Dielectric and Microwave Properties of Cured and Uncured Natural Rubber Composites

[Dr. Julie Charles]

***Abstract*—The backbone of Natural rubber (NR), polyisoprene, is derived from the polyacetylene backbone through the saturation of every other double bond. Polyisoprene is a potential candidate for materials used in various devices: solar cells, LEDs, and FETs. It is also used in electrically energized line services as electrical energy utilities without any energy breakdown. Therefore, analysis of dielectric property becomes inevitable since they determine the end electrical applications, per se, insulation. Present work is aimed at the determination of dielectric constant of natural rubber by using the method proposed by Robert and Von-Hippel. The effect of microwave frequency, namely 9.6 GHz and 26 GHz, as well as chemical modification brought about by vulcanization and reinforcement on dielectric constant values was probed. Impedance spectroscopy can be used to monitor the progress of reactions in polymer forming systems for in-situ real time process control. Dielectric constant and dielectric loss have also been evaluated to substantiate the insulating nature of cured and uncured polyisoprene over a range of temperature and frequency using impedance analyzer in the frequency region 50 Hz to 5 MHz. Density functional Coloumb attenuating method CAM-B3LYP/6-311++G(d,p) and Hartree-Fock HF/6-311++G(d,p) calculations were performed to obtain the total static dipole moment (μ_{tot}), polarizability (α), first hyperpolarizability (β_0) and natural bonding orbital (NBO) analysis of this isoprene molecule. The NBO calculation was performed using the NBO program as implemented in GAUSSIAN 09W package in order to understand the magnitude of atomic charges in the molecule. The present investigation intends to look into accurate electrical properties of natural rubber composites for practical uses as electrically insulating material and also to help one better understand electrical conduction mechanisms which eventually leads the material to dielectric breakdown.**

***Keywords*—polyisoprene; microwave; dielectric constant; dielectric loss; hyperpolarizability; NBO**

I. Introduction

Natural rubber (NR) which is structurally cis 1,4-polyisoprene, is a polymer of high molecular weight and viscoelastic properties. Isoprene is a diene and 1, 4 addition leaves a double bond in each of the isoprene unit in the polymer. Because of this, natural rubber shows all the reactions of an unsaturated polymer [1]. Due to unique combination of properties, Natural rubber finds application in the manufacture of a variety of products.

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Its application in electrically energized line services as electrical energy utilities such as rubber insulating gloves, high voltage insulating matting, line hoses and blankets is targeted in the present work [2]. These are considered as important articles of personal protection for electrical workers. Crude rubber does not possess the desirable properties. Discovery of vulcanization of rubber using sulphur facilitated overcoming this problem. Improvement in desired properties can be achieved by addition of compounding materials such as sulphur, accelerators and colouring agents. Sulphur is the principal vulcanizing agent used with natural rubber. Yet another method of enhancing desired properties in rubber is the addition of certain fillers to the rubber before vulcanization. Presently, carbon black is the outstanding reinforcing filler for both natural and synthetic rubber materials. The interaction between carbon black and polymer that starts during the mixing process itself was studied by a number of researchers and the conduction mechanism was discovered in such mixtures. Such fillers were found to enhance viscoelastic response to deformation and increase electrical conductivity and dielectric constant. Recently, Z.M Elimat et al have investigated casted thin films of polyethylene oxide/carbon black composites as a function of applied frequency in the range from 100 Hz to 5 MHz and found that dielectric constant and dielectric loss decrease with frequency, and increase with temperature, and carbon content [3]. Prasun Banerjee et al have reported that the conductivity of EVA Rubber Composites at microwave frequencies increases with carbon black loading [4]. In the present work, a systematic approach suggested by Robert and Von-Hippel was successfully employed where in microwave test bench method is utilized to study the dielectric properties of uncured and cured Natural rubber. The dielectric behaviour as a function of frequency and temperature has also been studied using impedance spectroscopy. This can be used as a tool to understand the charge transport mechanism in the insulating polymer matrix and in a morphology that consists of insulating matrix and carbon black filler.

A literature survey reveals that no DFT/HF [5, 6] quantum computational calculation of Natural rubber has been reported so far. Hence from the natural bonding orbital analysis, the atomic charges of isoprene molecule was calculated by Natural population analysis (NPA) and Mulliken population analysis (MPA) using CAM-B3LYP/6-311++G(d,p) and HF/6-311++G(d,p) theoretical methods. The total static dipole moment (μ_{tot}), polarizability (α) and first hyperpolarizability (β_0) were also evaluated. The more available polarisation mechanisms a material possesses, the larger its dielectric constant will be. For example, materials with permanent dipoles have larger dielectric constants than similar, non-polar materials. Since the magnitude of the

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dipole affects the polarisation and hence the dielectric constant in a material, the NBA study becomes very critical here.

II. Experimental Methods

Natural rubber was procured from Industrial Rubber Products (IRP), Chennai, India in the raw state before undergoing the processes of mastication and vulcanization. The sample obtained was in the form of slabs with thickness about 12 mm. Carbon black was used as the reinforcing filler and sulphur was used as the principle-vulcanizing agent for curing the rubber material in the industrial lab. After proper mastication, the rubber material was passed through the rolls of the roll mill equipment. The combined cure package comprises the cure agent sulphur together with accelerators like Dibenzothiazyl disulfide (MBTS) and Tetra Methylthiuram Disulfide (TMTD), activators like zinc oxide and stearic acid and antidegradants according to the desired formulations as presented in Table 1. Antidegradants are used to prevent degradation by heat, oxygen and ozone. The composites were vulcanized at 160^oC under a pressure of about $45kg/cm^2$. The time of vulcanization was 30 minutes. The reinforced rubber material was obtained by mixing carbon black filler to the vulcanizates in the roll mill equipment. The carbon black filler content was 50 Phr (Phr-parts hundred in rubber). After the rubber, filler, sulphur and organic accelerators were mixed, the compound was placed in molds and subjected to heat and pressure. The rubber article was intended to adopt the shape of the mold. The samples were obtained in the form of slabs with thickness about 8 mm.

A microwave test bench system with an X-band in the frequency range 8.2-12.4 GHz and a K-band in frequency range 18.0-26.5 GHz were employed for determining dielectric constants of uncured and cured Natural rubber using Von-Hippel method [7]. The microwaves, generated by a microwave source propagate through a rectangular waveguide connected by couplers and attenuators. At the end of the rectangular waveguide there is a provision to attach a short circuit plate so as to cause a standing wave pattern in the waveguide system. In order to make measurements possible on this stationary wave pattern, a part of the waveguide has a slotted section.

TABLE I. FORMULATIONS USED IN THE PREPARATION OF RUBBER MATERIAL [a]

Ingredients	Natural Rubber
NR	100
ZnO[b]	5
Stearic acid	2
MBTS[c]	1.5
TMTD[d]	0.5
Sulphur	2.5

a. In phr: parts per hundred parts by weight of rubber
b. zinc oxide
c. Dibenzothiazyl disulphide
d. Tetra Methylthiuram Disulfide

As a probe with a GaAs tip was moved along this slotted section, voltage proportional to the intensity of the stationary wave corresponding to different positions along the guide were noted. The mean distance between the maxima or minima of this sinusoidal voltage plotted with respect to the position of the probe gives, half the guide wave length ($\lambda_g/2$). The position of the first minima from load end was also noted. The cured and uncured rubber samples of thickness‘t’ were intercepted in the path of the microwave and readings corresponding to the standing wave pattern were noted. To prevent air gap between dielectric sample and the inner walls of the rectangular waveguide, the samples were shaped to the dimensions of the waveguide, that is 2.2 x 1 cm for X-band and 1.1 x 0.4 cm for K-band. The shift in the position of first minima (Δ) from the load end was noted. Now, the quantity X is calculated from the expression,

$$X= \lambda_g/ t \tan [(2\pi(\Delta+ t)/\lambda_g] \quad (1)$$

Another quantity V that has been defined as the number of wavelengths of microwave radiation in distance t in the dielectric filled guide is mathematically expressed as $V= t/\lambda_g'$, then $X= \tan (2\pi V)/V$ where λ_g' is the wavelength of the electromagnetic waves in the dielectric medium. If the phase constant of the medium is $\beta_d=2\pi/\lambda_g'$ then the dielectric constant of the given specimen ε_r, is obtained from the equation,

$$\beta_d=2\pi/\lambda_0[\varepsilon_r\mu_r-(\lambda_0/2a)^2]^{1/2} \quad (2)$$

where λ_0 is the free space wavelength of the microwaves, a is the wider dimension of the waveguide and the relative permeability (μ_r) of the material at these high frequencies is taken as unity. The dielectric behavior of Natural rubber has also been studied in the raw and cured state using impedance spectroscopy method [8, 9]. Impedance spectroscopy (IS) is a general term that subsumes the small-signal measurement of the linear electrical response of a material of interest (including electrode effects) and the subsequent analysis of the response to yield useful information about the physicochemical properties of the system. Analysis is generally carried out in the frequency domain, although measurements are sometimes made in the time domain and then fourier transformed to the frequency domain. The impedance analyzer used for the current dielectric study is H10 KI 3532 LCR HITESTER in the frequency region 50 Hz to 5 MHz. The analyzer is connected through an interface and is controlled by software such as z plot and z view. Sample materials were shaped in rectangular form with 10 mm dimensional area and 3.5 mm thickness and placed between silver electrodes. The cell used for the measurements was a parallel plate circular condenser made of copper. The dielectric nature of the raw and cured rubber material was analyzed by studying the dependence of dielectric constant and dielectric loss with frequency and temperature. The specification of the instrument is given in Table 2.

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A. *Computational Method*

In order to understand the magnitude of atomic charges in Natural rubber (Isoprene molecule), Coloumb attenuating CAM-B3LYP/6-311++G(d,p) and Hartree-Fock HF/6-311++G(d,p) correlation functional calculations have been carried out on a dual core 1.8 GHz personal computer with the GAUSSIAN 09W program [10]. The geometry optimization was carried out using the initial geometry generated from standard geometrical parameters at B3LYP and HF methods adopting 6-311++G(d,p) basis sets to characterize all stationary points as minima. Using natural bonding orbital (NBO) program with symmetry considerations along with available related molecules, NBO calculation was performed as implemented in GAUSSIAN 09W package. The total static dipole moment (μ_{tot}), mean polarizability (α_0) and the first hyperpolarizability (β_0) were also calculated at the two levels of theory.

TABLE II. SPECIFICATION OF IMPEDANCE ANALYZER

Specification	Range
General frequency range	10 Hz to 32 MHz
Resolution	10 Hz to 1 Hz
Amplitudes	0 to 3 V and 0 to 60 mA
DC bias	40.95 V and 100 mA
Capacitance ranges	Capacitance 1 F to 10 F
Resistance	10 m to 100 M
Inductance	100 mH to 1000 H
Interface	Parallel compiler with IEEE 488
Maximum data rate	1000 bytes

III. Results and Discussion

A. *Dielectric Measurements at Microwave region*

Natural rubber has a dielectric constant of 4.6015 in the X-band frequency region (10 GHz) and 1.2633 in the K-band frequency region (26 GHz). It is evident from Table 3 that dielectric constant evaluated in K-band is lower than in X-band. Hence, dielectric constant decreases with increasing frequency and the result is in coincidence with literature [11]. Polymers with low dielectric constant, high resistivity and negligible power factor are considered to be good insulators as they are able to withstand a potential difference with a passage of even small electric current and low dissipation energy. The vulcanized and reinforced Natural rubber has greater dielectric constant than in crude state. Physically it means the greater the polarisation is developed by cured natural rubber in an applied field of given strength. The scientific community in this particular subject area has done a lot of quantitative and qualitative work. [12] reported that dielectric constant of the blend IIR/EPDM increases with increasing carbon black content. According to [13], the dielectric properties of rubber ferrite composites can be enhanced by the addition of an appropriate amount of carbon black. N.M. Renukappa et al have studied the physicomechanical and electrical properties of SBR with varying amount of carbon black loading [14]. A drastic improvement in the dielectric constant of the composites has been noticed because of an increase in carbon black loading in the SBR phase. According to Daniel P. Kowalik et al., carbon black filled silicon can be used as a compliant thermoelectric material as it was found to exhibit absolute thermoelectric power of +2 V/^{o}C [15].

The dielectric constant is an important parameter in deciding the insulation characteristics of the material as the rubber material with low dielectric constant and high dielectric strength is an excellent insulator. Here the dielectric constant in cured state increases and the insulation characteristic decreases. When cured NR is used as electrical energy utilities such as insulating gloves, matting and blankets, care should be taken before commercialization. The curing agents like sulphur and carbon fibre are generally added to increase the stiffness and elasticity of the rubber material. But the polarity in NR is also increased by curing it with suitable concentrations of such curing agents. Increasing the amount of curing agents to NR can eventually lead the material to dielectric breakdown. In the polar state, the material is able to store large amount of charges at small applied electrical field. That is in order to maximise the charge that a capacitor can hold, NR when used as a dielectric material in a capacitor needs to have a high permittivity also having a high breakdown voltage. Hence natural rubber cured with desirable proportion of carbon black and other curing agents can be efficiently used as a dielectric in a capacitor. With increase in concentration of curing agents, the voltage to be applied for breakdown to happen in NR also increases.

TABLE III. DIELECTRIC CONSTANTS OF CURED AND UNCURED NATURAL RUBBER

Sample Material	Dielectric Constant	
	X-band (10 GHz)	*K-band (26 G Hz)*
NR	4.6015	1.2633
Cured NR	5.7862	3.0635

B. *Impedance Spectroscopy*

When an alternating voltage is applied to the dielectric, the bound charges in it will move back and forth with certain amplitudes, different amplitude for each type of bound charge. When the applied electric field is of unit intensity, the sum of the product of amplitude and charge extended over all of the bound charges in a unit volume of the material determines the dielectric constant. The energy dissipated as heat by the motions of these bound charges in the applied electric field represents the dielectric loss per second; a quantity which is proportional to the ac conductivity after the dc conductivity has been subtracted from it. The imaginary part of complex dielectric constant is proportional to the dielectric loss per cycle. The dielectric

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behavior of Natural rubber has been studied in the raw and cured states using impedance spectroscopy method. Figures 1-2 show the plot of dielectric constant (ε_r)and dielectric loss (D) as a function of frequency in the raw and cured state. Dielectric constant and dielectric loss were evaluated as a function of frequency for four different temperatures [16]. Initially the temperature was fixed at 308 K and the dielectric behavior of NR was studied in the frequency region 10 Hz to 10^7 Hz. Both dielectric constant and dielectric loss were found to decrease with increasing frequency. $\mathcal{E}_r$ and D are inversely proportional to frequency which is a normal dielectric behavior that can be understood on the basis of the mechanism of polarization.

As the frequency increases, the dipole polarization effect will tend to zero and the dielectric constant will tend to be dependent only on the electronic polarization. This gives rise to diminishing values of ε_r and D. When the frequency is very low, the dipole movements are able to keep in phase with changes in the electric field and power losses are low. As the frequency is increased the point is reached when the dipole orientation cannot be completed in the time available and the dipole becomes out of phase. The dielectric behavior of Natural rubber was studied in the similar manner in uncured and cured states by maintaining the constant temperatures at 328 K, 348 K and 368 K respectively. Dielectric constant and dielectric loss exhibited similar variation with frequency for all the four temperatures.

Since temperature affects the electrical property of rubber and polymeric materials, it is necessary to study the thermal effect on dielectric behavior of Natural rubber [17]. The dielectric behavior of uncured and cured rubber was studied with respect to temperature for four fixed frequencies such as 1 KHz, 10 KHz, 100 KHz and 1 MHz as shown in Figures 3-4. Dielectric constant (ε_r) and dielectric loss (D) were found to increase in the uncured and cured states when temperature was increased from room temperature (300 K) to a high temperature such as 380 K. It has to be noted that both ε_r and D increase only slightly with temperature for a high frequency of 1 MHz.

C. ***NBO Analysis***

The atomic charges of isoprene molecule in Natural rubber was calculated theoretically by Natural population analysis (NPA) and Mulliken population analysis (MPA) using CAM-B3LYP/6-311++G(d,p) and HF/6-311++G(d,p) theoretical methods. From Table 4, it can be seen from NBO analysis [18, 19] that the magnitude of the carbon atomic charges changed from -0.6108 to -0.0467 in CAM method and -0.5273 to -0.0395 in HF method. The magnitude of hydrogen atomic charges varied from 0.18665 to 0.21236 in CAM method and 0.17151 to 0.18433 in HF method. H_{12} and H_{13} have the same magnitude of atomic charges calculated by two levels of the scheme.

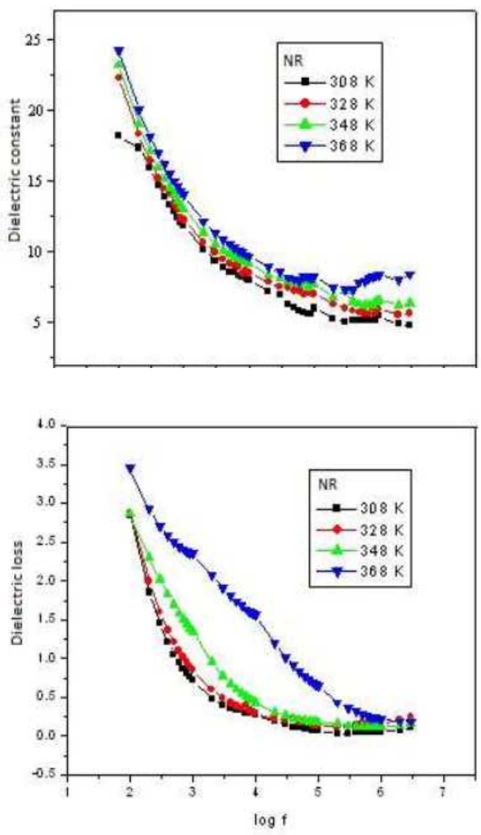

Figure 1 Frequency dependence of ε_r and D for uncured Natural rubber

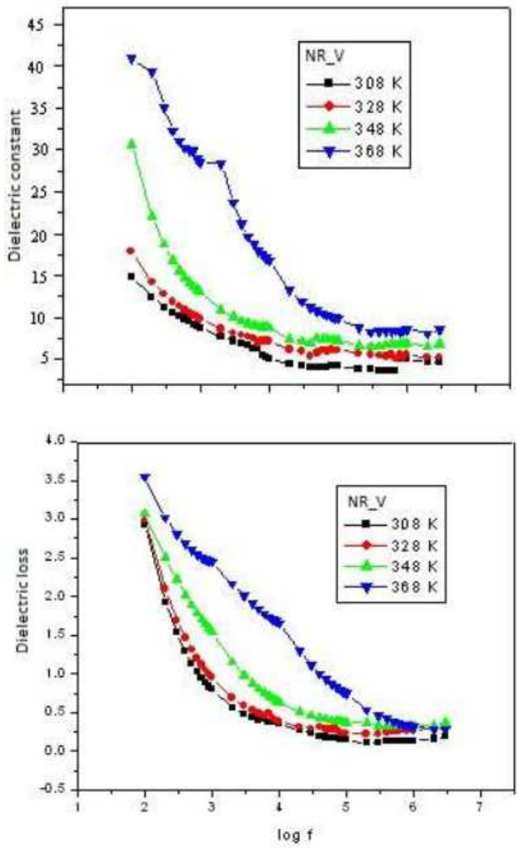

Figure 2 Frequency dependence of ε_r and D for cured Natural rubber

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Hydrogen atoms in the molecule lost electrons, hence the carbon (C_5) is more electronegative as three hydrogen atoms (H_{11}, H_{12}, H_{13}) are connected to it. C_3 is comparatively less electronegative than C_4 and C_1 as C_3 is bonded to one hydrogen atom whereas C_4 and C_1 are bonded to two hydrogen atoms. Further C_2 bonded with C_1, C_3 and C_5 is found to be more positive than the other carbon atoms. In general, the greater the difference in electronegativity between two atoms, the bond formed between them will be more polar with the atom having the higher electronegativity being at the negative end of the dipole. So for a material to act as a good insulator, it must have a weak polar covalent bond. The natural bonding orbital theoretical analysis is another confirmation of insulating nature of Natural rubber. In isoprene molecule, apart from C_5 which is connected to three hydrogen atoms, all the other C-C and C-H bonds relatively have a very weak polar covalent bond due to the small difference in electronegativities leading to an insulator.

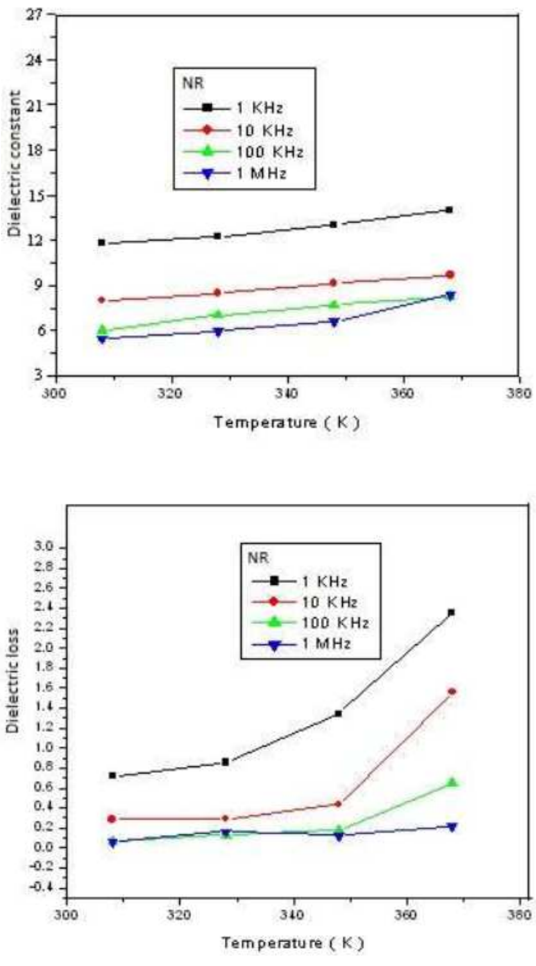

Figure 3 Temperature dependence of ε_r and D for uncured Natural rubber

D. ***Hyperpolarizability Calculation***

Non Linear Optics (NLO) finds lot of future applications in areas such as telecommunications, signal processing, optical interconnections and various emerging technologies. Few of the non-linear phenomena observed in materials are second harmonic wave generation, optical mixing and optical phase conjugation [20, 21]. In discussing NLO properties, the polarization of the molecule by an external radiation field is often approximated as the creation of an induced dipole moment by an external electrical field, and this change can be calculated as

$$pe_1 0 + \alpha E + \frac{1}{2}\beta : EE + ...$$

where $pe_1 0$ → dipole moment in the absence of an electric field; α → second rank tensor called the polarizability tensor; β → first in an infinite series of dipole hyperpolarizabilities.

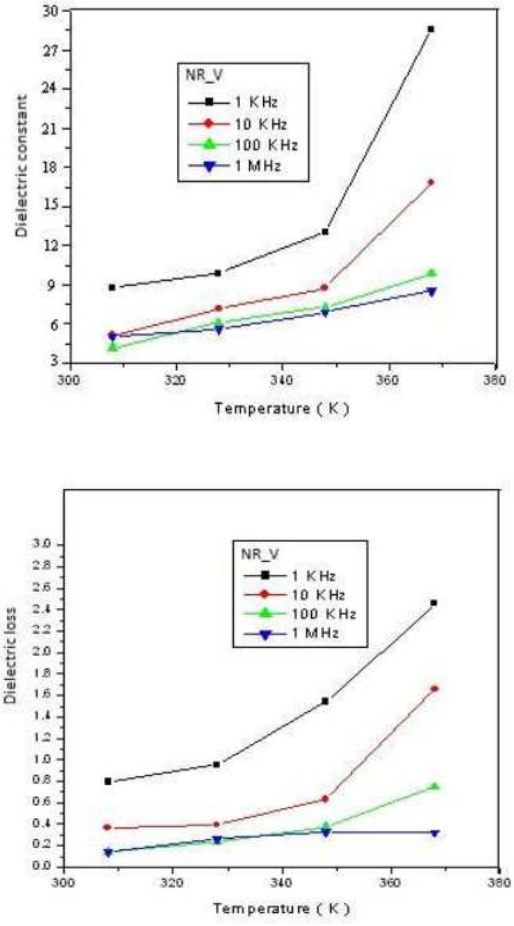

Figure 4 Temperature dependence of ε_r and D for cured Natural rubber

The complete equations for calculating the magnitude of total static dipole moment (μ_{tot}), the mean polarizability (α_0), the first hyperpolarizability (β_0) using the x, y, z components from Gaussian 09W outputs are as follows:

$$\mu_{tot} = (\mu_x{}^2 + \mu_y{}^2 + \mu_z{}^2)^{1/2}$$

$$\alpha_0 = \frac{1}{3}(\alpha_{xx} + \alpha_{yy} + \alpha_{zz})$$

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$$\beta_0 = (\beta_x^2 + \beta_y^2 + \beta_z^2)^{1/2}$$

where $\beta_x = \beta_{xxx} + \beta_{xyy} + \beta_{xzz}$;

$\beta_y = \beta_{yyy} + \beta_{xxy} + \beta_{yzz}$;

$\beta_z = \beta_{zzz} + \beta_{xxz} + \beta_{yyz}$.

The dipole moment which reflects the molecular charge distribution was calculated assuming the neutrality of the molecules. The components of dipole moment, polarizability and hyperpolarizability values of NR are given in Table 5. The magnitude of total static dipole moment (μ_{tot}) was found to be 0.2740 10^{-18} C.cm and 0.2685 10^{-18} C.cm from the CAM and HF methods. The mean polarizability (α_0) was also determined as 64.035 Fcm^2 and 63.549 Fcm^2 respectively from the CAM and HF methods.

The reason for performing polarizability and hyperpolarizability calculation here is because dielectric constant is found to be related closely to the polarizabilities of the assemblages of charged particles which the dielectric contains. A discrepancy may arise between the theoretical and the experimental polarizability due to the fact that the calculations considered a single molecule whereas the experimental values cannot ignore the intermolecular interactions.

TABLE IV. NATURAL ATOMIC CHARGES OF NATURAL RUBBER

Atom with Numbering	NPA		MPA	
	CAM	*HF*	*CAM*	*HF*
C_1	-0.3614	-0.3381	-0.6314	-0.8148
C_2	-0.0467	-0.0395	0.84205	1.11059
C_3	-0.202	-0.1824	-0.1417	-0.024
C_4	-0.373	-0.3447	-0.4088	-0.5615
C_5	-0.6108	-0.5273	-0.7905	-0.952
H_6	0.19051	0.17553	0.12614	0.15087
H_7	0.18938	0.1733	0.14213	0.1496
H_8	0.19598	0.17974	0.1486	0.19197
H_9	0.19562	0.18004	0.15087	0.16471
H_{10}	0.18665	0.17151	0.13428	0.14848
H_{11}	0.21099	0.18433	0.14168	0.14377
H_{12}	0.21236	0.18374	0.14328	0.14619
H_{13}	0.21236	0.18374	0.14327	0.14618

The computed first hyperpolarizability (β_0) of the present molecule is 4.556 x 10^{-31} cm^5/esu using CAM-B3LYP/6-311++G(d,p) method and 4.127 x 10^{-31} cm^5/esu using HF/6-311++G(d,p) method which is quite comparable with urea (β_0 of urea is 3.7289 x 10^{-31} cm^5/esu). Here threshold value of prototype urea is used for comparison in isoprene (NR) molecule as urea serves as an important molecular system in the study of Non Linear Optics (22). Thus we can definitely expect isoprene molecular system to exhibit non-linear effects when irradiated with high power laser.

TABLE V. CAM-B3LYP AND HF CALCULATED PARAMETERS FOR NR MOLECULE

Parameters	CAM-B3LYP	HF
μ_x	-0.0380	-0.0192
μ_y	0.2713	0.2678
μ_z	0.0	0.0
μ_{tot}	0.2740	0.2685
α_{xx}	89.754	90.275
α_{xy}	10.829	13.581
α_{yy}	60.197	58.342
α_{xz}	0.00089	0.00100
α_{yz}	0.00102	0.00096
α_{zz}	42.154	42.031
βxxx	38.456	33.172
βxxy	-3.140	1.605
βxyy	-15.596	-10.987
βyyy	-17.136	-6.702
βxxz	0.1669	-0.1005
βxyz	0.0857	-0.1163
βyyz	0.0384	-0.1060
βxzz	24.108	23.640
βyzz	-3.683	-8.367
βzzz	0.00730	-0.0208

IV. Conclusion

A characterization of Natural rubber based on its dielectric and microwave properties has been done utilizing important experimental techniques. The dielectric constant of uncured and cured rubber material was determined using X and K-band microwave test benches by employing Roberts and Von Hippel's method. Cured NR is found to have a high permittivity than uncured rubber. So in order to maximise the charge that a capacitor can hold, cured NR can be preferred as a dielectric material than uncured one. Moreover, cured NR also has a high dielectric breakdown voltage than the uncured one. Thus the insulation characteristic of cured NR is superior to the uncured one in this aspect. Using impedance spectroscopy, dielectric constant and dielectric loss of cured and uncured Natural rubber was evaluated as a function of frequency for four different temperatures. Also, the dielectric behavior of uncured and cured rubber was studied with respect to temperature for four fixed frequencies. The geometry of Natural rubber was optimized with both CAM-B3LYP and HF methods using 6-311++G(d,p) basis sets. The atomic charges of isoprene molecule in Natural rubber were calculated theoretically by NBO which confirms the insulating nature of Natural rubber. In isoprene molecule, other than C_5, all the other carbon atoms are less electronegative and are bonded by weak polar covalent bonds. The total static dipole moment, mean polarizability and the first hyperpolarizability values of NR were also

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theoretically calculated and from the results we can definitely expect isoprene molecular system to exhibit non-linear effects when irradiated with high power laser. The present investigation intends for accurate electrical properties of natural rubber composites and brings out the result that cured NR is better than uncured NR when used as a dielectric material in a condenser because of its high dielectric breakdown voltage. But cured NR is disadvantageous when used in practical applications such as rubber insulating gloves, high voltage insulating matting, line hoses and blankets as there is an increase in possibility of accident or shock due to dielectric breakdown in the rubber material.

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