**Characterisation of the dielectric properties of rubber latex from 0.5 to 33 GHz**

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**Abstract**

This paper presents a detailed characterisation of the dielectric properties of rubber latex over the entire microwave frequency range (0.5-33 GHz), for samples with a range of dry rubber contents and over the temperature range 10-40 °C. The relaxation processes observed are analysed and compared to pure water, as modelled with the Debye equation. It is shown that two relaxation processes exist in rubber latex, with one of these attributed to the presence of bound water molecules. The extended Debye equation is then applied to model the dielectric permittivity. Each relaxation time extracted from the modelling exposes a different physical mechanism in rubber latex. It is believed that this is the most extensive study of the microwave properties of rubber latex yet reported, and that the results are an important step in the development of microwave sensors for determining the quality of rubber latex for agriculture and industrial application.

**Key words:** dielectric measurements, dielectric materials, rubber industry, relaxation time.

**1. Introduction**

It is well known that real part () and imaginary part () of permittivity at microwave frequencies is related to the physical properties (for instance constituents, volume fraction, relaxation time, relaxation frequency, etc.) of a heterogeneous material. A substantial body of research has been published on the application of microwave complex permittivity measurements in determining the properties of various agricultural products (Guo, Wu, Zhu, Wang, 2011); (Julrat, Chongcheawchamnan, Kaoraopaphong, Robertson, 2012); (Khalid, 1991); (Nelson, 1992); (Nelson, 2005); (Nelson & Trabelsi, 2008); (Ragni, Gradari, Berardinelli, Giunchi, Guarnieri, 2006); (Sacilik, Tarimci, Colak, 2006); (Sosa-Morales et al., 2009); (Trabelsi, Krazsewski, Nelson, 1998); (Trabelsi, Nelson, Lewis, 2009); (Trabelsi & Nelson, 2006). Water, a major component, is sensitive to the applied microwave energy and so the measured complex permittivity is influenced by the water content. It is well known that bulk water molecules are dipolar and can polarise readily with the applied electromagnetic field at microwave frequencies. The bulk water molecules, formed in a tetrahedron shape (Eisenberg & Kauzmann, 1969); (Kaatze, 2005); (Sharp, 2001) are linked by the hydrogen bond. The behaviour of water molecules when solids are present is different to that in bulk water. In agricultural products, some water molecules are bound and the overall polarisation characteristic when an external electric field is applied will be influenced by these bound molecules. As the characteristic of bound water is generally different from bulk water (Nandi & Bagchi, 1997), the behaviour of bound water in agricultural products depends on its constituents, volume faction and environment. No single definition can completely describe the characteristics of bound water. However, the bound characteristic can be described by studying the relaxation time, which is related to the bond strength and is determined by measuring the permittivity as a function of frequency.

Measurement of the dielectric properties of rubber latex can be applied to determine the dry rubber content (DRC), which is a quality indicator for latex in industrial centrifugal processing of rubber. Methods for measuring DRC have been briefly reviewed by Julrat, et al. (2012). The latex obtained from rubber trees is a complex biological colloidal liquid consisting of a large water content (50-80%) with typically 25-45% hydrocarbon rubber (dry rubber) and 2-5% non-rubber constituents (Khalid,Hassan, Yusof, 1997). The non-rubber components are, for example, proteins, lipids, carbohydrates, inorganic ions and salts. The proportion of these natural contents depends on the variety, season and environment. The DRC in rubber latex reflects the fraction of dry rubber. Since the non-rubber content is quite small in rubber latex, the DRC can be related to the water content in rubber latex. Understanding the characteristics of bound water in rubber latex is necessary in order to develop a model that enables DRC to be extracted from the microwave measurements, thereby assessing the rubber latex quality.

The characteristics of bound water in rubber latex were first investigated over the frequency range 10-2-106 Hz (Hassan, 1999) by measuring the complex permittivity of rubber latex samples that have different water contents at -30 °C and comparing it to ice. The complex permittivity of bound water was found to lie between that of bulk water and ice, while the relaxation peak depends on the water content in rubber latex. However, there is little knowledge of how bound water affects the complex permittivity of rubber latex at microwave frequencies, where the two key parameters are volume fractions (DRC, water content and non-rubber content) and the ambient temperature,.

In this paper, the complex permittivity of rubber latex is extensively characterised, for the first time, over the full microwave frequency range. The relaxation characteristics of rubber latex with different ambient temperatures and volume fractions are analysed. Knowing the microwave properties of rubber latex is an important step in the development of microwave sensors for determining the quality of rubber latex for agriculture and industrial application. Section 2 describes the experimental setup and conditions for the measurements. In Section 3, the complex permittivity of several rubber latex samples with different DRC percentages is presented, for different conditions, and the Cole-Cole plots as a function of temperature and DRC are obtained. In Section 4, the relaxation processes and activation energy of rubber latex are analysed using a graphical method. In Section 5, the extended Debye equations are applied to model the rubber latex permittivity behaviour.

**2. Experimental setup**

As shown in Fig. 1, the complex permittivity of the rubber latex was determined using the Agilent 85070E dielectric probe kit (Agilent 85070E dielectric probe kit 200 MHz to 50 GHz) with a slim probe form, which is specially designed for liquid measurement applications. The reflection coefficient of the probe immersed in rubber latex was measured and the data transferred via GPIB (General Purpose Interface Bus) to a computer, where the dedicated Agilent dielectric measurement software extracted the complex permittivity. To obtain accurate and reliable measurement results, the dielectric probe kit needs to be calibrated. This was done by using a standard short circuit (series 85070-60004 short for the slim form probe), air, and then reference water, from 0.5 GHz to 33 GHz. The temperature of the water during calibration was recorded and subsequently used by the calibration software.

Each sample was prepared as 40 ml of liquid (30 mm high from the beaker bottom) in a 50 ml beaker with 60 mm height. The slim probe was placed at the centre of the beaker, 20 mm from the beaker bottom. The temperature of the rubber latex sample was measured by a thermometer that was dipped in the sample and positioned close to the probe. The temperature of the sample was controlled by immersing the beaker in a water bath at room temperature of 27°C. The desired temperature of rubber latex sample was achieved by mixing water in the bath with boiling water at 100°C, or cooled water at 0°C. The temperature in the water bath was monitored using a Fluke®62 mini IR thermometer. For each measurement, the controlled temperature liquid sample was left for 20 minutes and carefully stirred to obtain a uniform distribution of temperature and mixture in the liquid sample.

**3. Complex permittivity measurement**

The rubber latex samples were prepared to get different DRC percentages by diluting concentrated rubber latex (about 60% DRC, 38% water, 0.4% ammonia and non-rubber contents for the remainder) with milli-Q® water. Four different DRC percentages of latex samples covering DRC ranges of concentrated and natural fresh latex were prepared. The DRCs of the rubber latex samples were validated immediately after the measurement using the standard oven-drying method (ISO 126:1972 Dry rubber content, DRC).

Figs. 2 shows the Cole-Cole plots of the measured complex permittivity ( and ) of rubber latex samples at four different DRC percentages (60.80%, 45.05%, 37.79% and 23.25%) at 10°C, 15°C 27°C and 40°C. The conductivity region is clearly evident at frequencies below 2.5 GHz. The characteristic of is mainly influenced by the ionic conductivity caused by the non-rubber contents in the rubber latex samples, such as ammonia (ammonium hydroxide). It is shown in this frequency region that decreases with frequency but increases with the ambient temperature, because the conductivity increases with temperature.

It appears that the diameter of the semi-circular part of the plot decreases with DRC for all temperatures. The diameter of the semi-circular part of the Cole-Cole plot is proportional to the relaxation amplitude and also decreases with DRC value for each temperature, implying that the relaxation time of rubber latex decreases with temperature.

The temperature variation of the complex permittivity of low-DRC rubber latex is more prominent than that of high-DRC rubber latex. This is because of the dominant role of bound water in the low-DRC rubber latex. At very low frequency, the temperature dependence of the complex permittivity of the rubber latex is a result of the ionic conductivity. On the other hand, the complex permittivity of the rubber latex in the high frequency region changes little with temperature because the dielectric characteristic of rubber latex in this frequency region is mainly governed by the bound water molecules.

The complex permittivity above 2.5 GHz obviously varies with DRC and becomes less dependent on DRC at high frequency. We believe that this is because the water in the rubber latex is bound and therefore exhibits a different distribution of relaxation times for different DRC values.

# **4. Relaxation time and activation energy**

It is observed from Fig. 2 and from Hasted **(**1973) andKaatze (2005) that the Cole-Cole plots of the rubber latex have similar shape to that of bulk water. Hence, we can apply the same procedure as defined for bulk water to investigate the relaxation characteristic under the assumption that the dielectric properties of the rubber latex are a combination of the dielectric properties of the constituents in rubber latex. For bulk water, the procedure to determine the relaxation time is achieved by modelling the measured complex permittivity with the Debye equation (Haynes & Locke, 1995) as follows;-

(1),

where , is the dielectric permittivity extrapolated to high frequency, is the static dielectric permittivity, is the angular frequency of the applied electric field and is the Debye relaxation time. Consequently, the and of bulk water can be obtained as follows;-

(2a),

(2b).

Since the rubber latex has conductivity at low frequency, as shown in Fig. 2, the ionic conductivity term in rubber latex needs to be included in Equation (2b) as follows;-

(2c).

where is the conductivity of the rubber latex, which can be simply obtained by applying a regression technique. From Equation (2a) and (2c), the relationship between and can therefore be expressed in terms of either or (Connors, 1990) as follows:-

(3).

In Equation (3), can be characterised as a linear function of . The relaxation time is related to the slope, where is the intersection on the axis of the Cole-Cole plot. Hence, the Debye relaxation time () can be determined using a graphical method.

Fig. 3 shows the graphical analysis for the complex permittivity of rubber latex samples and bulk water at 10 °C15 °C, 27 °C and 40 °C. The plotted data from left to right represents the complex permittivity from 0.5-33 GHz . The bulk water data can be completely characterised with a single slope function, illustrating that only one relaxation process is present in the bulk water over the measured frequency range. The straight lines shown in Fig 3 separate two regions, indicating that two relaxation processes exist. Each region is characterized by the slope of the straight line fit. Each slope was determined by applying linear regression analysis with Equation (3). The obtained correlation coefficients *R*2 were in the range of 0.96 to 0.99.

The relaxation time obtained from the graphical analysis using Equation (3) is plotted with error bars for standard deviation in Fig. 4. Each relaxation time is computed from the graph slope as shown in Fig. 3. The two relaxation times ( and ) imply the existence of two mixing constituents in the rubber latex. In Fig. 4 (a), the relaxation times increase with DRC, showing that the bound water molecules in rubber latex take a longer time than bulk water molecules to align with the field. On the other hand, shown in Fig. 4 (b) decreases with DRC which is opposite to the previous case.

The activation energy parameter is applied in this paper to investigate the molecular mechanisms in rubber latex. Theoretically, the relaxation time is related to the activation energy () by using the Arrhenius equation (Hasted, 1973) as follows:-

(4a).

Using the logarithmic form, one can write Equation (4a) as:

(4b).

where is pre-exponential factor, is Boltzmann’s constant (8.314 J mol-1), and is the temperature in Kelvin. In this case, the term can be determined from the slope of the plot of versus of relaxation time in Fig. 4.

From Fig. 5, the of rubber latex at various DRCs can be determined, and the results are listed in Table 1. The calculated of water from Equation (4) is 18.96 kJ mol-1, which closely agrees with that reported by Chaplin (2007). The value of of 18.96 kJ mol-1 is related to the energy of hydrogen bonding of O\_H...O in the bulk water. In the relaxation process represented by , the of rubber latex (from 14.91 to 9.36 kJ mol-1) decreases with percentage of DRC (from 23.25 to 60.80%). This can be attributed to the characteristic of hydrogen bonding of N\_H...O, due to the presence of ammonium hydroxide in the rubber latex. For the relaxation process represented by , (from 17.18 to 21.09 kJ mol-1) increases with DRC (from 23.25 to 60.80%) and is slightly higher than bulk water. We believe that this is related to the bound water molecules in rubber latex that have higher bond strength than bulk water molecules.

# **6. Dielectric Model for Rubber Latex**

The characteristics of the relaxation time in rubber latex as shown in Fig. 4 can be related to different polarisation types and mixing constituents. This can then confirm that multiple relaxation processes exist in rubber latex. To demonstrate this, the extended Debye equations for the double relaxation processes obtained in Fig. 5 can be modelled as follows;-

(5),

where and are relaxation amplitude of the relaxation time region (Buchner, Baar, Fernandez, Schrodle, Kunz, 2005); (Stuerga, 2006), is electrical conductivity, is static permittivity and is the dielectric permittivity at high frequency. Next, nonlinear regression analysis was applied with Equation (5) to predict , , , and . The initial value of was chosen to be small (0.5×1012). The initial value of is equal to 5, which is the dielectric permittivity of water at very high frequency. The initial value of was set to be twice that of . The initial values of and were 5 and 10, respectively. For each and , the values , obtained in Fig. 4 and the measured were substituted into Equation (5). Nonlinear regression analysis was performed by setting the parameter ranges which are , , , and . The goal was to obtain the highest value of *R*2. Table II shows the model parameters and the R2 value. It is shown that changes only with temperature. , and change with DRC, and changes with DRC and temperature. The modelling results, describing the complex permittivity of the rubber latex using the relaxation time obtained from Equation (3), are shown in Fig. 6. From the R2 results, it is shown that the double relaxation model given in Equation (5) can accurately describe the dielectric properties of rubber latex.

**7. Conclusions**

The complex permittivity of rubber latex samples with various DRCs has been extensively characterised over the frequency range 0.5-33 GHz for an ambient temperature range of 10-40 °C. It is found that the of rubber latex increases with water content and exhibits both frequency and temperature dependency, like bulk water. Below 2.5 GHz, the value of is dominated by ionic conductivity, which is related to the non-rubber constituents. The results show that the of rubber latex is mainly governed by the water content. Consequently, determining the DRC value for a given rubber latex sample can be accomplished by measuring only . On the other hand, the imaginary part, , can indicate non-rubber content in the sample. By measuring complex permittivity for a range of frequencies and temperatures, it is found that the complex permittivity at low frequency is more frequency-dispersive than in the high frequency region. The relaxation process is analysed by using a graphical method that is based on the Debye equation. The results confirm that bound water is present, with behaviour different to bulk water, and that multiple relaxation times are observed due to the presence of non-rubber constituents and ammonium hydroxide. An extended Debye equation has been used to model the dielectric properties of rubber latex and shows a good fit to measured data. In conclusion, the extensive complex permittivity measurements and modelling results presented in this paper give a better understanding of the properties of rubber latex at microwave frequencies, and this is important for the future development of microwave sensors for use in agriculture and industrial processing.

**Acknowledgment**

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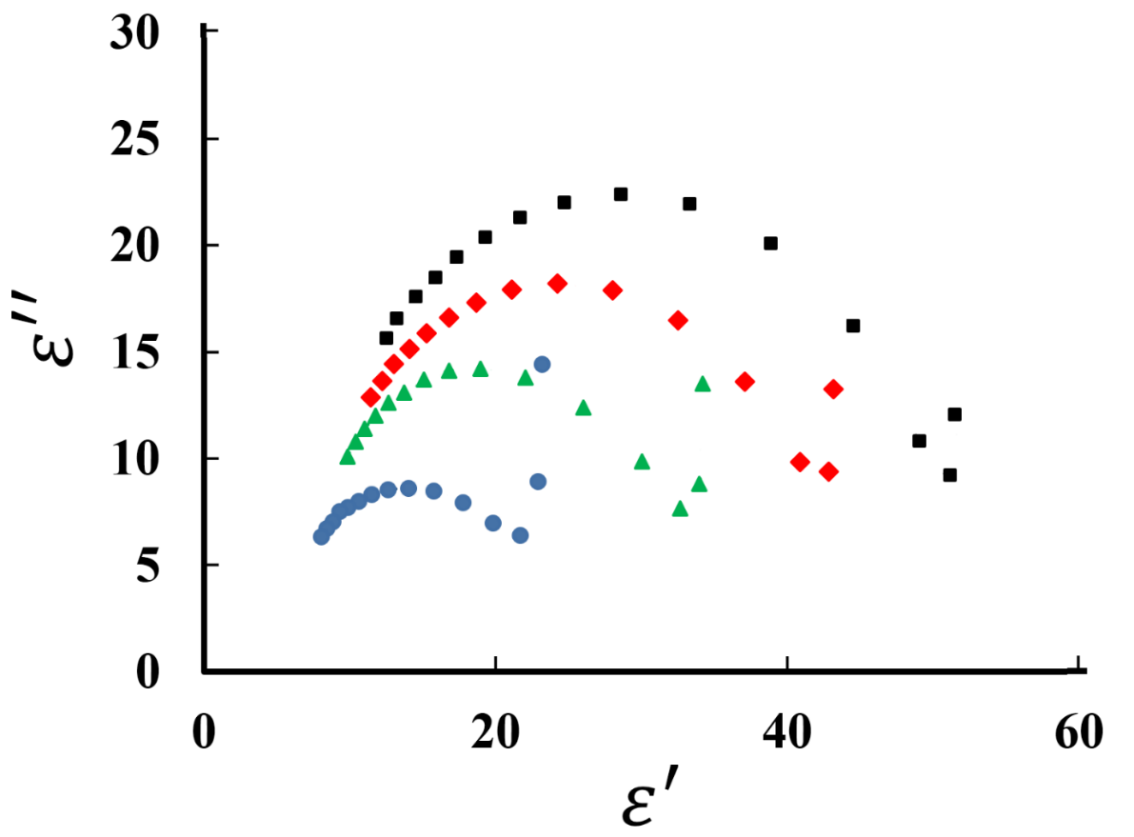
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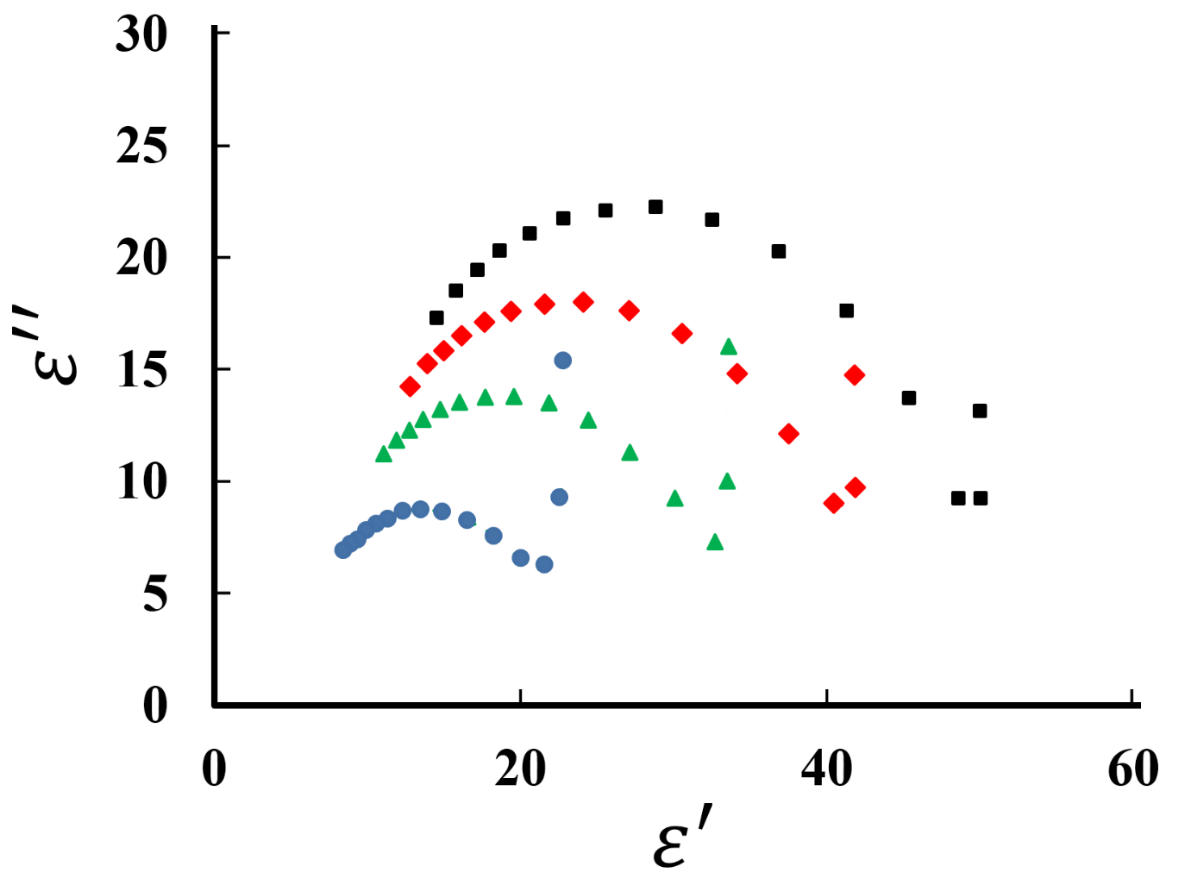
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Figure 1. Experimental setup

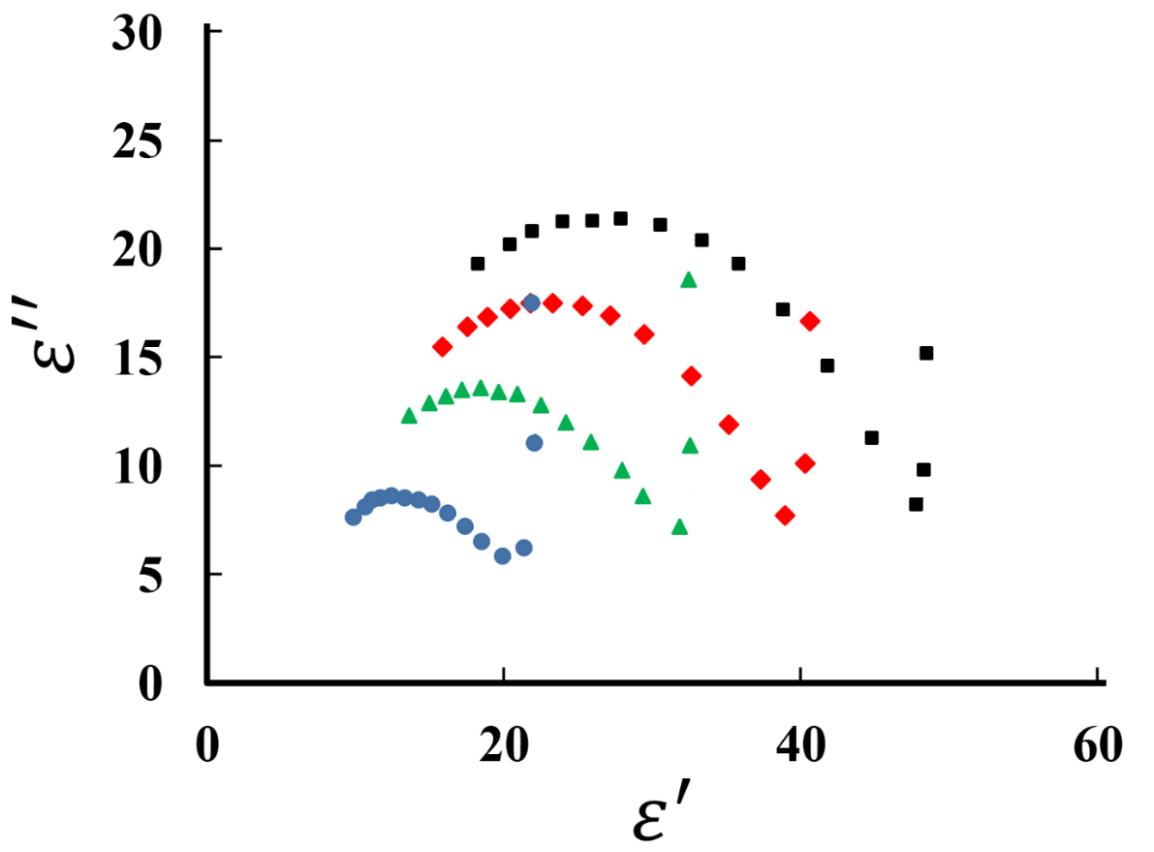
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Figure 2. Cole-Cole plots of rubber latex samples from 0.5-33 GHz for DRC 60. 80% (), 45.05% (),37.79% () and 23.25% () at temperature (a) 10 °C, (b) 15 °C, (c) 27 °C, and (d) 40 °C

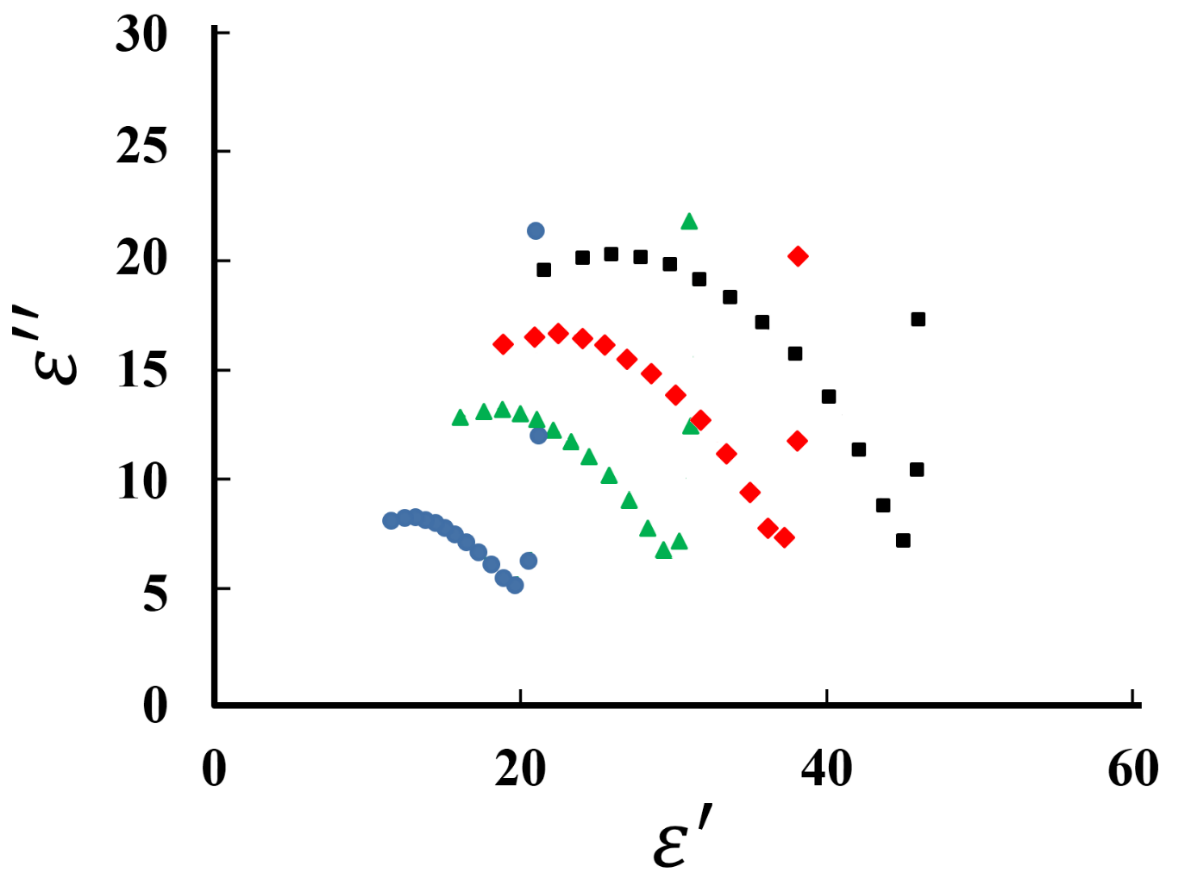
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(b)

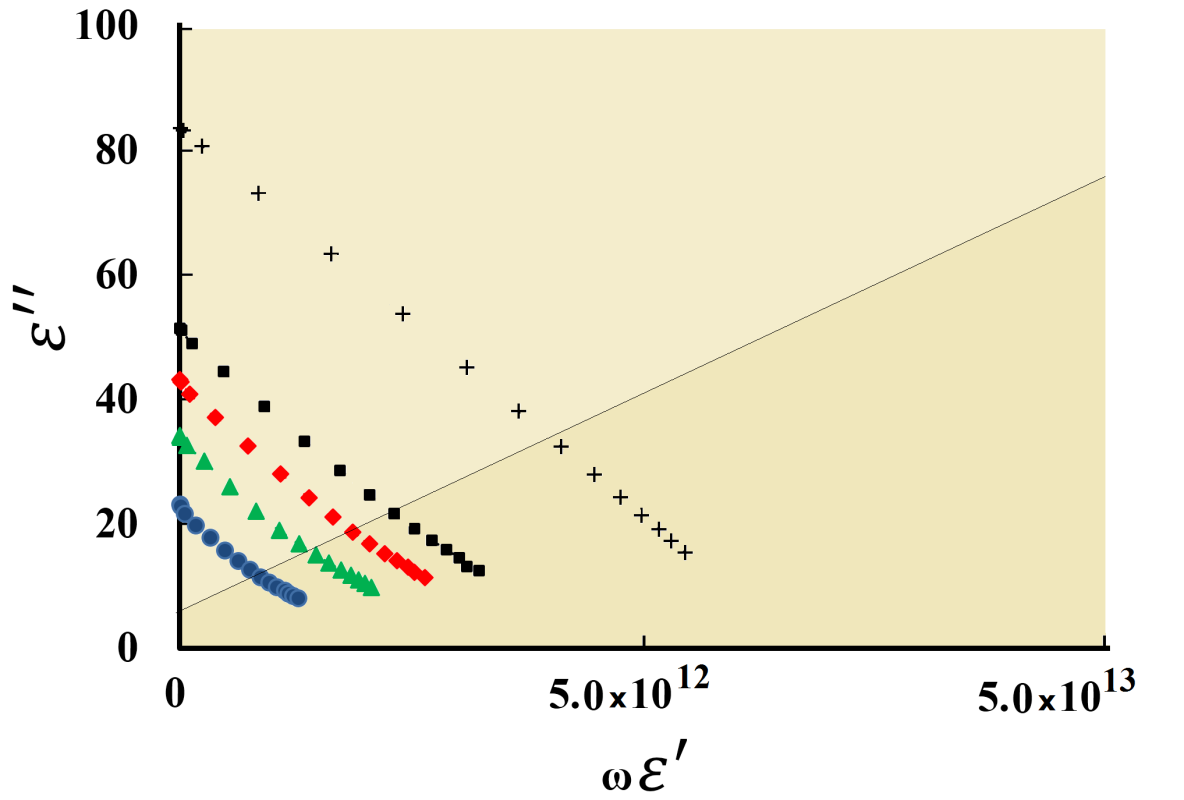


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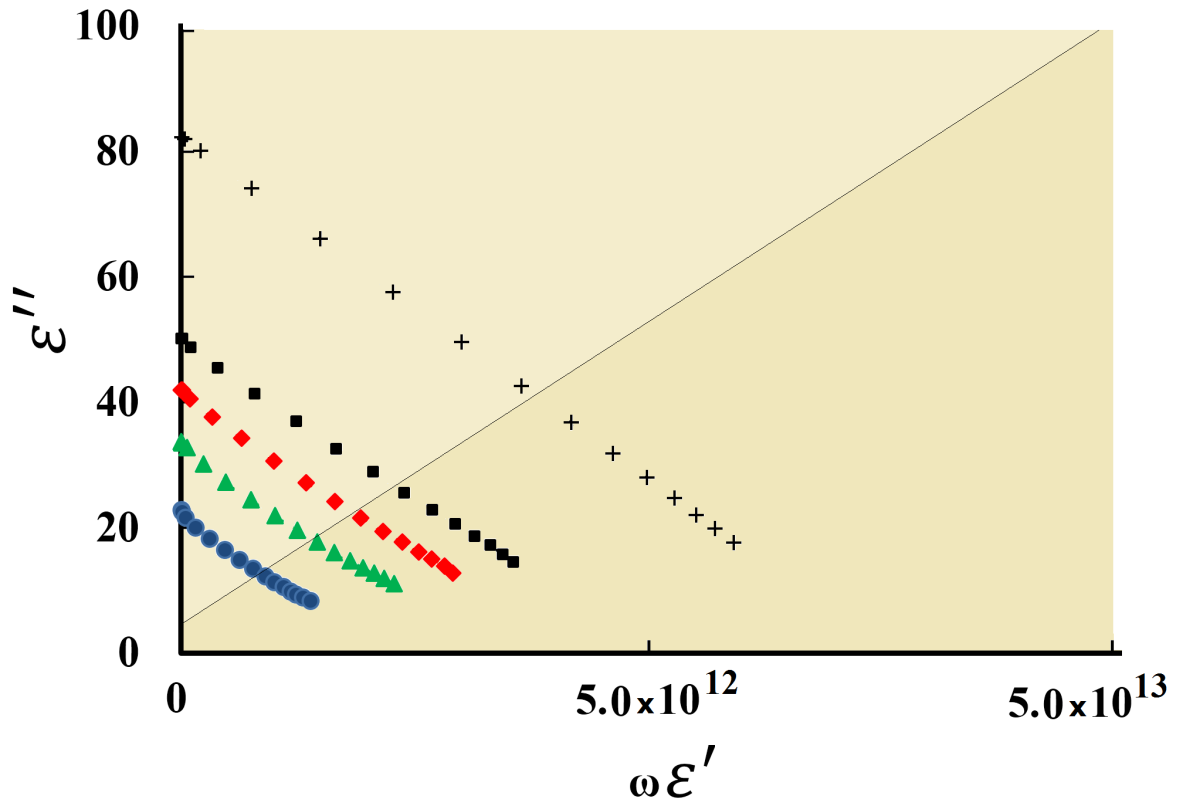


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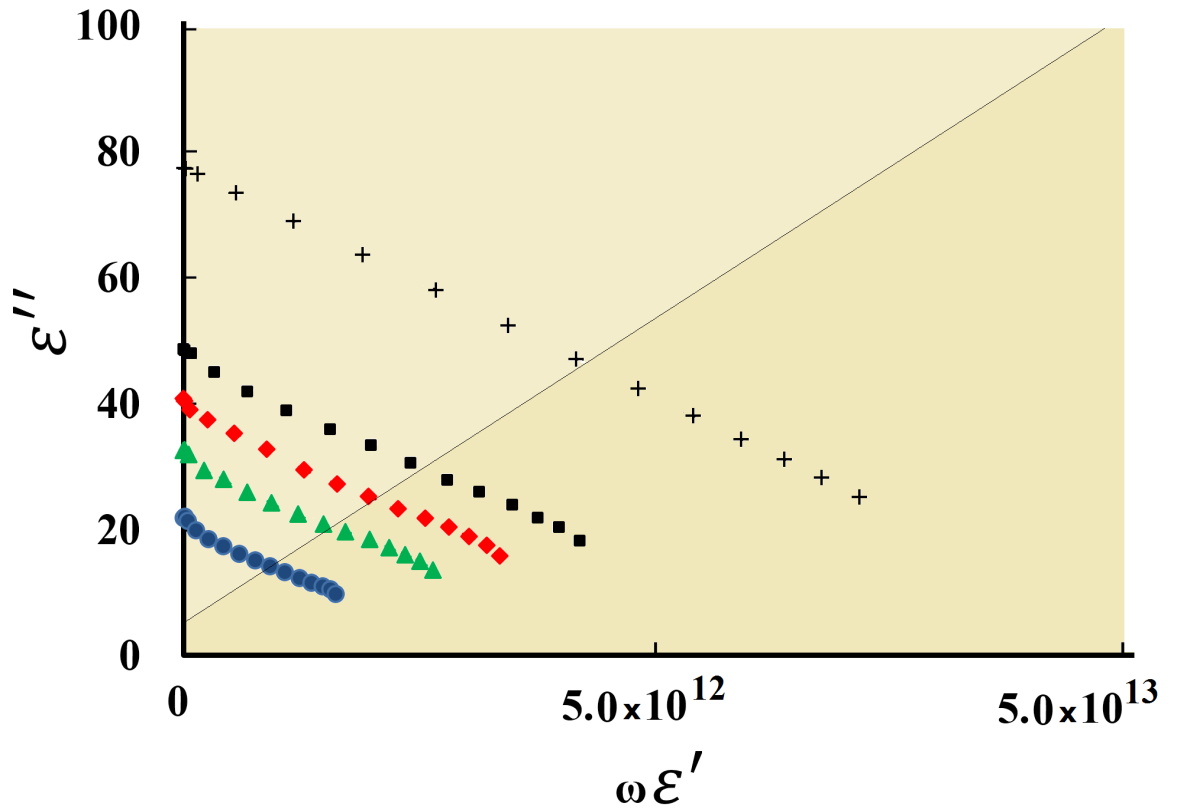
Figure 3. Analysis of complex permittivities of rubber latex samples for DRC 60.80% (), 45.05% (), 37.79% (), 23.25% (), and water () using Equation (3) at temperature (a) 10 °C, (b) 15 °C, (c) 27 °C, and (d) 40 °C



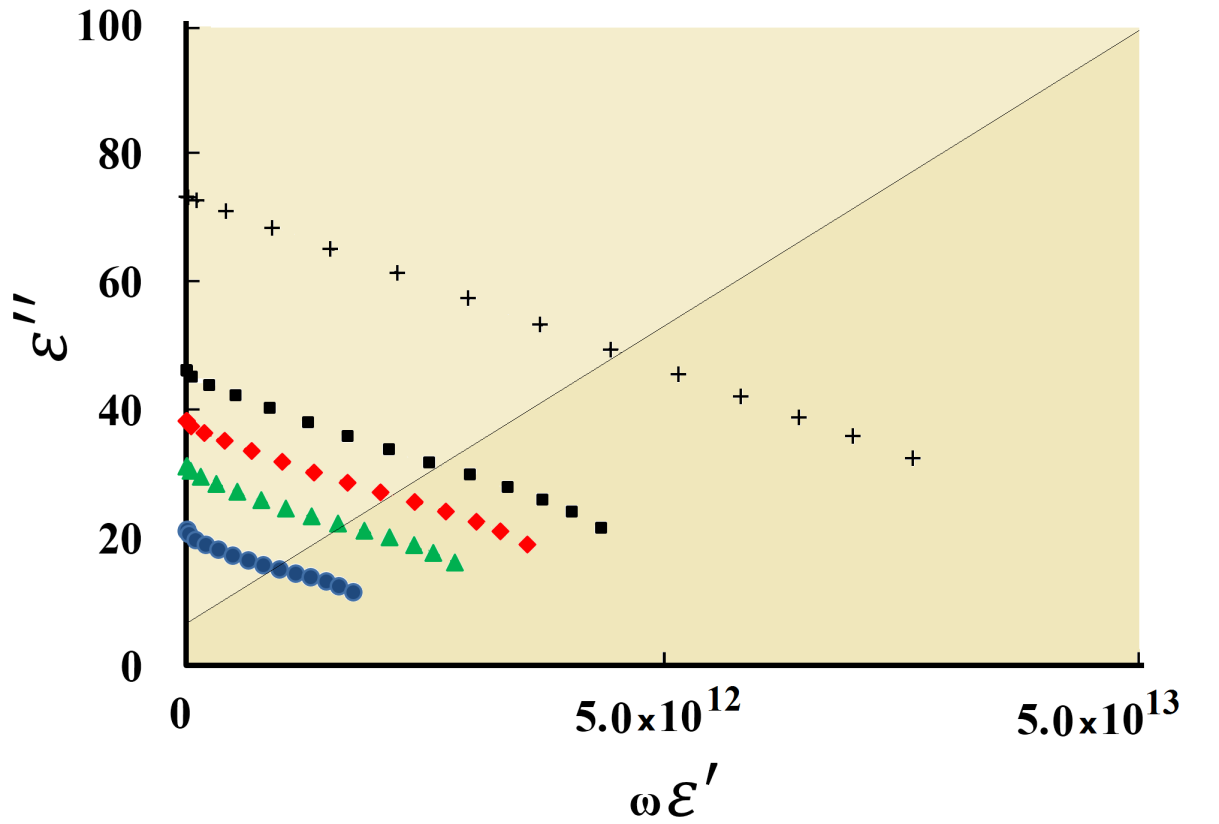
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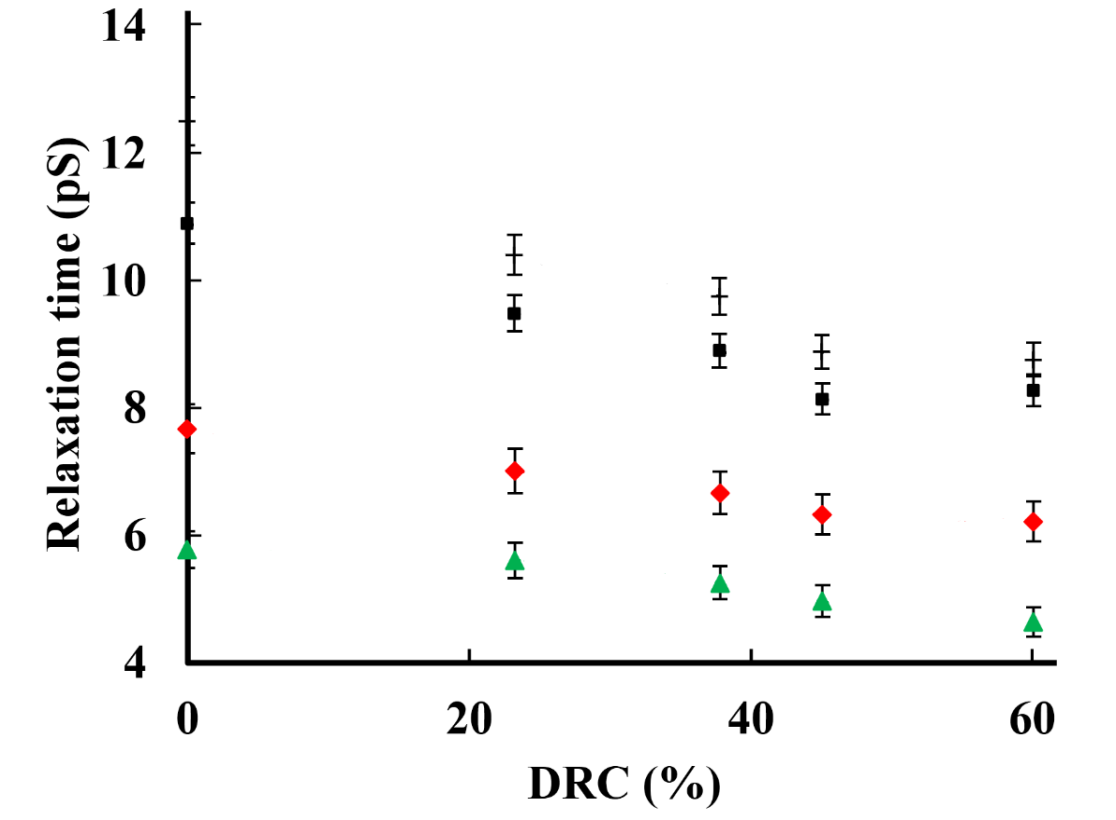


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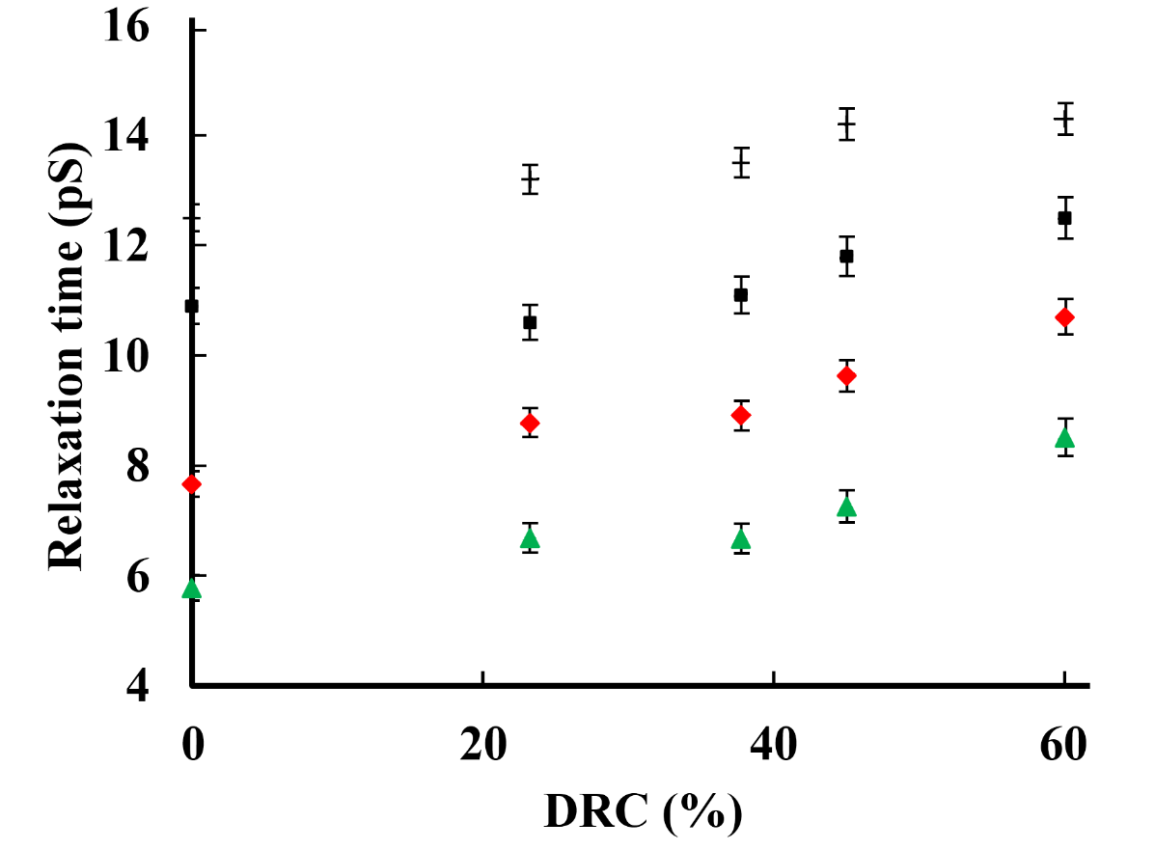


(d)

Figure 4. The relaxation time of rubber latex samples obtained from Equation at temperature 40.0 °C(),27.0 °C (),15.0 °C ( ), and 10.0 °C (), (a) and (b)

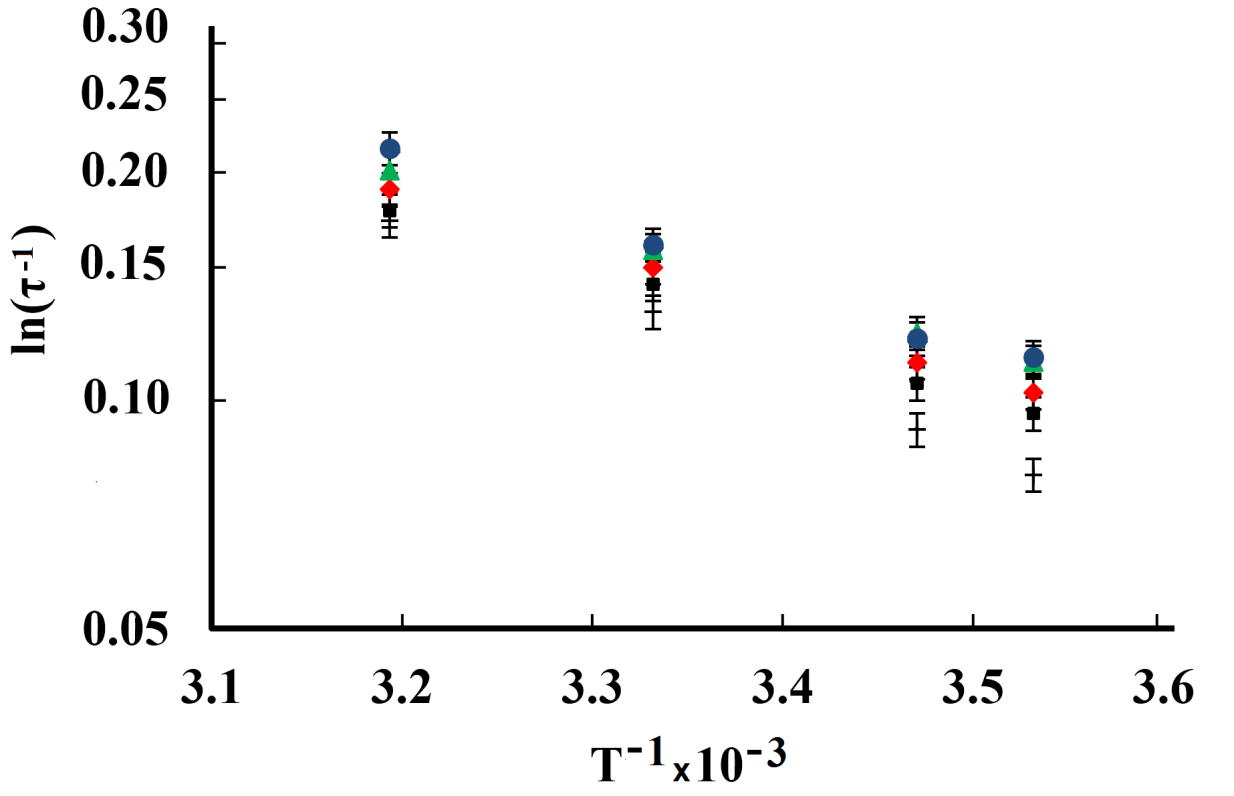


(a)

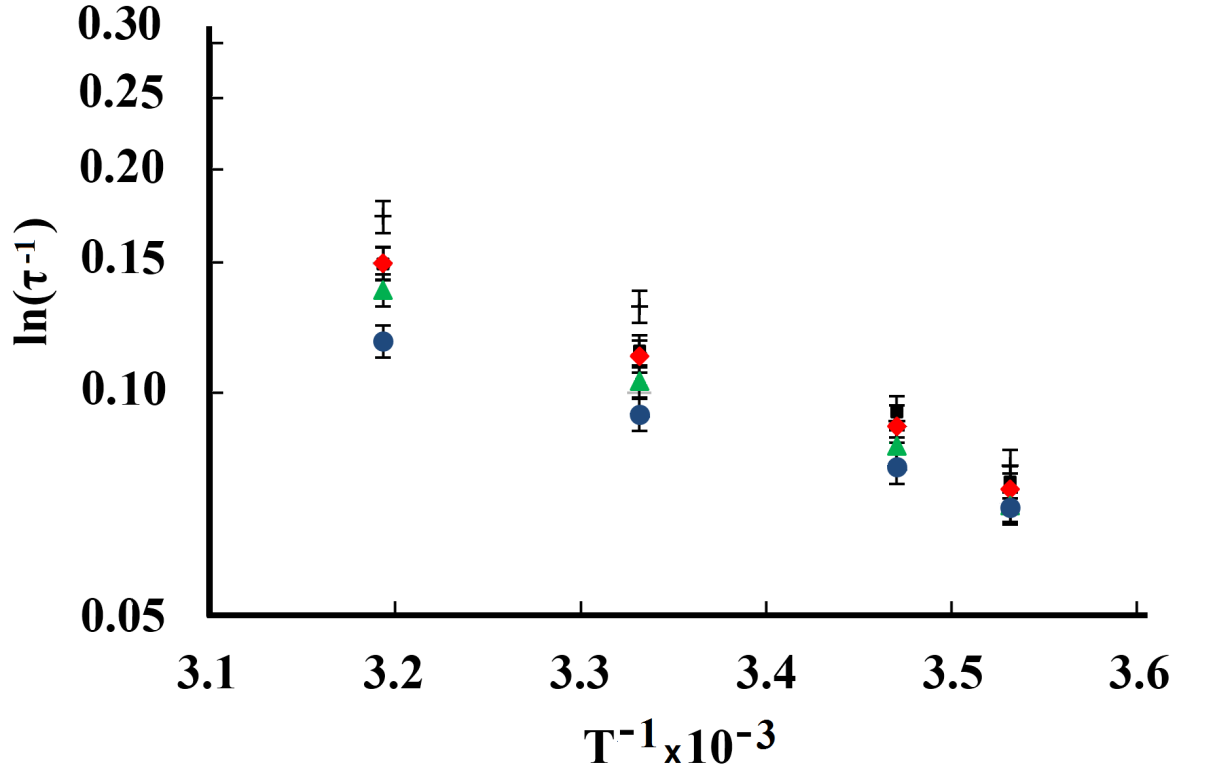


(b)

Figure 5.Activation energy of rubber latex samples obtained from Equation (4) at DRC , (a) and (b)

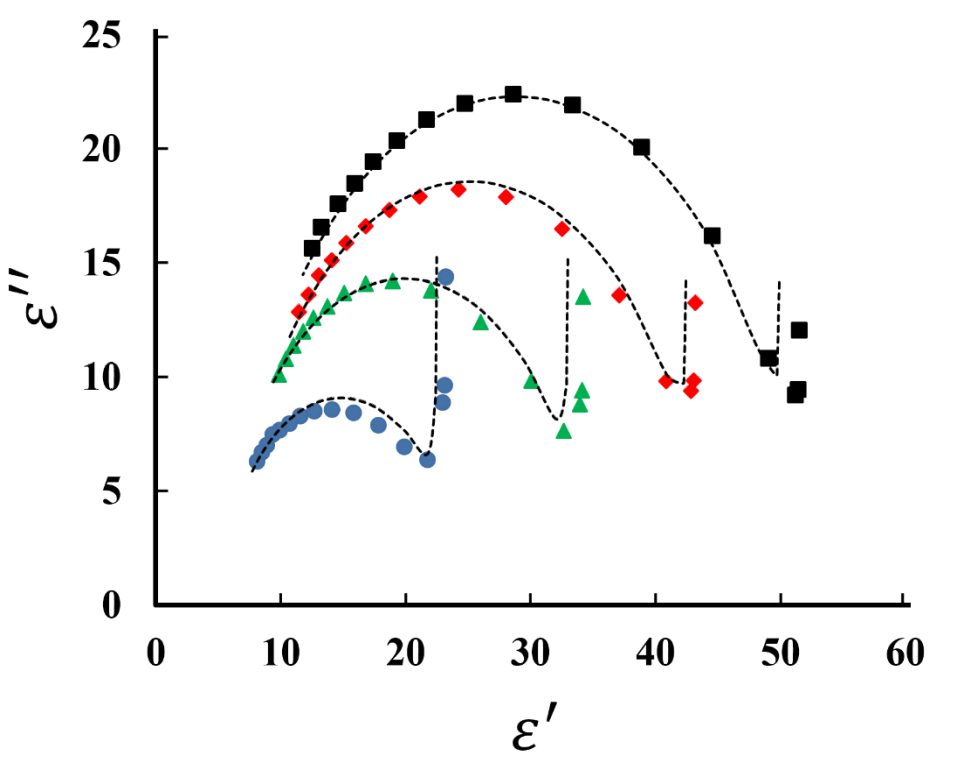


(a)

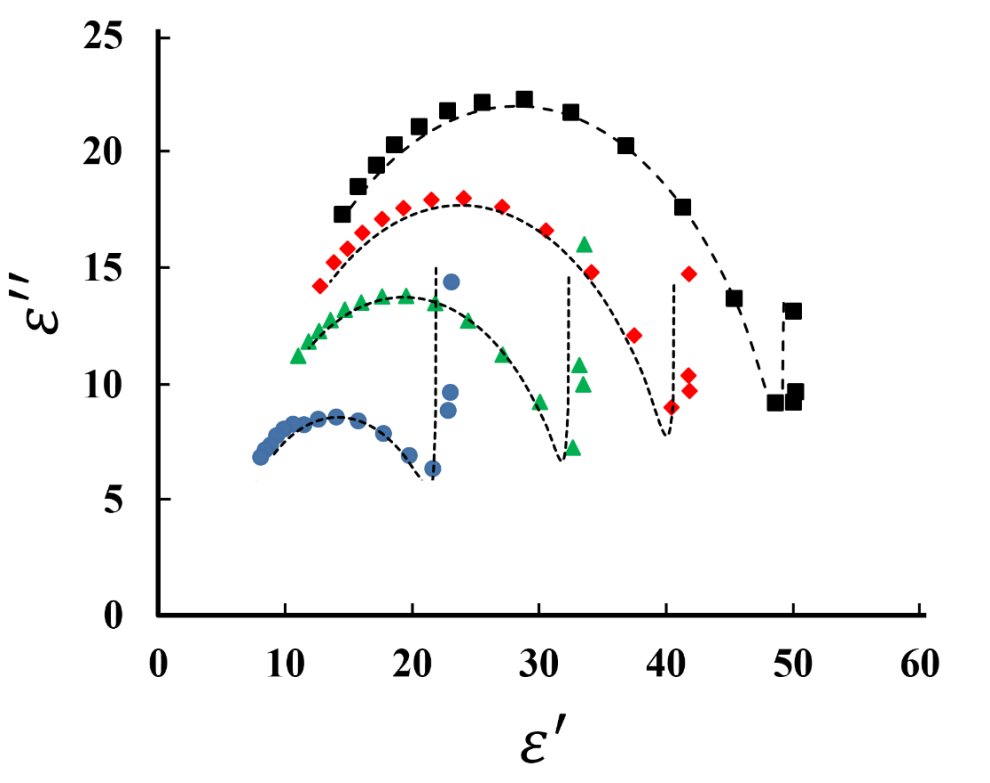


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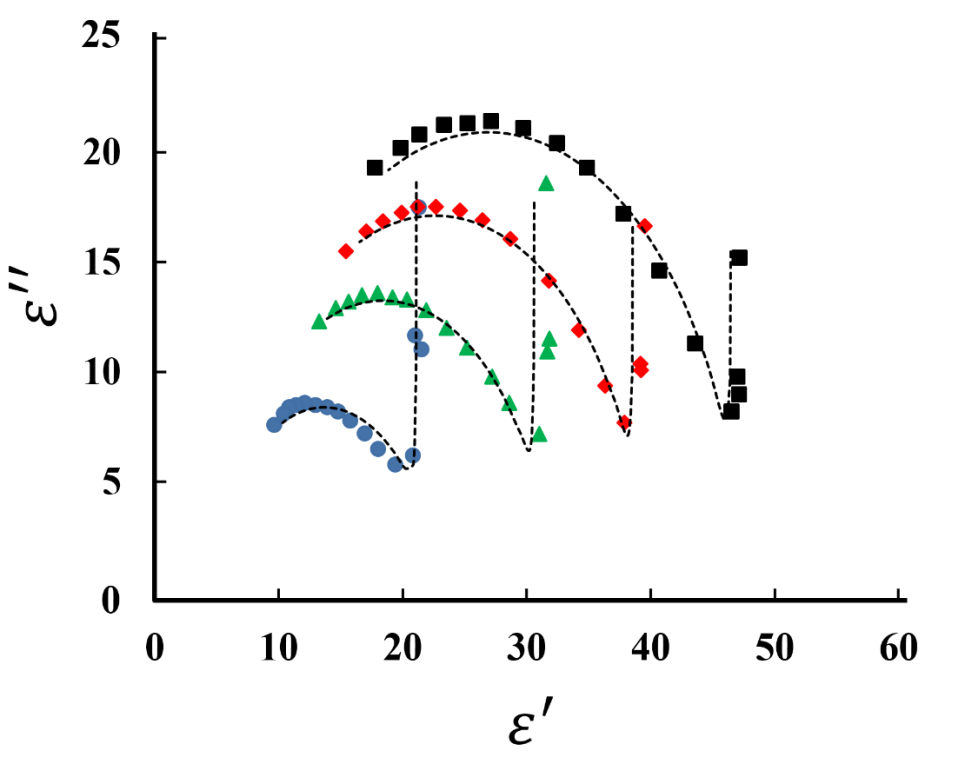
Figure 6 Fitting data from Equation (5) (Dash line) comparing with the complex permittivity data of rubber latex samples at DRC 60.08% (), 45.05% (), 37.79% (), and 23.25% (), at temperature (a) 10 °C, (b) 15 °C, (c) 27 °C, and (d) 40 °C.



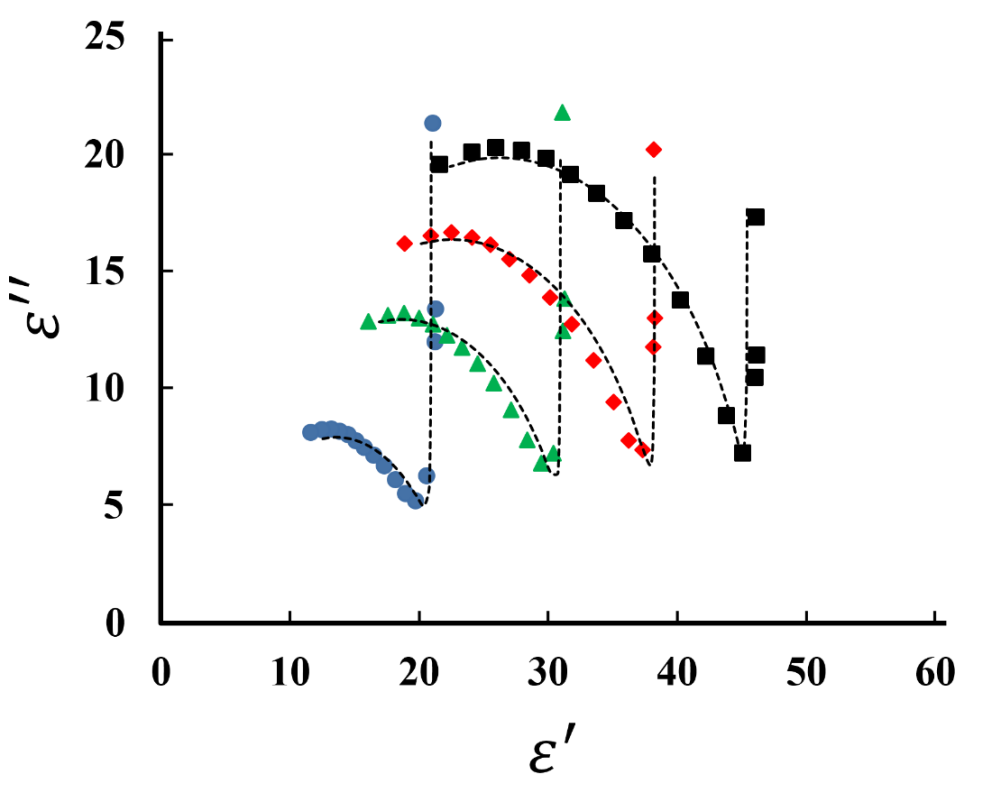
(a)



(b)



(c)



(d)

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Table 1 Activation energy *E*a of different DRC rubber latex samples

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Relaxation process | Activation Energy *E*a (kJ mol-1) | | | |
| DRC@23.25% | DRC@37.79% | DRC@45.05% | DRC@60.80% |
|  | 14.91±0.50 | 14.91±0.50 | 13.24±0.50 | 9.36±0.50 |
|  | 17.18±0.50 | 18.21±0.50 | 18.26±0.50 | 21.09±0.50 |

Table 2 Model parameters in Equation (5)

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Temp. (°C) | DRC (%) | Double relaxation model | | | | | | | R-square |
| (1012 S.m-1) |  |  |  |  | (ps) | (ps) |
| 10 | 60.80 | 0.45 | 5.50 | 10.00 | 4.50 | 12.30 | 14.30 | 8.76 | 0.96 |
| 45.05 | 0.45 | 5.60 | 10.50 | 4.90 | 22.50 | 12.40 | 8.88 | 0.92 |
| 37.79 | 0.50 | 6.20 | 11.00 | 4.80 | 31.00 | 13.50 | 9.75 | 0.94 |
| 23.25 | 0.45 | 6.40 | 12.00 | 5.60 | 38.50 | 13.20 | 10.40 | 0.97 |
| 15 | 60.80 | 0.50 | 5.50 | 10.00 | 4.50 | 12.00 | 12.50 | 8.28 | 0.96 |
| 45.05 | 0.50 | 5.60 | 10.50 | 4.90 | 22.00 | 11.80 | 8.14 | 0.93 |
| 37.79 | 0.50 | 6.20 | 11.00 | 4.80 | 29.80 | 11.10 | 8.90 | 0.98 |
| 23.25 | 0.50 | 6.40 | 12.00 | 5.60 | 37.50 | 10.60 | 9.49 | 0.99 |
| 27 | 60.80 | 0.63 | 5.50 | 10.00 | 4.50 | 11.80 | 10.70 | 6.22 | 0.99 |
| 45.05 | 0.62 | 4.90 | 10.50 | 4.90 | 21.10 | 9.63 | 6.33 | 0.98 |
| 37.79 | 0.60 | 6.20 | 11.00 | 4.80 | 28.80 | 8.91 | 6.67 | 0.98 |
| 23.25 | 0.60 | 6.80 | 12.00 | 5.20 | 36.00 | 8.78 | 7.01 | 0.99 |
| 40 | 60.80 | 0.70 | 5.50 | 10.00 | 4.50 | 11.00 | 8.51 | 4.64 | 0.99 |
| 45.05 | 0.70 | 5.60 | 10.50 | 4.90 | 20.50 | 7.26 | 4.97 | 0.98 |
| 37.79 | 0.70 | 6.20 | 11.00 | 4.80 | 27.30 | 6.68 | 5.26 | 0.97 |
| 23.25 | 0.70 | 6.40 | 12.00 | 5.60 | 33.50 | 6.69 | 5.61 | 0.99 |