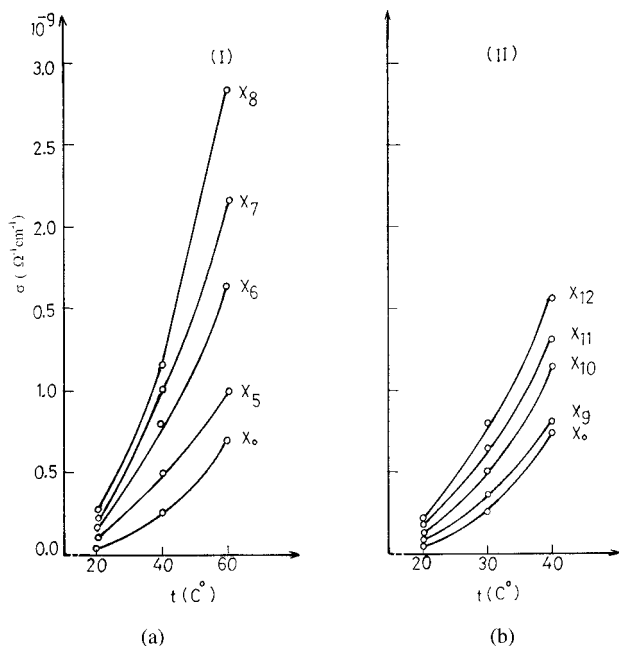


Figure 9 The relation between the conductivity ( $\sigma$ ) and temperature ( $t$ ) for (a) samples  $X_0, X_5, X_6, X_7,$  and  $X_8$  and (b) samples  $X_9, X_{10}, X_{11},$  and  $X_{12}$ .

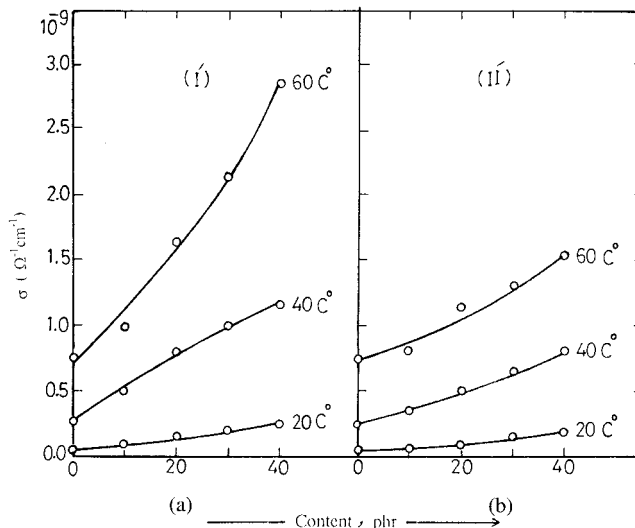


Figure 10 The relation between the conductivity ( $\sigma$ ) and the content of (a) PVP or (b) PAN at temperatures from 20 to 60°C.

decrease will not prevent utilization in electronic devices.

CONCLUSIONS

This study leads to a number of conclusions.

FEF CB

- The samples the of PVC–CB system containing a high content of CB possess much higher electrical conductivity because of the percolation phenomenon than those of PVC–DOP–CB system.
- The activation energy for the electrical conductivity is low for the PVC–CB systems, especially containing a higher concentration of CB, showing that they tend to behave as semiconductors.
- A higher loading of CB brings about the deterioration of the mechanical properties (tensile strength and percent elongation); therefore, the CB content must be reduced as much as possible.

PVP and PAN

- The electrical conductivity of a PVC–DOP system is increased by the addition of PVP or PAN up to 40 phr in the sequence PVP > PAN, especially at the lower frequency region and higher temperatures.
- The tensile strength and percent elongation are decreased with the increase of the concentration of PVP or PAN up to 40 phr in the sequence PVP < PAN. Therefore, plasticized PVC formulated with 40 phr PVP (sample  $X_8$ ) possesses higher



electrical conductivity and good mechanical properties, making it of great interest in the fabrication of electronic and microwave devices and battery electrode applications.

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