

Dielectric Relaxation and Poole–Frenkel Conduction in Poly(vinyl chloride) Blends with Bisphenol A/Egyptian Corncob Resin

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Received 22 August 2007; accepted 27 December 2007

DOI 10.1002/app.28075

Published online 20 March 2008 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The dielectric constant (ϵ'), dielectric loss index (ϵ''), direct-current conductivity, and current–voltage (I – V) characteristics of pure poly(vinyl chloride) (PVC) and blends of PVC and bisphenol A/Egyptian corncobs (BCC) were investigated at different temperatures. The relaxation processes for PVC and its blends revealed that PVC and BCC had an incompatible phase. PVC blends with 5 wt % BCC exhibited a peculiar I – V behavior. Both ϵ' and ϵ'' were used to study miscibility and phase behavior in

blends of PVC. The activation energies of all PVC samples were calculated. At higher voltages, the conduction mechanism could be identified as the Poole–Frenkel type. In addition, the ionic groups of BCC could enhance the PVC conductivity. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 182–189, 2008

Key words: activation energy; charge transport; conjugated polymers; dielectric properties; immiscibility

INTRODUCTION

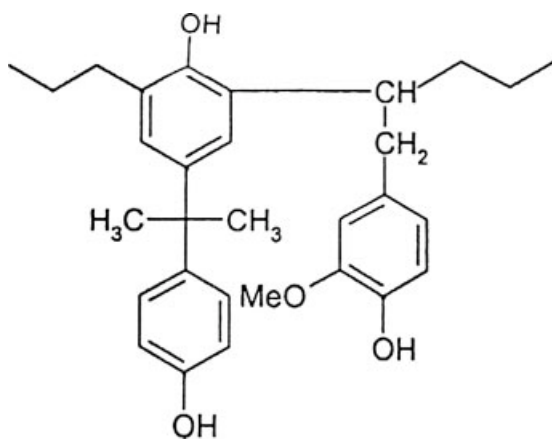
Blends allow us to combine the useful properties of different molecular species and provide an incredible range of physical and morphological states. The complexity of these systems requires careful investigation of features such as the miscibility and glass-transition temperature (T_g). The study of the relaxation behavior in polymers and polymer blends (by nuclear magnetic resonance and mechanical and dielectric techniques) constitutes a powerful approach for obtaining information about the nature of molecular motion and the manner in which they are affected by the chemical composition, molecular structure, and morphology.^{1–4} Many polymers and copolymers have been mixed with poly(vinyl chloride) (PVC) to improve its mechanical, thermal, and dielectric properties.^{5–7} The compatibility of many PVC-based blends has been reported by the detection of T_g , which involves solid-state analysis. In the amorphous phase of polymers, there are two main-chain motions: micro-Brownian motion, which occurs at T_g , and a more local type of motion, which occurs in the glassy state.

The relaxation behavior in polymeric blends has been related to the heterogeneous character of the α -relaxation process associated with the concentration fluctuations in the blends at a microscopic scale.⁷

These dynamic heterogeneities should yield a distribution of the relaxation processes characteristic of each polymer. The study of the amorphous loss process (α) provides the most reliable means of assessing miscibility in polymer blends. In a binary blend, the α -relaxation process is based on the occurrence of either one or multiple loss peaks. A single α -relaxation peak corresponds to a miscible mixture. The dielectric properties of PVC are considered to be a good tool for studying the α -relaxation mechanisms dealing with the dynamics of the mobile groups. Hence, it is very important to examine the dielectric relaxation of PVC as a result of the variation of the vibration of its molecular motion, which is affected directly by bisphenol A/Egyptian corncobs (BCC). Moreover, the detection of a steady-state current is one approach that leads to deeper information on the kinds of charge carriers and transport processes linked to the PVC–BCC structure and the chain motion.⁸

The optimum preparation conditions and characterization of bisphenol A/Egyptian bagasse pith polycondensation products and corncobs have been reported.^{9–11} Both chemical analysis and IR spectroscopy have shown that BCC resin has a complicated structure. The resin structure includes furan and lignin hydrolysate units, which are present in a random alternation in the resin chains. IR spectroscopy of soda lignin/bisphenol resin shows the presence of aliphatic methyl, methylene, $-\text{OCH}_3$, and strong OH groups.^{9,10} The chemical structure of BCC is shown in Scheme 1.

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Scheme 1.

This study was aimed at exploring the miscibility and phase behavior of PVC–BCC blends by the use of a dielectric relaxation techniques. Because phenyl, $-\text{OCH}_3$, and $\text{C}-\text{Cl}$ groups have strong dipole moments, relaxation processes of the PVC–BCC system can be examined with dielectric measurements. Subsequently, the dielectric data can also give hints about the molecular interactions in such blends. Also, to get evidence about the carrier traps and type of conduction mechanism in PVC blends, the current–voltage (I – V) characteristics and direct-current (dc) conductivity at different temperatures were determined.

EXPERIMENTAL

PVC was supplied by Polymer Laboratories, Ltd. (Essex, UK) The weight-average molecular weight was 2×10^5 , and the weight-average molecular weight/number-average molecular weight ratio was 1.9. The BCC resin was specially prepared.¹¹ An aqueous solution of PVC was obtained by the addition of 1 g of PVC to 20 mL of tetrahydrofuran (THF) and the heating of the mixture to 40°C with continuous stirring. An aqueous BCC solution was obtained by the addition of BCC in a proper mass fraction (W) of 5, 10, 15, 20, 25, or 30 wt % to 10 mL of THF at 40°C . The mass fraction was calculated according to this formula:

$$W(\text{wt}\%) = \frac{\omega_{\text{bis}}}{\omega_{\text{p}} + \omega_{\text{bis}}} \times 100 \quad (1)$$

where ω_{bis} and ω_{p} represent the weights of BCC and PVC, respectively. The BCC solution was added to the PVC solution at 40°C , and their mixture was cast into Petri dishes placed on a leveled plate for 3 days until the solvent was completely evaporated. The obtained film of 0.1-mm thickness was cut into square pieces and then coated with silver paste to achieve ohmic contacts. The dielectric measurements

were carried out with a Hioko 3531 RLC bridge (Nagano, Japan) in the frequency and temperature ranges of 1 kHz to 5 MHz and 300–410 K, respectively. The dielectric constant (ϵ') and dielectric loss index (ϵ'') were calculated as follows:

$$\epsilon' = \frac{Cd}{\epsilon_0 A} \quad (2)$$

$$\epsilon'' = \epsilon' \tan \delta \quad (3)$$

where C is the capacitance of the sample-filled capacitor, d is the sample thickness, ϵ_0 is the vacuum permittivity, and A is the electrode area. dc was measured with a Keithley 485 autoranging picoammeter (Cleveland, OH, USA). The temperature of the sample was measured with a T-type thermocouple. The measurement accuracy for the temperature was about ± 1 K.

RESULTS AND DISCUSSION

Dielectric properties

The frequency dependence of ϵ'' of pure PVC at different temperatures (300, 330, 360, and 410 K) is shown in Figure 1(a). It is clear that two relaxation peaks, ρ and α , can be observed. The ρ -relaxation

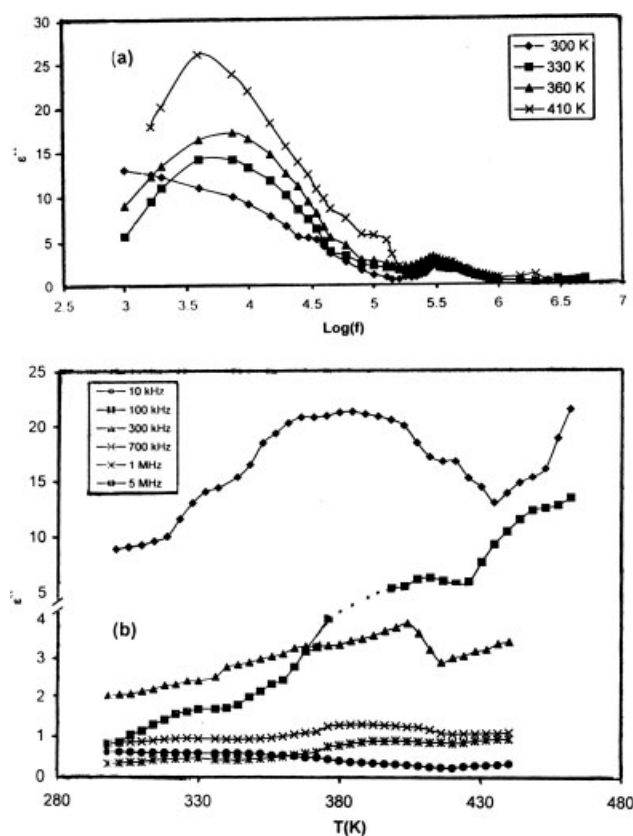


Figure 1 Frequency (f) and temperature (T) dependence of ϵ'' for pure PVC.

peak can be found at 1 kHz, whereas the α -relaxation peak dominates at 300 kHz. The same behavior has been noted for the temperature dependence of ϵ'' , especially at 10 kHz, as shown in Figure 1(b). These two relaxations processes (α and ρ) are presented at 370 and 440 K, respectively. In addition, the peak height of the α -relaxation process is shifted to higher temperatures with increasing field frequencies.

The α -relaxation peaks show an increase in magnitude with the temperature as a result of micro-Brownian motion of the investigated sample. This may be due to the increase in the density of dipoles, which contributes to the relaxation process, with increasing temperature. Moreover, the α -relaxation peak can be ascribed to the release of the frozen-in dipolar C—Cl groups and their cooperative motion with adjoining segments of the main chain.^{12,13} The ρ -relaxation peak can be attributed to the electrical conduction in the material. The electrical conductivity in the polar polymer is not due to the polymer chain itself but is probably caused by a very low level of ionic impurities inside the material.¹⁴

The ρ -relaxation process seems to depend on the nature of the polymer. For a semicrystalline polymer, chain trapping of interfaces or Maxwell–Wagner polarization phenomena are more likely.¹⁵ For amorphous materials, this effect is related to cooperative parameters such as impurities, injected space charges, and electrode effects. The ρ process clearly originates in the motions of space charges that are accumulated in the polymer close to the electrodes during the formation.¹⁶

The study of miscibility in polymer blends by dielectric relaxation involves principally the assessment of the presence of one or more α -relaxation peaks and the accurate locations of the temperature and frequency. The complete set of ϵ'' values of PVC blends with 5 or 30 wt % BCC at 300, 330, 360, and 410 K is shown in Figure 2(a,b). This figure shows that the peak positions of the ρ - and α -relaxation peaks of PVC are still unchanged. However, two other relaxation peaks are observed: one at approximately 63 kHz and the other at approximately 3 MHz.

The dynamic dielectric results for PVC–BCC blends show that the location of the high-temperature ϵ'' peak of pure PVC is unaltered. According to these results, PVC and BCC are incompatible because the value of T_g of each polymer does not change with the composition. Similar results have been observed for PVC/high-density polyethylene blends, which show that the melting temperature of polyethylene is unaltered by the composition.⁵ Also, blends of PVC and chlorinated polyethylene (CPE) with 36 wt % chlorine are incompatible: the peak position of the α process of each polymer does not

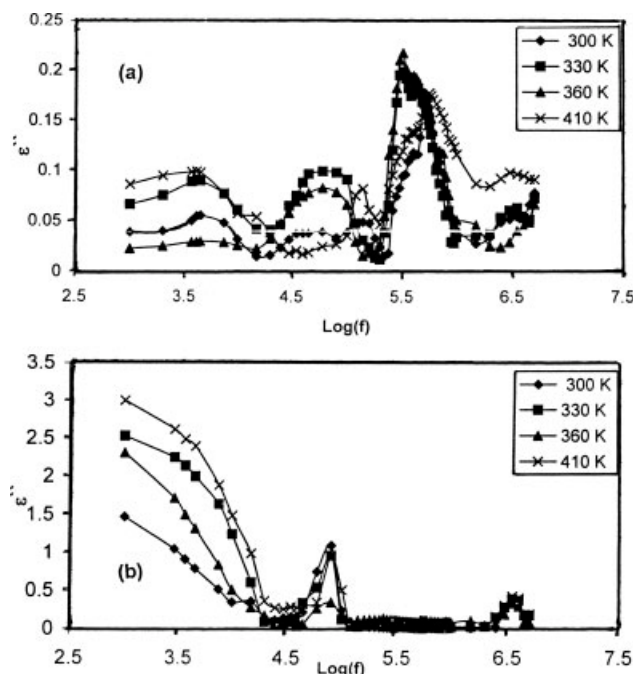


Figure 2 Frequency (f) dependence of ϵ'' for PVC blends with 5 or 30 wt % BCC at different temperatures.

change.¹⁷ In addition, PVC/ethylene–vinyl acetate–carbon monoxide polymer blends have been reported to be miscible¹⁸ on the basis of the specific interaction of carbonyl with α -hydrogen of PVC.¹⁹

On the other hand, T_g and the α -relaxation process of pure PVC and its blends with CPE have been studied by means of dynamical mechanical spectroscopy. According to this study, PVC/CPE (40% chlorine) blends are incompatible because T_g of each polymer remains constant with the composition. These results are similar to those obtained with a PVC/high-density polyethylene blend.²⁰ The compatibility of PVC with linear polyethers has been examined with dynamical mechanical analysis.²¹ This indicates that all polyethers show limited miscibility in the melt at high PVC contents. Moreover, the dynamic mechanical behavior of polymer blends of three conventional polymers—poly(vinylidene fluoride), poly(vinyl acetate), and poly(methyl methacrylate)—has been examined.²² The relaxation processes, corresponding to T_g of these blend compositions, reveal that relaxation tends to occur at notably lower temperatures in comparison with the pure polymer.

The frequency dependence of ϵ' and ϵ'' at 330 K for PVC blends with 5, 10, 15, 20, 25, or 30 wt % BCC is presented in Figure 3(a,b). All PVC blends undergo three relaxation processes: the α process of BCC at 63 kHz, the α process of PVC at 500 kHz, and the β process of BCC at 3 MHz. An additional loss peak, called the α' process, has been observed

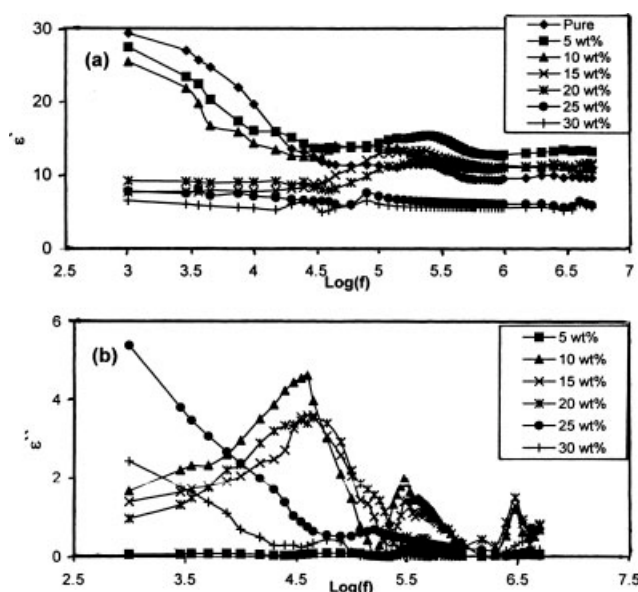


Figure 3 Frequency (f) dependence of ϵ' and ϵ'' for pure PVC and its blends with different concentrations of BCC at 330 K.

for pure PVC and PVC blends containing 5, 10, 15, or 20 wt % BCC at 260 kHz. An α' -relaxation process has been reported for PVC,^{14,18} but this effect is weak and is not always observed in comparison with the main α -relaxation process. It has been found that the α' -relaxation process is present mainly in the methyl ester form polymer and is obscured by more intense peaks.²³ It represents the cooperative dipole motion of C—O—CF₂—CF(CF₃) for the perfluorocarboxylate polymer. In this work, the α' -relaxation process can be caused by the cooperative dipolar motion of the PVC end groups²⁴ or the segmental motion of the branching of the PVC main chain. With the BCC concentration increasing

from 5 to 20 wt % inside the PVC sample, the peak height of α' and α peaks of PVC is decreased, and with BCC further increasing, the α' peak disappears. It can be suggested that the phenol and aliphatic methyl groups of BCC may screen the C—Cl groups of PVC and prevent the cooperative motion of C—Cl as well as the end groups of PVC.

The α relaxation of BCC at 63 kHz may originate from the normal segmental motion of the main chain, where the phenol groups align themselves in such a way and participate in the cooperative motion of the main chain. Moreover, the polarization of the aliphatic methyl, methylene, or —OCH₃ groups plays an important role in the β process of BCC, which is located at 3 MHz. The loss curves at various temperatures can be reduced to a master curve with the peak position (f_{\max}) and peak height (ϵ''_{\max}) for the α -relaxation process. Figure 4(a,b) shows the master curve for pure PVC and its blends with 5 wt % BCC, respectively. The shape shows an asymmetric behavior, and it is broader than that of Debye relaxation. This indicates that there is a distribution in the relaxation times. The peak frequency [$f_{\max}(\epsilon'')$] as a function of temperature can be described by the Arrhenius equation:^{25,26}

$$f_{\max} = f_0 \exp - \left(\frac{E_a}{kT} \right) \quad (4)$$

where f_0 is a constant, k is Boltzmann's constant, and E_a is the activation energy. The calculated value of the apparent activation energy of the α -relaxation process was found to be 0.048 eV, which is consistent with previously reported data.²⁶

Figure 5 shows the temperature dependence of ϵ'' for pure PVC and its blends with 5, 10, 15, 20, 25, or 30 wt % BCC at 300 kHz. It can be observed that the peak height of the α -relaxation process decreases

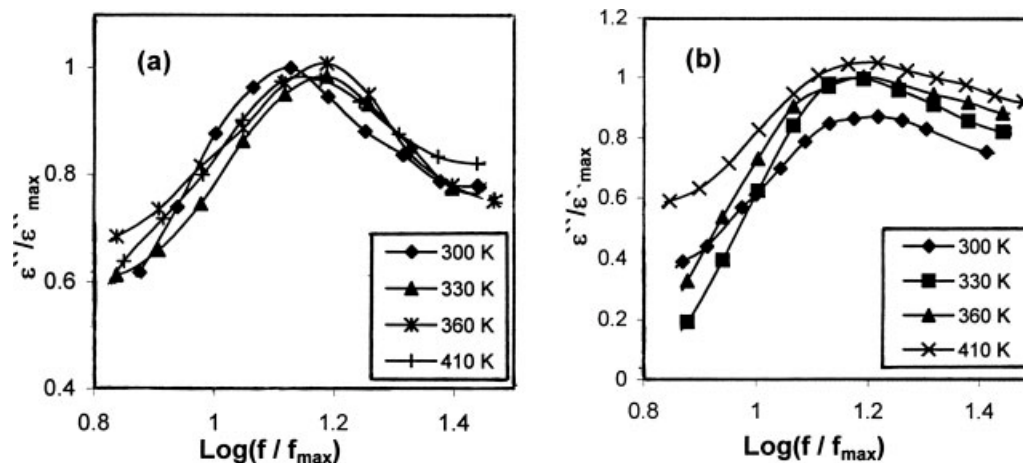


Figure 4 Master curve of $\epsilon''/\epsilon''_{\max}$ versus $\log(f/f_{\max})$ for pure PVC and its blend with 5 wt % BCC at different temperatures.

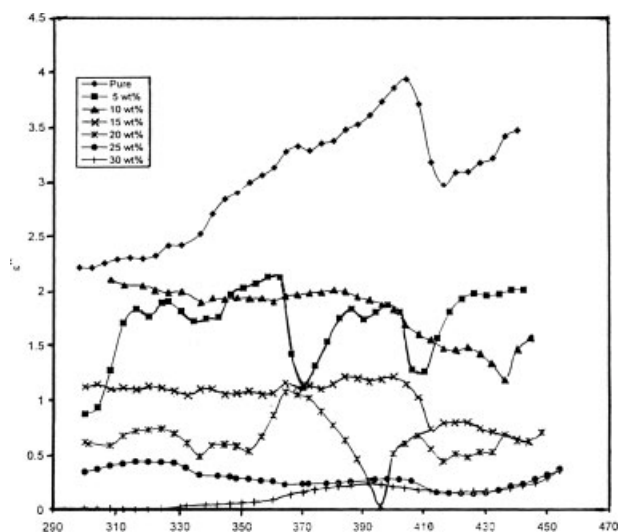


Figure 5 Temperature (T) dependence of ϵ'' for pure PVC and its blends with different concentrations of BCC at 300 kHz.

with the increase in the BCC ratio. The α -relaxation process completely disappears at 25 and 30 wt % concentrations of BCC. This result can be attributed to the lateral groups of the additive resin, which tend to screen C—Cl groups of PVC. Also, the PVC blend with 5 wt % BCC exhibits five relaxation processes. The α -relaxation process of BCC is split into two peaks at 320 and 360 K. The first peak (at 320 K) can be attributed to the rotational polarization of the phenol with $-\text{OCH}_3$ groups. With increasing temperature, the rotational motion of the two phenol groups, in addition to the contribution of the two aliphatic methyl groups, may be responsible for the existence of the second peak (at 360 K). Similarly, the α -relaxation peak of PVC has also been split into α' - and α -relaxation peaks at 386 and 397 K. In addition, the ρ -relaxation peak for all PVC samples has been observed above 430 K. One can note that, with the BCC concentration inside PVC increasing from 10 to 30 wt %, the peak height of all relaxation processes decreases. Also, 5 wt % BCC leads to T_g of PVC decreasing, and this indicates that at this concentration it behaves as a plasticizer for PVC.

The temperature dependence of ϵ' for pure PVC and its blends with 5, 10, 15, 20, 25, or 30 wt % BCC at 300 K is presented in Figure 6. The data show that the values of ϵ' increase to a maximum for the PVC blend with 5 wt % BCC and then decrease with increasing BCC. The main-chain mobility depends slightly on the case of lateral group motion. Therefore, the respective positions of the methyl and methylene groups influence this mobility because of their important steric volume.²⁷ Because the strong steric hindrance results from the meta position ($-\text{OCH}_3$), the phenyl groups seem to be highly re-

stricted in rotation. Therefore, the structure of the investigated samples becomes inflexible, and the main chain does not change its configuration more easily. The positions of the aliphatic methyl and methylene groups in both lateral segments exhibit different behavior. Because of the repulsive forces²⁷ from the crowding of methyl and methylene groups via the increase in the BCC content in the PVC structure, the segmental motion of bisphenol corncobs may be limited. Consequently, the contribution of dipole-dipole interactions becomes weak, and the polarization and value of ϵ' of the PVC blend sample decrease with increasing BCC content (>5 wt %).

dc conductivity

The temperature dependence of the dc conductivity for pure PVC and its blends with 5, 10, 15, 20, 25, or 30 wt % BCC is shown in Figure 7. The dc conductivity of PVC exhibits two distinct regions (I and II) located at low and high temperature ranges of 300–370 and 390–460 K, respectively. Similar behavior has been observed for PVC blends with 20, 25, or 30 wt % BCC. However, PVC containing 5, 10, or 15 wt % BCC shows anomalous behavior for dc conductivity. This may be attributed to different competing conduction mechanisms, such as the injected carriers from the electrodes, and the motion of C—Cl groups of the PVC and methyl, methylene, OCH_3 , and OH groups of BCC. It has been found that PVC conductivity can be affected by the dopant resin content. The maximum and minimum values of the conductivity correspond to the PVC samples with 5 and 30 wt % BCC, respectively.

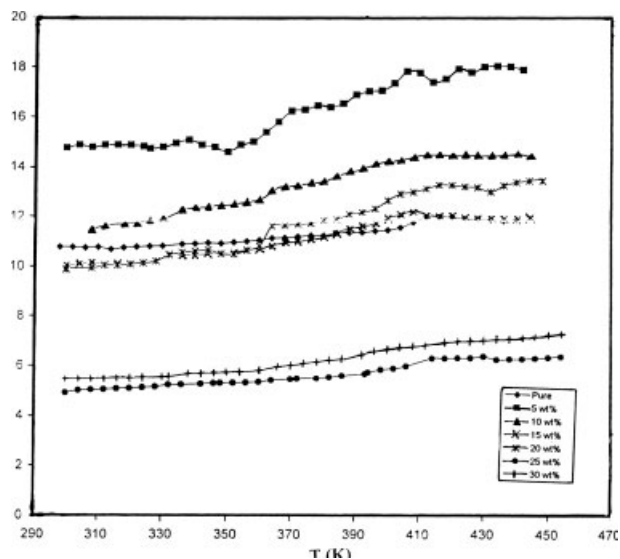


Figure 6 Temperature (T) dependence of ϵ' for pure PVC and its blends with different concentrations of BCC at 300 kHz.

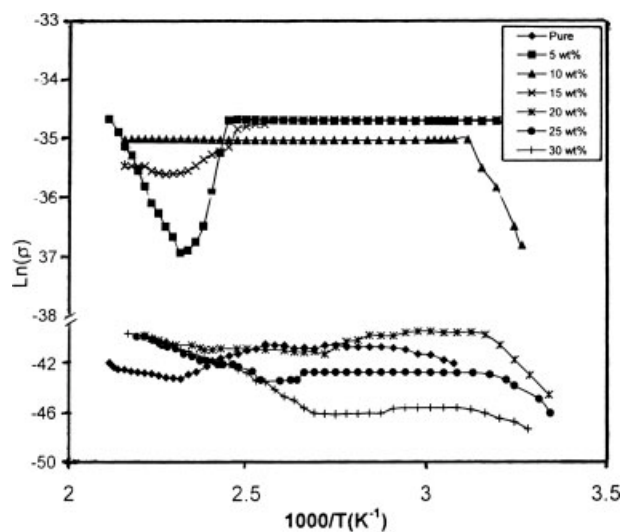


Figure 7 Temperature (T) dependence of the dc conductivity (σ) of pure PVC and its blends with different concentrations of BCC.

The activation energies of all investigated samples were calculated with the following Arrhenius-type equation, and they are listed in Table I:

$$\sigma = \sigma_0 \exp\left(\frac{E_a}{kT}\right) \quad (5)$$

where σ is the dc conductivity, σ_0 is a constant, E_a is the activation energy, and T is the absolute temperature. The values of the activation energy are strongly dependent on the concentration of BCC. In conclusion, the mass fraction (5 wt %) of BCC can be considered an optimum concentration to be added to PVC because the minimum value of the activation energy and maximum conductivity are achieved.

The conduction mechanism of the sample can be explained as follows. The configurations of polymer chains in the amorphous parts provide various heights of potential barriers against the migration of charged particles as well as the motions of chains themselves.²⁸ Below T_g , the motion of the C—Cl groups of the main chain is frozen in. Therefore,

TABLE I
Values of the Activation Energy (eV) for Pure PVC and Its Blends with Different Concentrations of BCC in Regions I and II

PVC sample	Region I	Region II
Pure PVC	0.58	0.39
PVC + 5 wt % BCC	—	—
PVC + 10 wt % BCC	1.14	—
PVC + 15 wt % BCC	—	—
PVC + 20 wt % BCC	1.86	0.23
PVC + 25 wt % BCC	1.46	0.76
PVC + 30 wt % BCC	0.65	1.05

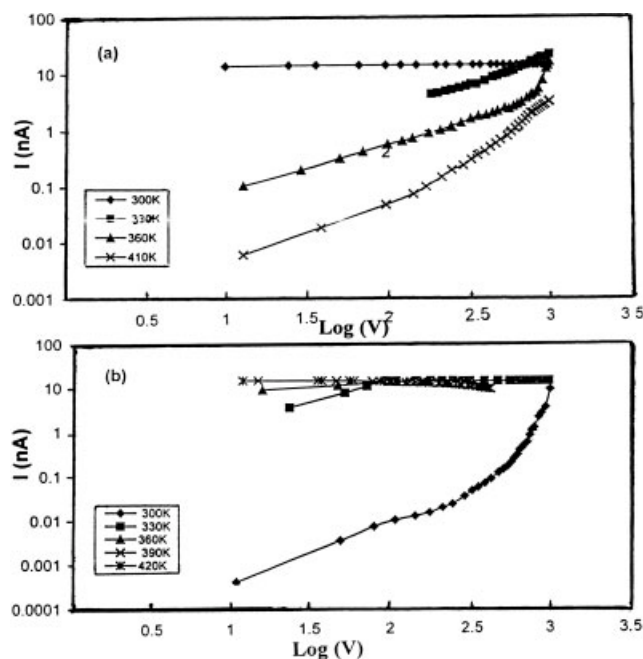


Figure 8 I - V characteristics of pure PVC and its blend with 5 wt % BCC.

extremely high barriers may eventually block the migration of the ions. Around T_g , the high potential barriers that restrict the dipole rotation disappear or change their positions.²⁹ In addition, the polar groups of the additive polymer act as sources of carrier enhancement of dc conductivity of the PVC sample.

Ionic conduction is also provided by a strong correlation between ϵ' and dc conductivity, and this is explained by the reduction of the coulombic forces between ions in a high- ϵ' medium.³⁰ Moreover, the reduction of the conductivity of the PVC samples, especially at a high concentration of BCC, can be ascribed to the steric effect and the repulsive force between the phenyl groups of BCC, as mentioned before.

I - V characteristics

A logarithmic I - V plot for pure PVC and PVC containing 5 wt % BCC within a temperature range of 300–410 K is shown in Figure 8(a,b). Both the pure PVC sample and electrodes are characteristically ohmic at 300 and 330 K. With the temperature increasing from 360 up to 410 K, the PVC sample exhibits two distinct regions. The first region obeys Ohm's law ($I \propto V$) with a slope of approximately 1. The second region is located at voltages (>500 V) at which the current increases rapidly ($I \propto V^2$) up to 1000 V with a slope of approximately 2. The PVC blend with 5 wt % BCC undergoes anomalous I - V behavior with increasing temperature rather than

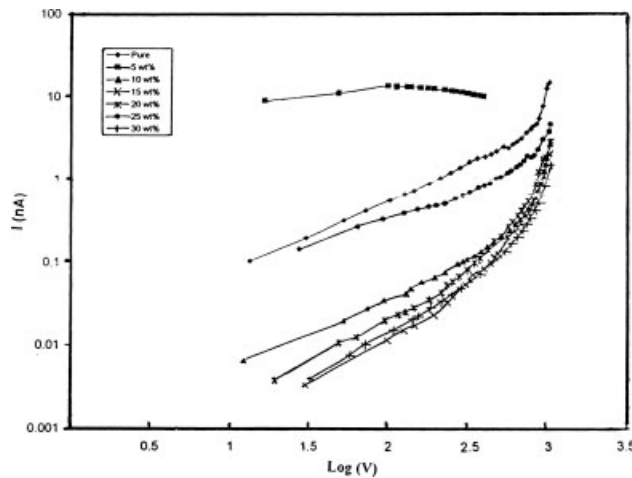


Figure 9 I - V characteristics of pure PVC and its blends with different concentrations of BCC at 360 K.

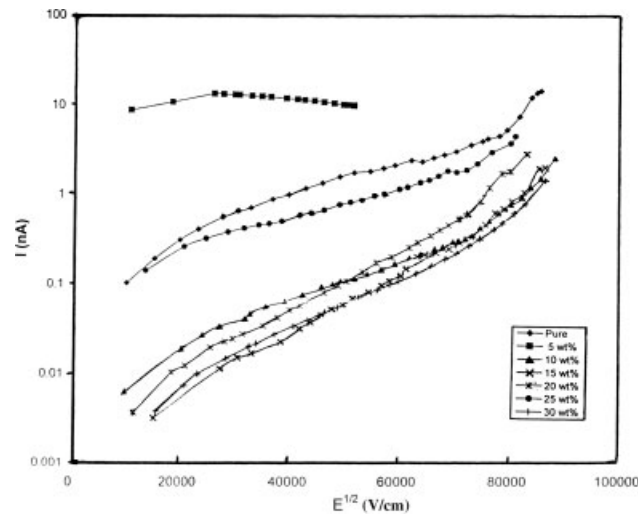


Figure 10 $\log I$ versus $E^{1/2}$ for pure PVC and its blends with different concentrations of BCC at 360 K.

300 K. The current is nearly constant and decreases with increasing applied voltage at 330, 360, and 410 K.

This can be interpreted as follows. With increasing temperature, the lateral movement does not occur without a correlated motion of part of the main chain or without a change of conformation of neighboring lateral groups. These intermolecular movements hinder reorientation, contribute to the barriers, and lengthen the relaxation time. The two methyl and methylene groups have an influence on the flexibility of the ionic groups in the free volume of PVC. However, at a higher temperature (420 K), the fluctuation or micro-Brownian motion of the main chain of the PVC blend sample dominates. Then, the charge carriers may be scattered because of the repulsive force among phenyl, aliphatic methyl, and methylene groups of BCC. This may be responsible for the decreasing current values.

I - V characteristics for all PVC samples at 360 K are shown in Figure 9. According to this figure, PVC doped with 5 wt % BCC shows anomalous I - V behavior, as mentioned previously. Also, Figure 10 shows that the relation between the logarithm of the current ($\log I$) and the square root of the electric field strength ($E^{1/2}$) (V/cm) for all PVC samples at 360 K. This figure indicates that, in the second region (>500 V), the current is governed by either the Richardson-Schottky or Poole-Frenkel mechanism.³¹ The current density/voltage expression is given by¹

$$J = AT^2 \exp\left(\frac{-\phi_s}{kT}\right) \exp\left(\frac{\beta_s V^{1/2}}{kTd^{1/2}}\right) \quad (6)$$

for the Schottky effect (which is a thermoionic emission over a field-lowered barrier) and by³²

$$J = J_0 \exp\left(\frac{\beta_{PF} V^{1/2}}{kTd^{1/2}}\right) \quad (7)$$

for the Poole-Frenkel effect, which is the lowering of the coulombic potential barrier when it interacts with the electric field. In the expression, J is the current density, V is the voltage, $A = 1.2 \times 10^6$ A/m² is the Richardson constant, T is the absolute temperature, ϕ_s is the Schottky barrier height at the injecting electrode interface, d is the sample thickness, and J_0 is the low-field current density. β_s and β_{PF} are the Schottky and Poole-Frenkel field lowering coefficients, respectively.

Because the β coefficient is an essential factor that determines the magnitude of the coulombic field, the β_s and β_{PF} values are theoretically calculated as follows:¹

$$2\beta_s = \beta_{PF} = \left(\frac{e^3}{\pi\epsilon_r\epsilon_0}\right)^{1/2} \quad (8)$$

where e is the electronic charge, ϵ_r is the relative permittivity, and ϵ_0 is the relative permittivity of the free space.

TABLE II
Calculated Values of the β Coefficient (eV V^{-1/2} m^{1/2}) for Pure PVC and Its Blends with Different Concentrations of BCC

PVC sample	β
Pure PVC	1.50×10^{-5}
PVC + 5 wt % BCC	—
PVC + 10 wt % BCC	3.72×10^{-5}
PVC + 15 wt % BCC	3.03×10^{-5}
PVC + 20 wt % BCC	4.35×10^{-5}
PVC + 25 wt % BCC	1.50×10^{-5}
PVC + 30 wt % BCC	3.00×10^{-5}

By taking the high-frequency ϵ' value to be 6, we have calculated β_s , and β_{PF} to be 1.548×10^{-5} and $3.09 \times 10^{-5} \text{ eV V}^{-1/2} \text{ m}^{1/2}$, respectively. The experimental value of the β coefficient (Table II), determined from Figure 10, is higher than 1.548×10^{-5} for the PVC blend samples. These experimental values of β are closer to the calculated β_{PF} value. Therefore, one can suggest that the dominant conduction mechanism for the PVC blend samples is the Poole-Frenkel type.

CONCLUSIONS

In this study, ϵ' , ϵ'' , dc conductivity, and I - V characteristics in PVC blends with BCC resin were analyzed. The α -relaxation processes for PVC and its blends revealed that PVC and BCC were incompatible. PVC blends with 5 wt % BCC exhibited a peculiar I - V behavior. The Poole-Frenkel conduction was predominant for PVC-BCC in a high-voltage region. A concentration of 5 wt % BCC was the optimum concentration for BCC to act as a PVC plasticizer. The PVC blend with 5 wt % BCC had maximum conductivity and minimum activation energy.

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