EPOXIDATION OF NATURAL RUBBER LATEX

AND

ITS TECHNOLOGICAL EVALUATION

BY

AMAL SHANTHA MUNASINGHE DISSANAYAKE

Thesis submitted in the partial fulfilment of the requirements for the degree of Master of Science of the faculty of natural science, University of Sri Jayawardanapura, Nugegoda, Sri Lanka.

1999

EPOXIDATION OF NATURAL RUBBER LATEX

AND

ITS TECHNOLOGICAL EVALUATION

by

A. S. M. DISSANAYAKE

ABSTRACT

The high ammonia centrifuged natural rubber latex was epoxidised with performic acid generated in-situ. The latex was stabilised against coagulation by adding 10% non ionic surfactant and bringing down the dry rubber content up to 30% . The reaction temperature was maintained at 50 ± 1 c^0. It was targeted to prepare 50 mole % epoxidised NR after completion of 2.5 hrs. of reaction time. The epoxidation proceed rapidly and effectively reaching to pure ENR of 30 mole % epoxide content and was unable to reached the targeted value. The system was coagulated after obtaining 30 mole % epoxidised rubber. Two laboratory trials were done namely, trial-1 and trial-2 giving the same experimental conditions.

Samples of each trial were subjected to quantitative analysis by FTIR and ^{1}H-NMR spectroscopy. The amount of natural rubber epoxidised was calculated as % peak ratio obtained by FTIR and degree of epoxidation obtained by ^{1}H-NMR. The data obtained from two spectroscopic methods were not exactly tally with each other but shows gradual increase in the epoxide content and finally come to 30-35 mole % epoxidation level, before it coagulated. The two spectroscopic methods were made use to identify the ring open products formed during epoxidation. It has found there were no ring open products formed and pure ENR with various levels of epoxidation can be obtained.

The data obtained from trial-1 using ^{1}H-NMR were subjected to co-relation and regression analysis and found there is a lenear regression of the % epoxidation on reaction time at 5% level of significance as described by the regression equation $\mathbf{Y_i = 4.5 + 10.8\ X_i}$ (X_i = reaction time and Y_i = % epoxidation). Co-relation coefficient "r " is 0.9897 where it indicate better co-relation between two parameters.

Two large batches (weight of the latex = 1kg.) which were targeted at 15 and 30 mole % epoxidation were done satisfactory. Raw rubbers obtained were thoroughly washed with running water and fully dried before characterisation. The Mooney viscosity and the density of NR, ENR and NR/ENR blends were studied. It has found the density was increased with the increasing level of epoxidation in all cases. The values obtained for the mooney viscosity of ENR is higher than that of NR and found in increasing order with the increasing level of epoxidation.

Four types of rubbers were prepared with ENR-15, ENR-30, NR/ENR-15(50:50) blend and NR/ENR-30(50:50) blend for the technological evaluation . NR also used as a control.

Technological properties of each were studied in detail. The vulcanisates with low level of accelerator (0.8 phr of TMTD) and high level of sulphur (2.2 phr) indicate small scorch time hence giving low processing safety. The ageing resistance which was obtained from tensile properties was poor. The blends which used high level of accelerator (0.8 phr of MBTS and 0.2 phr of DPG) and low level of sulphur (1.2 phr) indicate comparable large scorch time hence better processing safety.

Tear and the compression set of two blends found to be higher than others where abrasion is poor. Ageing performance of blended rubber was unpredictable and aged values found to be improved .

The solubility related parameter was studied by calculating the degree of swelling after immersing the samples in three different kinds of hydrocarbon oils. NR vulcanisate shows the highest degree of swelling in hydrocarbon oils. When the epoxide content is high or in other words the polar fraction is high it shows resistance to non polar solvents. The resistance towards swelling is also high with the high level of epoxidation.

CHAPTER ONE - INTRODUCTION

CHAPTER TWO - EXPERIMENTAL

CHAPTER THREE - RESULTS AND CALCULATIONS

CHAPTER FOUR - DISCUSSION