CHAPTER THREE

Measurement of Dry Rubber Content in Natural Rubber Latex with a Capacitive Transducer

3.1: Introduction

In October 1745, Ewald Georg von Kleist of Pomerania in Germany invented the first recorded capacitor, a glass jar with water inside as one plate held, on the hand which acted as the other plate. A wire in the mouth of the bottle received charges from an electric machine, and released it as a spark. A typical capacitor consists of a pair of conductors separated by a thin layer of dielectric substance - that is, by an electrical insulator that contains essentially no mobile, current-carrying, charged species. The simplest capacitor consists of two sheets of metal foil separated by a thin film of dielectric such as air, oil, plastic, mica, paper, ceramic, or metal oxide. A useful property of a capacitor is its ability to store an electrical charge for a period of time and then to release the stored charge when needed. The capacity is defined as the charge that the capacitor can store per unit of potential difference between the
plates. The capacity of a condenser is increased by interposing a dielectric material between the plates.

In tensammetry the capacitance of the electrical double layer of an electrode is measured. The capacitance value is strongly influenced by the presence of absorbing species. Although tensammetry is possible at all kinds of electrode surfaces, mercury is the most widely used electrode material. Detectors with mercury electrodes (mercury-coated, dropping mercury, and hanging or static modes) are mainly used for the determination of substances in solutions. Other electrode materials based on the capacitance principle have also been developed. A tantalum capacitance sensor is used for the detection of proteins by immunochemical binding reactions. Berggren and Johansson immobilized monoclonal antibodies on gold substrates. The antibody layers and their interactions with antigens in solution were investigated with capacitance measurements and with cyclic voltammetry. The capacitance change versus the logarithm of antigen concentration is linear over three decades. A gas sensitive semiconductor based on a palladium MOS field effect capacitor for the determination of ammonia in air and aqueous solutions has also been described. The main problem in preparing a stationary working electrode for tensammetric measurements is the formation of a homogeneous and well-defined surface. Double layers are not a special feature of the electrode-electrolyte interfaces; they are a general consequence of the junction of two phases at a boundary. Owing to its high dielectric
constant (78.5 at 25 °C) water is the primary species, which can be measured by changes of the capacitance\textsuperscript{11}. Capacitive transducers are used for root extent measurement\textsuperscript{12} of plants and estimating root mass in maize\textsuperscript{13}.

Time-resolved patch clamp capacitance measurements are now widely used to study the changes in plasma membrane area associated with exocytosis and endocytosis\textsuperscript{49}. For high resolution recordings, a sinusoidal voltage is added to the holding potential, and the resulting current is analyzed by a two-phase lock-in amplifier. When the phase of the lock-in amplifier is properly adjusted, the two outputs directly provide the changes of membrane conductance in one channel and the changes of membrane capacitance in the other channel\textsuperscript{50-53}. This method is widely used to detect very small capacitance changes and the opening of individual fusion pores in single secretory granules\textsuperscript{54-57}.

Capacitive transducers are extensively used for measuring various physical parameters such as displacement\textsuperscript{15}, mass\textsuperscript{21}, torque\textsuperscript{23}, micrometer displacements\textsuperscript{24}, force\textsuperscript{25}, strain\textsuperscript{26}, speed\textsuperscript{31,41}, acceleration\textsuperscript{32}, noise\textsuperscript{45}, distance\textsuperscript{47}, surface force\textsuperscript{48}, pressure\textsuperscript{36}, dielectric parameters\textsuperscript{30} and contact stiffness\textsuperscript{40}. Another major application of capacitive transducers is in Ultrasonics\textsuperscript{17, 20, 46, 59-65, 67-80} and its imaging\textsuperscript{27, 29}. Capacitive transducers are also used for the detection of chemicals\textsuperscript{98}, endotoxin\textsuperscript{33} and even heavy metals\textsuperscript{34}. 
In textile manufacturing, capacitive transducers are used for the measurement of yarn evenness parameters and liquid absorption in non-woven fabric. Capacitive transducers are used in multi-sensor systems, acoustic emission, and diffraction loss measurements, two-dimensional space-time analysis, and in miniature microphones. In agriculture, capacitive transducers are widely employed for the measurement of water intake of plants, soil water content, soil salinity effects, soil water dynamics, soil analysis, herbage yield, and for irrigation control.

In this work, our objective is to develop a method to determine the DRC of rubber latex, by measuring the variation of the capacitance of a specially designed capacitive transducer with the latex acting as the dielectric. The method is found to be accurate, fast, inexpensive, user-friendly and adaptable to varying environmental conditions. The correlation of DRC values with capacitance variation and the related electrical properties, such as impedance, loss factor etc. are established and discussed. The shortcomings and limitations of the method are also discussed.

3.2: Principle of the Method

An elementary parallel plate capacitor consists of two conducting plates, electrically isolated from one another by an insulating medium. The capacitance (C) of this elementary capacitor is proportional to (i) the cross-
sectional area $A$ of the plates, (ii) the permittivity (or dielectric constant $K$) of the insulating medium and (iii) the reciprocal of the separation, $t$, between the plates. The relation is given by

$$C = \frac{KA}{t} \quad \ldots \ldots \ 3.1$$

If the area of the plates and the separation between them are kept constant, the capacitance of the capacitor is directly proportional to the dielectric constant or permittivity of the medium. If there is a direct relation between the DRC and dielectric constant of latex, then the same relationship should hold good for the capacitance and DRC of rubber latex, if used as the dielectric. A capacitor, when connected to a sinusoidal voltage source, responds to it sinusoidally with definite impedance following the relation,

$$V = V_o e^{i\omega t} \quad \ldots \ldots \ 3.2$$

where the angular frequency $\omega = 2\pi f$, stores, $f$ being the test frequency. When vacuum is it’s dielectric, the induced a charge,

$$Q = C_0 V \quad \ldots \ldots \ 3.3$$

and draws a charging current given by

$$I_c = \frac{dQ}{dt} = j\omega C_0 V, \quad \ldots \ldots \ 3.4$$
Fig 3.1: Current - voltage relation in an ideal capacitor

which leads the voltage by a temporal phase angle of 90° (Fig. 3.1). $C_0$ is the vacuum (or geometrical) capacitance of the capacitor. Here, $V$ is the instantaneous voltage, $V_0$ is the peak value of voltage, $\omega$ is the signal angular frequency and $t$ is the time. When filled with a dielectric medium of absolute permittivity $\varepsilon'$, the capacitance value gets modified as

$$C = C_0 \frac{\varepsilon'}{\varepsilon_0} = C_0 K'$$  \hspace{1cm} \ldots 3.5,$$

where $C_0$ is the capacitance with air or vacuum as the dielectric, $\varepsilon_0$ is the permittivity of free space and $K'$ is the dielectric constant of the medium. A dielectric material with higher relative permittivity enhances the storage capacity of a capacitor by neutralizing the charges at the electrode surfaces, which otherwise would have contributed to the applied external field. The impedance of a capacitor is not a pure reactance, but is modified by the series resistance of the leads and plates, losses in the dielectric, parallel resistance
of the plates and leakage effects. One way to handle this complex situation is
to combine all these effects into an equivalent series resistance ($R_s$),
measured directly with an impedance bridge or indirectly with instruments
such as Q meters. The overall impedance of the system is given by

$$ Z = R + jX $$  

...3.6

where $X$ is the reactance of the capacitor. The impedance, $Z$, is the inverse
of admittance, $Y$,

$$ Y = 1/Z $$  

......3.7

or $Y = G+jB$  

......3.8

where $G = 1/R$ and $B = \omega C_p$

Here $G$ is the conductance, $C_p$ is the parallel plate capacitance and $B$ is the
susceptibility of the medium.

$$ \omega = 2\pi f, \ f \text{ being the test frequency.} $$

The dissipation factor $D$ can be expressed as

$$ D = 1/Q $$  

......3.9

where $Q$ is the quality factor. The equivalent resistance ($R$) of the capacitor is
given by

$$ R = |Z| \cos \theta $$  

......3.10
where \( |Z| = \sqrt{R^2 + X^2} \), and \( \theta = \tan^{-1}(X/R) \), \( \theta \) being the phase lag due to capacitive reactance.

3.3: Capacitance Transducer Design and Measurement Method

3.3.1: General description

Experimental set up presented in this chapter consists of a specially designed capacitive transducer and an LCR (Inductance, Capacitance, Resistance) meter. The designed capacitive transducer consists of six concentric metallic cylinders with increasing diameters, insulated from each other and firmly fixed. Each cylinder acts as the plate of a capacitor with the alternate cylinders connected externally in parallel to increase the effective area of the electrodes.

The equivalent diagram of the capacitor combination and a schematic diagram of the assembled capacitive transducer are shown in Figures 3.2 and 3.3 respectively. The block diagram of the experimental set up used for measurements is shown in Figure 3.4. The effective capacitance of the capacitive transducer is \( C \) and is given by

\[
C = C_1 + C_2 + C_3 + C_4
\]  

\[\ldots... 3.11\]
Fig 3.2: Assembled capacitive transducer

Figure 3.2 shows arrangement of the individual capacitors in the capacitance transducer. The capacitance transducer is fabricated using six hollow tubes (Aluminum) of increasing diameters. The tubes are finely machined to obtain a uniform wall thickness throughout its length. The length of each tube is seven centimeters and all the tubes are arranged according to the diameters as shown in the figure. The cylinders are placed 1 mm apart and insulated using a Teflon spacer. Holes of size 0.5 mm are drilled into the walls of the tubes vertically to take out the connection leads. High quality wires are positioned into these holes and punched using special tools. The connections
are checked thoroughly to ensure that there are no failures. Parallel connections are derived from the transducer as shown in the figure. Assembled tubes are then firmly fixed on a Teflon sheet to hold the tubes in position.

![Equivalent Capacitive transducer](image)

**Fig 3.3: Equivalent Capacitive transducer**

![Block diagram of the measurement set up](image)

**Fig 3.4: Block diagram of the measurement set up**
This capacitive transducer is immersed in a beaker containing a constant volume of latex (400 ml), whose capacitance is to be measured. The terminals A and B of the assembled capacitive transducer (Figure 3.1) are connected to an LCR meter (Agilent Technologies Model 4263 B) for measuring the capacitance with natural rubber latex as the dielectric medium. The capacitance, as well as other parameters, is measured at a frequency of 100Hz under standard laboratory conditions. All measurements have been carried out within four hours of collecting the samples from the collection Centre. Other parameters such as resistance (\( R \)), dissipation factor (\( D \)), impedance (\( Z \)) and susceptibility (\( G \)) are also measured with the same LCR meter. All these parameters for different samples with different DRC values are measured under the same physical and environmental conditions for direct comparison.

3.3.2: Sample collection and preservation for measurement

The latex samples for all the experiments described in this work, are collected from the Factory Management Division (FMD) of Rubber Research Institute of India, Kottayam, Kerala, India. This division collects latex from small holders as well as from the experimental farm of Rubber Research Institute of India to make value added latex products. For the work presented in this thesis, latex samples (Clone: RRII 105, Year of planting: 1989-93, D\(_3\) tapping system) with wide variations in DRC are collected. The latex samples
are collected after filtration and an anticoagulant is added to each sample to preserve it. The amount of anticoagulant (Ammonia) added to each sample is kept constant to ensure that the effect of the anticoagulant is the same in all measurements. After the preparation of latex samples by adding the anticoagulant, 10-15 ml of each sample is collected in separate containers to determine their DRC values following the standard laboratory drying method and another 400 ml of the latex for capacitance measurement, as outlined above. The general procedure of the laboratory drying method is to coagulate a known weight of representative sample of the latex with dilute acetic acid, sheet the coagulum and dry it at about 75°C in an oven. The DRC of the latex is therefore the percentage by weight of the dry sheet over the weight of latex under test.

3.4: Results and Discussion

The experimental data collected from a series of measurements on different sets of samples are presented in Table 3.1. The parameters tabulated and their units are listed at the bottom of the table. The estimated uncertainties of each of the parameters are also indicated in this table. Figures 3.7 to 3.10 show the variations of series and parallel capacitance values of the capacitive transducer as well as other parameters with DRC of the latex. The results obtained are analyzed using standard statistical tools.

The parallel and series capacitance values exhibit high negative correlations (-0.84 and -0.86 respectively), whereas the Dissipation factor (D)
and Resistance \((R)\) exhibit a high positive correlations (0.76 and 0.79 respectively). Impedance \((Z)\) shows a medium positive correlation (0.59). The uncertainties indicated in the figures take into account all the uncertainties involved in the measurement of DRC and capacitance values. One can notice that both series and parallel capacitances of the capacitor are inversely proportional to the DRC of the latex. The proportionality constants obtained are 0.123nF/%DRC and 0.117nF/%DRC respectively.

The purpose of this work is to see whether a relation between the DRC of rubber latex with its dielectric properties could be established. With a proper design of the present capacitive transducer, we see that the capacitance, in general, is inversely proportional to the DRC and it can be measured with sufficient sensitivity and accuracy. The other electrical/dielectric properties naturally follow the dielectric constant or permittivity of the medium. For the design of a practical measuring instrument one need not measure all the parameters presented in Table 3.1. We find that the series capacitance of the measuring capacitor is most sensitive to variations in DRC compared to other measured parameters.

While designing a practical DRC meter following this scheme, one need to provide provision to measure just the series capacitance accurately. We think that such an instrument can be made at a comparatively low cost and the measurements can be done in the field with a battery-operated instrument. Since the DRC measurements need to be made by non-technical personnel working at latex collection centers, it is important that the instrument
is made user friendly and the measurements are done in a short period of time. The design and fabrication of such an instrument, we think, is feasible.

Table 3.1: Variation of capacitance and other physical parameters with DRC of latex.

<table>
<thead>
<tr>
<th>No</th>
<th>DRC (%)</th>
<th>Cp (nF)</th>
<th>Cs (nF)</th>
<th>D</th>
<th>R (Ω)</th>
<th>Z (Ω)</th>
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<tbody>
<tr>
<td>1</td>
<td>25.2</td>
<td>928.4</td>
<td>928.8</td>
<td>0.0234</td>
<td>40.75</td>
<td>1.7167</td>
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<td>2</td>
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<td>928.7</td>
<td>0.0241</td>
<td>42.33</td>
<td>1.7145</td>
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<td>3</td>
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<td>928.0</td>
<td>928.5</td>
<td>0.0246</td>
<td>43.54</td>
<td>1.7147</td>
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<tr>
<td>4</td>
<td>30.3</td>
<td>927.9</td>
<td>928.3</td>
<td>0.0244</td>
<td>42.91</td>
<td>1.7152</td>
</tr>
<tr>
<td>5</td>
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<td>0.0246</td>
<td>43.50</td>
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<td>6</td>
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<td>45.79</td>
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<td>926.7</td>
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<td>47.35</td>
<td>1.7222</td>
</tr>
</tbody>
</table>

Cp- Parallel capacitance, Cs- Series capacitance, D-Dissipation factor, R- Resistance, Y- Admittance, G-Susceptance, nF- nano farad, Ω- ohm, ms- milli siemens.
Figure 3.5: Variation of series capacitance with % DRC of latex. Measurement uncertainties are indicated in the figure.
Figure 3.6: Variation of parallel capacitance with % DRC of latex.

Measurement uncertainties are indicated in the figure.
Figure 3.7: Variation of dissipation factor with % DRC of latex. Measurement uncertainties are indicated in the figure.
Figure 3.8: Variation of resistance with % DRC of latex. Measurement uncertainties are indicated in the figure.
3.5: Limitations of the Method

Though we could establish a good correlation between DRC values and the corresponding capacitance values of natural rubber latex, this technique suffers from several limitations. Natural rubber latex, as already explained, contains lutoids and inorganic ions which contribute to ionic activity in this colloidal dispersion. As time passes breaking of more lutoids takes place thereby releasing more ion pairs into the medium. These ion pairs certainly increases the ionic activity in latex contributing to a change in capacitance values of the latex. Owing to this activity and the possible variation in capacitance measurements this method suffers from nonreproducibility of the results. Another drawback of this method is the effect of the anticoagulant. The latex, immediately after collection at the collection centre is treated with anticoagulant for preservation. The addition of anticoagulant also alters the polar nature of the medium and this in turn affects the capacitance measurement. Moreover, this technique requires high sample volume (400 ml) and so a tree-wise measurement of DRC of latex for scientific purposes is rather difficult.

3.6: Conclusions

We have been able to establish a good correlation between DRC and dielectric/electrical properties of natural rubber latex samples. It is found that DRC is sensitive to the capacitance of the specially designed capacitor, which could be used to design a practical instrument, based on the above principle.
Even though we have been able to establish the relationships between DRC and dielectric/electrical properties of rubber latex, we have not attempted to bring out the microscopic phenomena responsible for the observed effects. Further, we have not investigated the influence of non-rubber constituents and adulterants in these measurements. Interpretation of the results in terms of the molecular polarizability of the medium would be very informative to understand the electrochemical processes relevant to this complex medium. We estimate that a practical measurement system following this method will have measurement uncertainties up to ± 2% due to various factors. However, we think that the measurement uncertainties can be reduced considerably by optimizing the transducer design and measurement procedures. In spite of these positive aspects, the method suffers from the limitations discussed earlier. Due to these limitations it is not advisable to make efforts to develop instruments to measure DRC of latex based on capacitive transducers.
References


