



## Dielectric and Conductive Characteristics of Polyaniline Coated Short Nylon Fiber Chloroprene Rubber Composites: A Comprehensive Study on Frequency, Temperature and Loading Dependencies

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Received: 2 December 2023;

Accepted: 1 January 2024;

Published online: 31 January 2024;

AJC-21530

In delving into the realm of conducting polymer composites (CPCs), specifically focusing on polyaniline (PANI), polyaniline coated short fibers of nylon (PANI-N) and chloroprene rubber (CR), this study takes an exploratory journey. Blending PANI/PANI-N/CR and PANI/CR CPCs mechanically, while adding short nylon fibers significantly improves their mechanical strength. These strong and durable CPCs possess significant promise for a wide range of applications in various devices. Present research methodically probes the dielectric features of CR, PANI/CR and PANI/PANI-N/CR composites across a frequency range of 0.1 to 8 MHz and a temperature span of 303 to 393 K. The results highlight potential correlations between mechanical properties and dielectric behaviour, paving the way for tailored approaches in developing high-performance conducting polymer composites for advanced applications. The study also utilizes theoretical equations and mixing models, which ensures a rigorous validation process that enhances accuracy.

**Keywords:** Conducting polymers, Nylon, Polyaniline, Chloroprene rubber, Dielectric permittivity, AC conductivity.

### INTRODUCTION

Polyaniline (PANI) and different PANI/polymer blends have been shown to be effective in achieving a high dielectric constant. As a result, the conducting composites made of these materials are being considered for potential large-scale applications in microwave absorption and electromagnetic shielding [1-5]. This efficacy has been extensively examined through numerous investigations exploring the dielectric behaviour of PANI and its composites [6-8]. Specifically, the materials that contain some crystalline PANI have shown an impressive dielectric constant of  $10^4$  [9]. In the microwave range, PANI-carbon nanotube composites have been reported to exhibit higher permittivity and loss factor compared to PANI films, emphasizing the interaction between carbon nanotubes and PANI molecule chains, in enhancing PANI film conductivity [10]. Additionally, researchers have reported the electrical properties of various polymer blends, including PANI/polyimide, PANI/polychloroprene, PANI/polyvinyl chloride and PANI/polyvinyl alcohol blends [11-18].

The dielectric properties of conducting composites are affected by various parameters, including conductivity, mole-

cular structure, particle size, crystal structure and processing techniques [8,19-22]. External factors like the frequency of applied voltage, temperature, pressure and humidity further impact these properties [23-26]. Understanding the dielectric behaviour of conducting polymers and conducting polymer composites (CPCs) is crucial, especially concerning their potential applications in electronic devices and other technological advancements. The expressions of dielectric properties, conductivity and complex permittivity, are dependent on AC/DC voltage and temperature [27-29]. The investigation into the temperature and frequency dependence of CPC dielectric characteristics provides illumination into the underlying conduction mechanisms and dielectric polarization.

Within the wide range of CPCs, the combination of polyaniline, PANI-coated short nylon fibers and chloroprene rubber offers a fascinating opportunity for further investigation. While previous investigations have explored the dielectric behaviour of PANI and its composites, the present study takes a distinctive approach by focusing on PANI-based composites with chloroprene rubber (CR). To enhance both mechanical properties and conductivity, PANI-coated short nylon fibers (PANI-N) were

introduced alongside PANI in the CR matrices. The synthesis of PANI/CR (P series) CPCs and PANI/PANI-N/CR (F series) CPCs is detailed elsewhere [30]. The amalgamation of these constituents, particularly in the PANI/PANI-N/CR composites, exhibits promise in enhancing mechanical characteristics. This heightened mechanical integrity renders these composites potentially valuable for a wide array of applications in various devices.

This study meticulously examines the dielectric characteristics of CR and PANI/PANI-N/CR CPCs, aiming to elucidate how these properties fluctuate with temperature and frequency. Various theoretical models and mixture equations are employed to align with the obtained CPC dielectric data. The application of theoretical frameworks aims to enhance the precision and reliability of the analytical findings and to explore the conduction mechanisms.

## EXPERIMENTAL

**Sample preparation:** Polyaniline (PANI) was prepared using ammonium persulfate and hydrochloric acid. Short nylon fibers underwent a chemical etching process before being coated with aniline, resulting in PANI-coated short nylon fibers (PANI-N). The preparation and characteristics of these two processes is detailed elsewhere [31]. The composites were prepared by mechanical mixing. The amount of the three components for the preparation of the composites is detailed in Table-1, which indicates the amounts of PANI and PANI-N in the P-series and F-series, respectively. The preparation process and other properties has been extensively described elsewhere [30]. In this segment, we specifically focus on presenting the dielectric characteristics of these composites.

TABLE-1  
FORMULATION OF CONDUCTING POLYMER COMPOSITES

Components (phr <sup>*</sup> )	P series				F series		
	CP <sub>0</sub>	CP <sub>1</sub>	CP <sub>2</sub>	CP <sub>3</sub>	CF <sub>1</sub>	CF <sub>2</sub>	CF <sub>3</sub>
CR	100	100	100	100	100	100	100
PANI	0	50	100	150	100	100	100
PANI-N <sup>#</sup>	0	0	0	0	40	80	120

<sup>\*</sup>Parts per hundred rubber; <sup>#</sup>PANI coated short nylon fiber. Stearic acid- 1 phr and DCP- 3 phr were used in all composites.

**Dielectric measurements:** The methodology for evaluating AC conductivity from dielectric measurements is based on the approach provided by Goswamy [32]. Using a dielectric cell Hewlett Packard 4192A, the dielectric characteristics of the CPCs were explored. Teflon pellets and lead were used to standardize the cell. Following the method recommended by Ramasasthry & Rao [33], the fringe capacitance was eliminated. The set up consists of two copper disc electrodes (diameter 12 mm, thickness 2 mm). The samples were placed between these electrodes and the capacitance was measured in the range 0.1 to 8 MHz range and 303 to 393 K. The impedance analyzer was interfaced with a computer, automating the data acquisition process through a graphical program based virtual instrumentation package using LabVIEW.

## RESULTS AND DISCUSSION

### Dielectric permittivity

**Dielectric properties of PANI:** The frequency and temperature dependence of pristine PANI was reported elsewhere [34]. The permittivity was found to decrease as frequency rises at all temperatures, particularly noticeable at lower frequencies, reaching a constant value. This behaviour implies significant low-frequency interfacial polarization in the systems, a characteristic exhibited by PANI. Maxwell-Wagner theory for interfacial polarization explains this behaviour [35]. The permittivity decreases slightly until 333 K and subsequently increases with temperature at all frequencies, with a more pronounced effect observed at lower frequencies. The dependence of dielectric loss factor of PANI with frequency was also studied [34].

Like dielectric permittivity, it decreased with frequency and approached a constant value at all temperatures, especially at lower frequencies. The loss factor had a linear relationship with frequency at lower frequencies and lower temperatures, deviating from the straight line above 353 K.

**Dielectric properties of chloroprene rubber (CR) gum vulcanizate:** The depiction of the dielectric permittivity variations in CR gum vulcanizates concerning both frequency and temperature is shown in Fig. 1. Specifically, the dielectric permittivity of the CR gum vulcanizate registers a value of 5.3 at 0.1 MHz indicating a decline with the escalating frequency. At higher frequencies, this decrease is more evident highlighting the favourable dielectric properties of CR. This diminishing trend is ascribed to a decrease in polarization as the frequency increases, with a specific emphasis on the lagging interfacial polarization at elevated frequency ranges. Moreover, the permittivity experiences an initial upswing up to 333 K, followed by a decrease as the temperature continues to ascend. This initial increase is especially observable at higher frequencies and is due to the increased polarity of the C-Cl bond. However, at exceedingly high temperatures, there is a consequential decrease in the matrix density, resulting in a subsequent decline in dielectric permittivity.

**Frequency dependence of CPCs:** In the investigation of dielectric characteristics, the P-series and F-series composites were subjected to measurements across the frequency spectrum from 0.1 to 8 MHz. The impact of frequency on the dielectric permittivity of selected composites at varying temperatures is presented in Fig. 2. Within the CPCs, the conducting particles find themselves surrounded by a matrix that is insulating, giving rise to space charge polarization at the interfaces, a phenomenon elucidated by the Maxwell-Wagner model. The interfacial polarization is viable primarily at the low frequencies. As frequency increases, the interfacial charges take longer to polarize, resulting in a decrease in dielectric permittivity. Composite CP<sub>3</sub> achieves a dielectric permittivity of up to 177 at 0.1 MHz and 303 K, while composite CF<sub>3</sub> exhibits a permittivity of 30 at the same conditions. These values are comparable to the PANI/PU blends reported by Chang *et al.* [4] ( $\epsilon' = 167$ ) at 0.1 MHz.

**Temperature dependence of the CPCs:** Fig. 3 shows the effect of temperature on the permittivity of CPCs by displaying

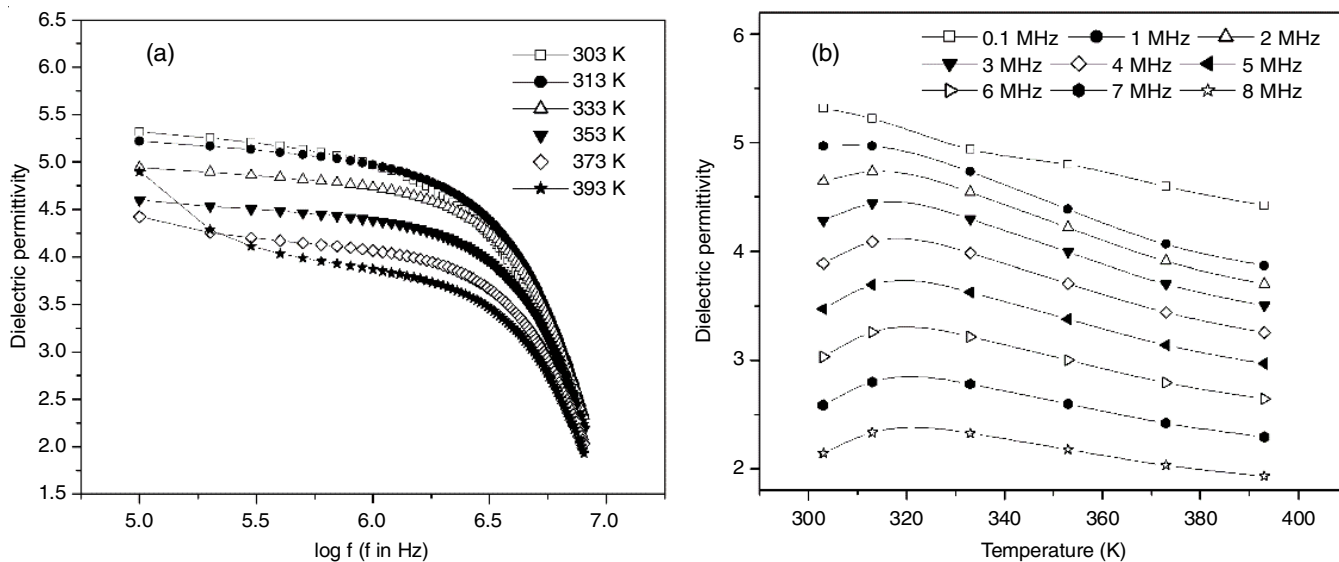


Fig. 1. Variation of dielectric permittivity of CR gum vulcanizate with (a) frequency and (b) temperature

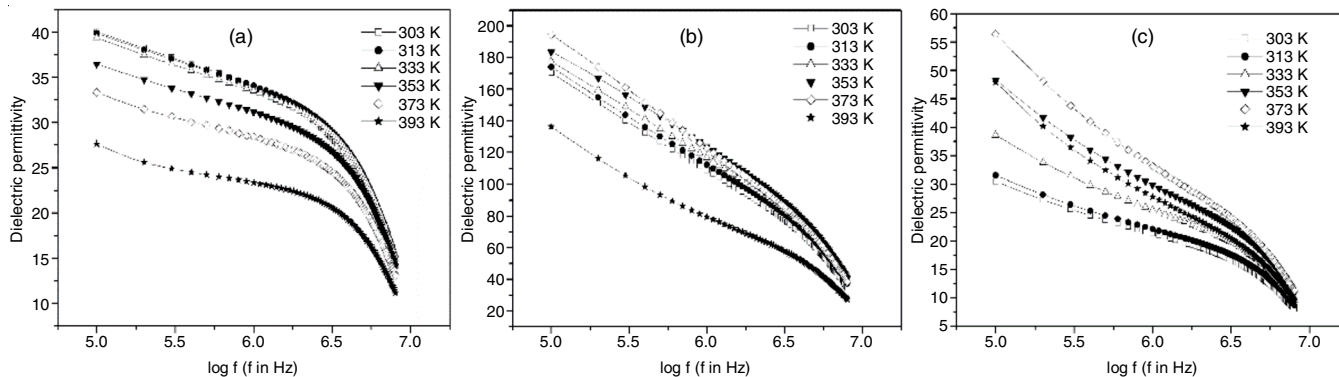


Fig. 2. Effect of frequency on the dielectric permittivity of CR/PANI composite (a) CP<sub>2</sub>, (b) CP<sub>3</sub> and (c) CF<sub>3</sub>

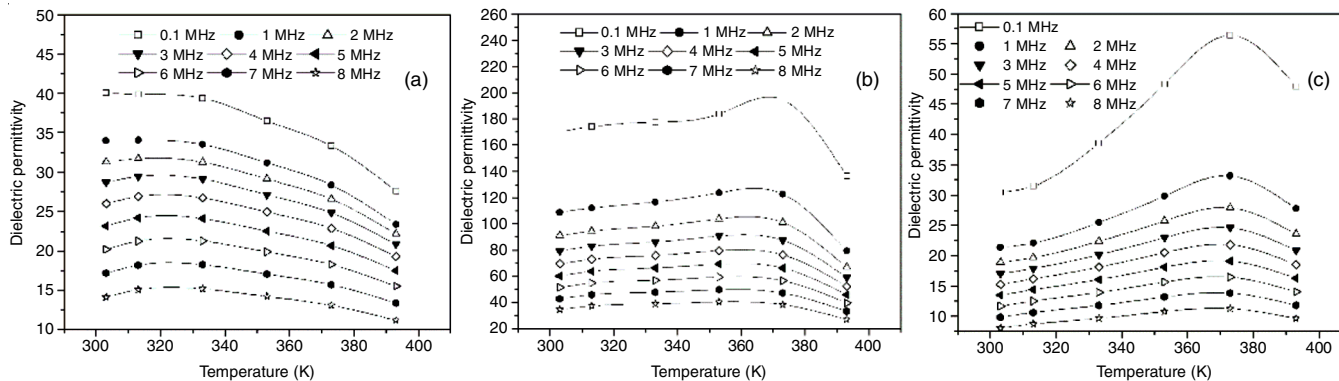


Fig. 3. Dielectric permittivity vs. temperature plots of (a) CP<sub>2</sub>, (b) CP<sub>3</sub> and (c) CF<sub>3</sub>

various PANI and PANI-N loadings at different frequencies. In case of CP<sub>2</sub>, the dielectric permittivity experiences a marginal increase till 313 K, followed by a slight decrease at higher temperatures. A similar temperature-dependent trend is observed for CP<sub>3</sub> up to 353 K. For CF<sub>3</sub>, an increase is up to 373 K, where the initial increase may result from the heightened interfacial polarization facilitated by the temperature increase, but the reduction in polymer matrix density counteracts this effect, leading to a subsequent decrease in dielectric permittivity.

**Loading dependence of the CPCs:** Fig. 4 displays the fluctuation in the dielectric permittivity for P-series and F-series CPCs with PANI and PANI-N loading at 303 K. For P-series composites, the dielectric permittivity rises with increasing PANI loading at all frequencies, particularly at lower frequencies. Conversely, for F-series, the dielectric permittivity generally rises with PANI-N loading but exhibits a decrease at higher loadings. For instance, composite CF<sub>1</sub>, loaded with 40 phr PANI-N, achieves a maximum dielectric permittivity of 70.

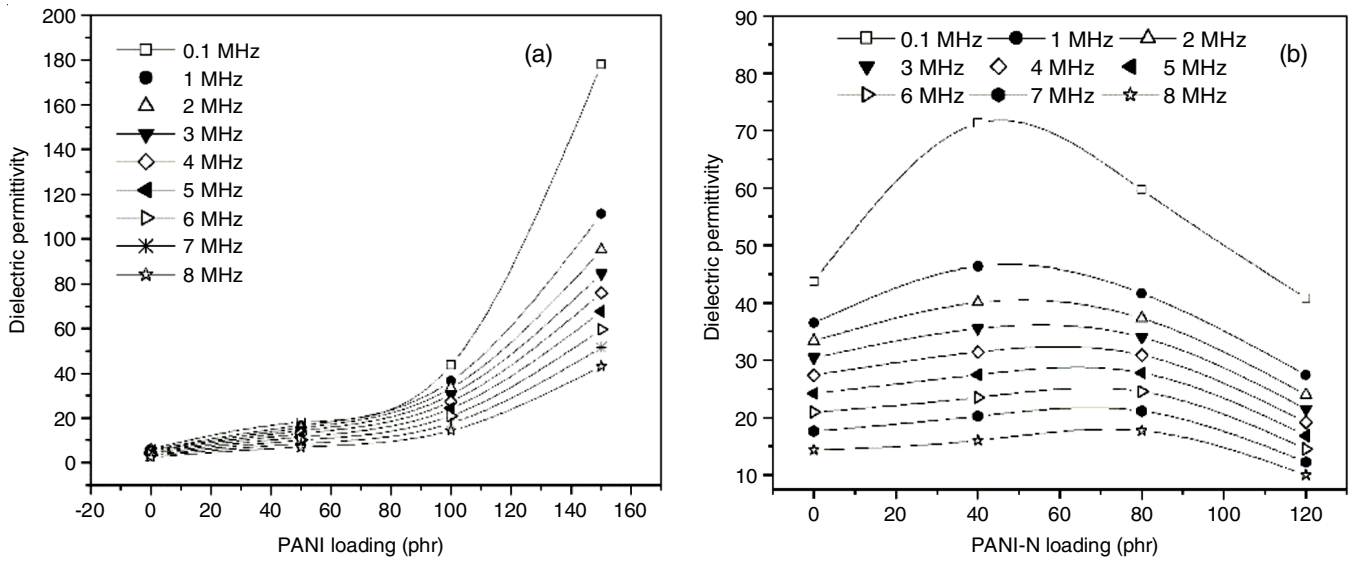


Fig. 4. Variation of dielectric permittivity with (a) PANI loading for P series and (b) PANI-N loading for F series

**Tailoring of dielectric permittivity of the CPCs:** This study aims to establish a correlation between the dielectric constant of the composites and that of PANI and CR gum. The dielectric permittivity of the composites is effectively predicted using diverse mixture equations [36], considering the composite as a blend of multiple elements. Empirical equations, with slight modifications, proved to be instrumental in predicting the permittivity of the CPCs by considering the host matrix and the conducting polymer. One such equation used was the Lichtenecker equation [36-38]. The treatment of this equation has been reported earlier [34].

$$\log \epsilon_{\text{eff}} = (1 - V_f) \log \epsilon_m + V_f \log \epsilon_f \quad (1)$$

where  $\epsilon_{\text{eff}}$  denotes the dielectric constant,  $V_f$  signifies the volume fraction of the filler and  $\epsilon_m$  and  $\epsilon_f$  denote the dielectric constant of the matrix and filler, respectively.

In CPCs, the initial disparity between these equations and experimental data could be attributed to the potential formation of agglomerates of PANI within the matrix. As a result, modified equations were applied, taking into account the uniform dispersion of spherical conducting particles within the non-conductive medium.

$$\log \epsilon_{\text{eff}} = (1 - W_f)(\log \epsilon_m)^k + W_f(\log \epsilon_f)^k \quad (1)$$

$$\log \epsilon_{\text{eff}} = \log \epsilon_m \left( \frac{1 - 2W_f \left( \frac{\epsilon_m - \epsilon_f}{2\epsilon_m + \epsilon_f} \right)}{1 - W_f \left( \frac{\epsilon_m - \epsilon_f}{2\epsilon_m + \epsilon_f} \right)} \right) + \log k \quad (2)$$

$$\log \epsilon_{\text{eff}} = \log \frac{\epsilon_m \epsilon_f}{\epsilon_m y_2 + \epsilon_f y_1} + \log k \quad (3)$$

where  $w_f$  represents the weight fraction of the filler and  $k$  is an empirical constant.

The computed logarithmic values were plotted against  $\log f$  and the resulting plots, depicted in Fig. 5, were compared with the experimentally determined values. Notably, with increasing loading, the calculated value of permittivity begins to deviate from the measured values, particularly at lower frequencies. Within the composites, the value of  $k$  is observed to span from 0.23 to 0.88. The slight discrepancies in the coefficient  $k$  and the deviation from measured values under high loads can be attributed to differences in the distribution of the filler and the complex interactions occurring between the filler and the matrix.

**AC conductivity:** AC conductivity was computed from the dielectric data using eqn. 4:

$$\sigma_{\text{AC}} = 2\pi f \tan \delta \epsilon_0 \epsilon_r \quad (4)$$

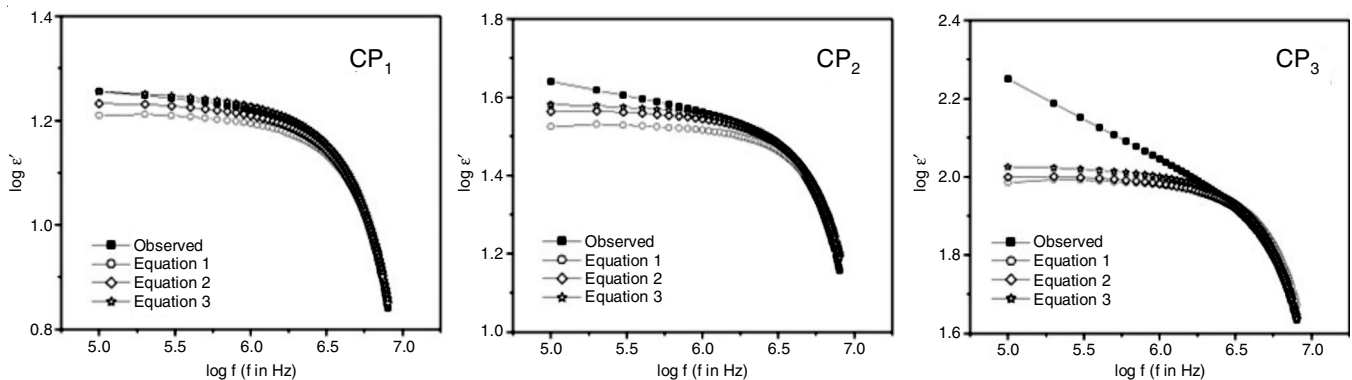


Fig. 5. Log permittivity vs. log frequency for the observed and computed permittivity of the composites

The frequency and temperature variations of AC conductivity distinguish between various conduction mechanisms. The frequency-dependent variation of AC conductivity, indicated a decrease at higher frequencies due to the Maxwell-Wagner two-layer or the heterogeneous model [34]. Hopping of charge carriers is identified as the main mechanism for AC conduction in pure PANI. Conductivity increased with the temperature due to the increase in drift mobility of thermally activated electrons.

**Frequency dependence of the CPCs:** Fig. 6 illustrates the influence of frequency on  $\sigma_{AC}$ , of CR gum and the composites at 303 K. The various ingredients added during vulcanization gives gum vulcanizate its conductivity. CR is a semi-crystalline polymer that resembles the continuous matrix of an amorphous polymer with randomly dispersed crystalline patches. The presence of crystallite centers often leads to a reduction in conductivity. The elastomer can be conceptualized as a double layer comprising crystalline and non-crystalline centers, akin to the Maxwell-Wagner two-layer concept. Little ion mobility through the crystalline zone occurs in the ionic conduction scenarios, while in electronic conduction scenarios,

the crystalline-amorphous boundary may serve as an area of entrapment. At lower frequencies, the interface might be more active. As frequency increases, more ions traverse the interface, consequently enhancing the conductivity. There is a frequency limit where the applied frequency surpasses the hopping rate, resulting in a decline in conductivity.

The conductive behaviour exhibited by the CPCs, varying PANI and PANI-N loadings, mirrors that of gum vulcanizate. The conductivity follows a pattern of increasing, reaching a maximum and subsequently decreasing. A higher rate of the conductivity growth was recorded for the CPCs compared to gum vulcanizate.

**Effect of temperature on the CPCs:** In Fig. 7, the influence of temperature on the  $\sigma_{AC}$  of the CPCs is presented. The conductivity of composite CP<sub>2</sub> exhibits a slight increase with increasing temperature. However, with the elevation of frequency, the conductivity experiences a decrease, eventually stabilizing. Up to 353 K, there is an overall increase in the conductivity, but at higher loading (150 phr), the conductivity starts to decrease.

For composite CF<sub>3</sub>, the conductivity demonstrates an increase until reaching 373 K, after which it begins to decrease.

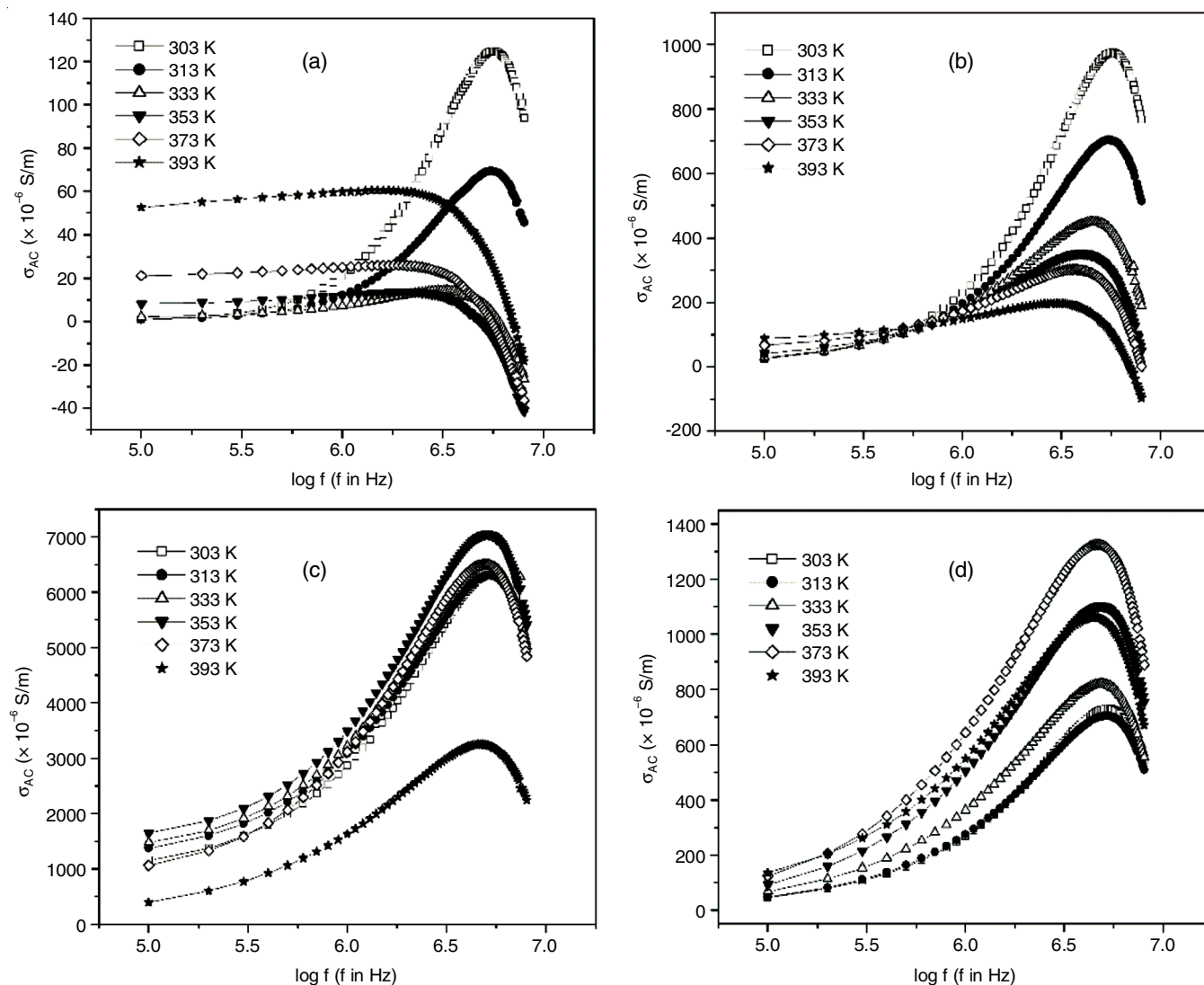


Fig. 6. AC conductivity vs. frequency of (a) CR gum vulcanizate, (b) CP<sub>2</sub>, (c) CP<sub>3</sub> and (d) CF<sub>3</sub>

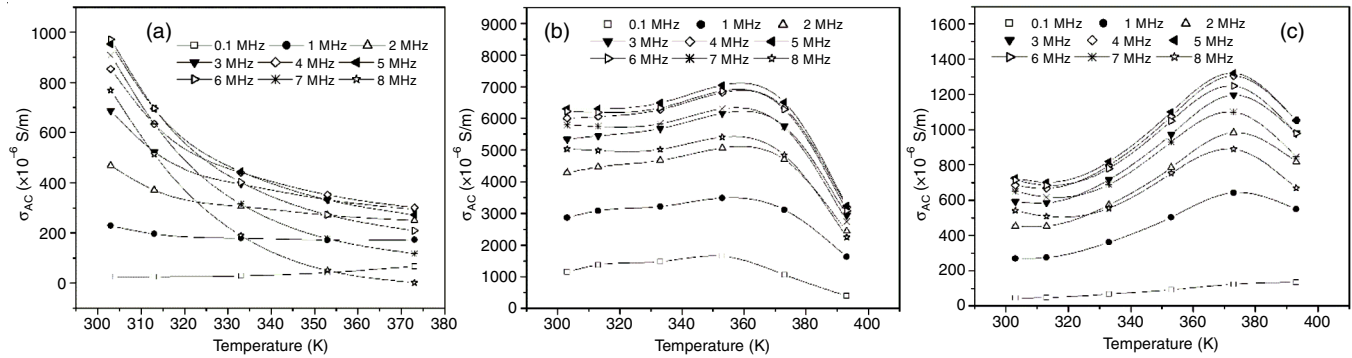


Fig. 7. Effect of temperature on the AC conductivity of (a) CP<sub>2</sub>, (b) CP<sub>3</sub> and (c) CF<sub>3</sub>

The initial upswing in conductivity is attributed to an improvement in conduction by hopping. At exceptionally high temperatures, the mobility of the polymer chain increases, leading to a subsequent reduction in conductivity. The F-series composite displays a higher temperature cutoff point for the onset of conductivity deterioration than the P-series composite.

**Loading dependence of the CPCs:** Fig. 8 illustrates the impact of loading on  $\sigma_{AC}$  of the composites. As expected, conductivity increases with higher PANI content, with a more pronounced increase at higher frequencies. Similar to dielectric permittivity, the AC conductivity of F-series composites increases with PANI-N loading but experiences a decline at higher loadings. At 5 MHz, the highest conductivity of  $6.20 \times 10^{-3}$  S/m was recorded for CP<sub>3</sub>, slightly surpassing values obtained for analogous NR-based CPCs reported earlier [34]. The F-series composite achieved a conductivity of  $1.01 \times 10^{-3}$  S/m (CPC CF<sub>3</sub>) for 120 phr loading.

**Conclusion**

This work successfully explores the dielectric properties of PANI-based composites with chloroprene rubber (CR), across a 303 to 393 K and 0.1 to 8 MHz. The dielectric permittivity of PANI/CR and PANI/PANI-N/CR composites displays minor changes in response to changes in frequency, temperature and

loading. As the frequency increases, the dielectric permittivity of the composites decreases owing to a reduction in interfacial polarization, while it increases with the loading for both series. The permittivity peaks and subsequently decreases with an increase in temperature. At 303 K and 0.1 MHz, a CPC with 150 phr PANI loading achieves a dielectric permittivity of 177, nearly double the value for previously reported for natural rubber. The effective permittivity of CPCs can be predicted using well-established empirical equations that explain the dielectric dispersion. The AC conductivity is significantly elevated in CR-based composites when contrasted with NR-based composites, attaining a peak value of  $6.20 \times 10^{-3}$  S/m at 5 MHz for CP<sub>3</sub>. The main conduction mechanism in CPCs is charge carrier hopping. The AC conductivity analysis reveals the impact of temperature and frequency on the conduction mechanisms within the composites. The observed correlations between the mechanical and the dielectric properties suggest a tailored approach for achieving CPCs with enhanced overall performance. This work contributes valuable insights into the intricate interplay between mechanical and dielectric attributes in conducting polymer composites, paving the way for their potential applications in advanced electronic devices and technological advancements.

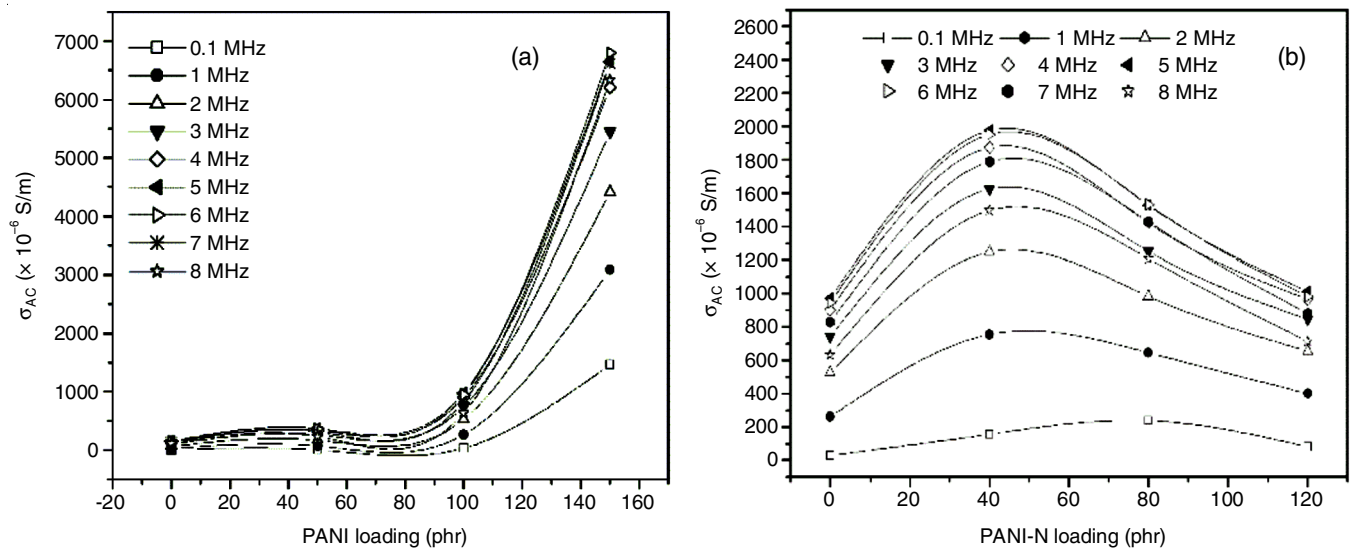


Fig. 8.  $\sigma_{AC}$  vs. loading for (a) P series and (b) F series

### CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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