Hevea brasiliensis, a forest tree, is the only major commercial source of natural rubber (NR) in the world. Polyisoprene extracted from Hevea brasiliensis is known as Natural Rubber. The elastic properties of NR have eventually led to a multi-billion dollar industry world wide and a source of livelihood for several million people across the globe. In India commercial cultivation of Hevea had started in the year 1902. In terms of productivity, growth in area, production and the extent of price realization, the Indian rubber plantation industry is ahead of all other major natural rubber producing countries in the world. Today, India is the fourth highest producer of rubber in the world having a total of 554000 hectares under rubber cultivation with a total production of 605045 tons per year.

Hevea brasiliensis, once planted in the field, will become a mature tree over a span of 6-7 years and is ready for harvesting. During fresh tapping by a process of wounding, white milky fluid coming out of the Hevea tree before preservation or concentration is known as field latex. Latex is a special form of cytoplasm containing a suspension of rubber and non-rubber particles in an aqueous serum. Besides rubber and water, fresh latex contains Lutoids, carbohydrates, proteins, lipids and
inorganic salts. Concentrated latex as such is used for manufacturing several latex based products. Latex collected from the field is coagulated, processed and dried under standard conditions to produce raw natural rubber, which is used for the manufacture of various rubber based goods, including automobile tyres.

This thesis embodies the work done by me on the development of instrumentation techniques to measure the dry rubber content in natural rubber latex.

Importance of Dry Rubber Content (DRC)

Dry Rubber Content or DRC is an important parameter for natural rubber latex (NRL), which is to be measured quickly for various purposes for the rubber industry. DRC is defined as the mass in grams of solid rubber present in 100 g of latex. The DRC of latex varies depending upon season, tapping system, weather, soil conditions, clone of the tree and environmental conditions. The DRC of *Hevea* latex is a very familiar term to all in the rubber industry. It is probably one of the few properties of latex first recognized and widely used for trade and processing, ever since the commencement of commercial exploitation of *Hevea* trees. The DRC is an essential parameter for ensuring fair prices for latex during commercial exchange. It has been the basis for incentive payments to tappers who bring in more than the daily agreed poundage of latex rubber. It is an important parameter that determines the yield of useful
rubber from any latex, and is required for various decision making processes in the automation of industrial processes in rubber products manufacturing.

Techniques to measure DRC

The most accurate method to determine the DRC of latex is the Standard Laboratory Method (SLM). The general procedure of this method is to coagulate a known weight of the representative latex sample with dilute acetic acid, sheet the coagulum and dry it at about 75°C in an oven for nearly 12 hours. The DRC of the latex is given by the percentage by weight of the dry sheet over the weight of the latex taken. Numerous other methods have also been cited in literature for the measurement of the DRC of latex. The hydrometer is probably the most rapid one, even though its accuracy is limited by a wide range of conditions encountered in practice. For latex, the average error of the measurement with hydrometer is about 4% of the value estimated by the standard laboratory method. The 'Spot Method' is popular in many laboratories, which uses an accurate weighing balance to weigh the coagulum from a 0.3 g sample dried on a steam bath oven and expressed as a percentage of the original mass. The measurement time is about 30 minutes, with an error of about 1%. The main disadvantages of these drying and weighing methods are that they are time consuming and labor intensive.
Other methods that have been reported in literature for the determination of DRC include Viscosity method, Latex film dialysis, Titration method, Microwave attenuation, Low resolution pulsed NMR technique, Spin eco- technique etc. The shortcomings of these methods are that they either require a specialist for measurements, they are time consuming, expensive, have limited accuracy or could not be adapted for field measurements.

Due to these limitations the above methods have not gained much popularity among stakeholders in the rubber industry. The NR industry is still in need of a viable, inexpensive and accurate method to determine the DRC of natural rubber latex. In view of this scenario we decided to undertake research work on development of DRC measurement techniques, with the following definite objectives.

**Objectives of the work**

1) Study various characteristic properties of natural rubber latex following different optical, dielectric and thermal instrumentation techniques and develop measurement techniques that are fast, accurate and economical for the determination of the DRC of natural rubber latex.

2) Use the Scientific information collected from the research work to design and develop a viable and accurate DRC measurement
instrument which is user friendly, fast, inexpensive and usable in the field.

**Outline of the work**

Initially, we studied various characteristic properties of natural rubber latex, such as optical absorption in the MIR region and thermal properties by thermo-gravimetric analysis (TGA). In the second phase, we designed and fabricated a special capacitive transducer to measure the variations of the dielectric properties of latex samples with their DRC values. Then we conducted a series of measurements to study the variation of optical absorption of latex samples in the near Infrared (NIR) region with the corresponding DRC values. Finally, we conducted Differential Scanning Calorimetric studies on different latex samples to understand the relation between change in enthalpy per unit mass over a defined temperature range of the samples and their corresponding DRC values. In all these methods direct relationship between DRC values and the relevant measurands have been established experimentally. We have developed these five measurement techniques for the estimation of DRC of natural rubber latex and the information collected from these studies have been used to design and develop practical DRC measurement systems wherever possible.
Organization of the thesis

The thesis is presented in six chapters as outlined below.

Chapter One: Review of the Existing Techniques for the Determination of Dry Rubber Content in Natural Rubber latex

A brief history of Hevea brasiliensis and Indian rubber plantation sector is included in the first part of this chapter. Subsequently, we have included a brief description of the properties of latex, importance of DRC in NR industry and the technical details of the different known techniques used for the determination of the DRC of latex. Finally, a brief summary of the shortcomings of the existing DRC measurement methods and an outline of the entire research work are presented.

Chapter Two: Estimation of Dry Rubber Content in Natural Rubber Latex by FTIR Spectroscopy and TGA Techniques

This chapter embodies the work done and results obtained with analytical techniques such as Infrared optical absorption and thermo gravimetric analysis. A general description of Infrared absorption measurements using a Fourier Transform Infrared Spectrometer and its various applications are included in this chapter, followed by a brief outline of the experimental set up for absorption measurements, method of sample preparation, results obtained and a discussion of the results. We have been able to establish a linear relation between IR absorption
around 835 cm\(^{-1}\) with the DRC values of NR latex samples. Work done on thermal analysis following Thermo Gravimetric Analysis (TGA) is also presented in this chapter. The details of the experimental setups, methods of sample handling, experimental details, results and discussion etc for these measurements are included in the latter part of this chapter.

**Chapter Three: Measurement of Dry Rubber Content of Natural Rubber Latex with a Capacitive Transducer**

In this chapter we have presented and discussed various applications of capacitive transducers, principle of the capacitance method, constructional details of a capacitive transducer designed by us and experimental set up used for measurements. The results obtained from a series of measurements are compiled and presented in the later part of the chapter, followed by a discussion of the results. In this method we have established a correlation between dielectric properties, such as dielectric constant, dielectric loss, a.c. conductivity etc of latex and with the corresponding DRC values. The limitations and shortcomings of the capacitance method are discussed in detail.

**Chapter Four: Near IR Spectral response of Natural Rubber Latex in the Reflectance Mode**

This chapter begins with a general description of the near IR absorption spectroscopy. Various applications of NIR spectroscopy in
agriculture, medical and dental sciences are subsequently discussed. The theory of optical absorption, experimental method developed and the results obtained from the measurements are included in the second part of this chapter. We have developed a spectral reflectance technique for the measurement of optical absorption in the NIR region. The details of the technique and the results obtained are presented in this chapter. A direct proportionality between NIR reflectance intensity at 1460 nm and DRC has been established. Finally the results obtained are compiled and discussed.

Chapter Five: Estimation of Dry Rubber Content in Natural Rubber Latex by Differential Scanning Calorimetry

A general description of differential scanning calorimetry (DSC) and its various applications, with a special reference to thermal analysis of polymers, are included in the first part of this chapter. We have shown that the mass normalized change in enthalpy in a defined temperature range is proportional to the DRC of latex samples. The principle of the method, experimental method adapted and the results obtained are presented and discussed in the second part of the chapter. Finally, the results obtained from the DSC measurements are compiled and we have established a correlation between mass normalized areas and the corresponding DRC values of latex samples. Subsequently the results
are discussed and general conclusions drawn from the investigations which are presented chapter five.


**Chapter Six: Summary and Conclusions**

In this chapter a summary of entire research work carried out is included. The overall conclusions drawn from the work and scope for doing further research in DRC measurement techniques are also presented in this chapter.

**Papers published**


(2) Reji Kumar, R and Philip J., Determination of Dry Rubber Content in Natural Rubber Latex from NIR Reflectance Measurement, *Proc. of the Kerala Science Congress* (2009) **10-16**:774-776

**Popular article published**


**Presentations in Seminars/ Symposia**

(1) Reji Kumar, R., Najamul Hussein, S and Philip, J., Measurement of Dry Rubber Content of Natural Rubber Latex with a Capacitive
Transducer, presented at the National symposium on Instrumentation held at CUSAT during 30th November - 2nd December, 2006.

(2) Reji Kumar, R., Najamul Hussein, S and Philip, J., Measurement of Dry Rubber Content of Natural Rubber Latex with a Capacitive Transducer, presented in the 366th Scientific Seminar of the Rubber Research Institute of India, held on 22nd December, 2005.


Papers communicated


(3) Reji Kumar, R and Philip, J., Estimation of Dry Rubber Content in Natural Rubber Latex by FTIR Spectroscopy and TGA techniques, J. Analytical Sciences (2009)