

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS OF CHLORINATED
ACRYLATE ESTER AND
INCORPORATION EFFECT ON
PREVULCANISED NATURAL
RUBBER LATEX PROPERTIES**

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ABSTRACT

Chlorinated acrylate ester (CAE) has been prepared by esterification reaction process. Ethylene glycol, acrylic acid and chloroacetic acid was used as components in CAE. Esterifications were done in two different methods; one pot reaction (method A) and stage reaction (method B). Cleaned CAE was characterized by Fourier Transform Infrared (FTIR) and Nuclear Magnetic Resonance ($^1\text{H-NMR}$) analysis to confirm the formation of ester and the present of chlorine and acrylate functionality. From FTIR analysis, appearance of peak at wavenumber 3200 cm^{-1} attributed to the C-OH bonds which presented in chloroacetic acid, ethylene glycol and acrylic acid. The disappeared of the peak in CAE showed the bond were subjected to breakage and form a new bond. Peak at wavenumber 1073 cm^{-1} , 1036 cm^{-1} and 956 cm^{-1} show the present of C-O-C bonds in CAE. The absorption peak at 698 cm^{-1} and 781 cm^{-1} revealed the present of C-Cl bonds. From $^1\text{H-NMR}$ analysis, the peaks at δ 4.4 ppm, δ 4.2 ppm, δ 4.07 ppm, δ 6.15- δ 6.08 ppm, δ 6.36 ppm and δ 5.79 ppm proved a successful of the esterification reaction and the formation of CAE. The effect of CAE in pre-vulcanized natural latex system was investigated. Formulation with 3 %, 5%, 10% and 15% (wt/wt) of CAE (method B) into prevulcanised natural latex were prepared. The stability of formulations was achieved with the addition of ammonia to remain the pH of latex at 10. Stable formulation was formed into the rubber sheet and cured at room temperature for 24 hours and followed by 80°C in oven for another an hour. FTIR analysis of the cured sheet show the decrease of intensity at 1662 cm^{-1} which represent the breakage of C=C bond. The absence of peak at 1728 cm^{-1} of C=O bond present in CAE and the appearance of peak of OH stretching at $\sim 3394\text{ cm}^{-1}$ showing that the ester was introduced onto the rubber system. The substitution of Cl in the system assigned by the peak at $\sim 756\text{ cm}^{-1}$ absorption bands of C-Cl stretching. Thermal Gravimetric analysis (TGA) depicted the initial degradation at 240°C . Glass transition temperature (Tg) was recorded by Differential Scanning Calorimetry (DSC) at 60°C for each formulation. TGA and DSC analysis of the samples discovered that the modification with low percentage of CAE not affect the thermal stability of the rubber. The tensile test of the cured sample shows that the tensile strength was decreased as the CAE amount increased. The adhesion property of modified sample was examined by single-lap-shear test. The bond strength between rubber and zinc was increased as the amount CAE was increased until 10% by weight.

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CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

Natural rubber is an important industrial material, particularly in the tire industry or in the production of various products including household, medical, engineering and commercial goods. It is abundant renewable resources which have interesting mechanical properties and can be chemically modified. The chemical modification of natural rubber is one of alternative practice to increase its value and to develop versatile applications. Whereas, the natural rubber double bonds in the chains are probable to varieties of chemical reactions. An alternative way to transform natural rubber into useful products is by starting with a low molecular weight natural rubber which is widely known as liquid or natural rubber latex (Ananchaenwong, 2011).

Chlorination of natural rubber is one of a leading method practiced in industry. It was beneficially improved the rubber products' quality by reducing surface friction coefficient, reduces surface tackiness and creates a silky surface finish (Siriwat, Sombat, & Tawechai, 2009). In fact, reaction of chlorine with the natural rubber product's surface molecule could form a structure that restrict access of oxygen or ozone to the rubber and increase its barrier to absorption of oils and greases during storage and use. It prevented the breakdown in the rubber surface structure; discoloured and brittle. Chlorination also gives benefit of deactivate the potentially harmful proteins from natural rubber products which in turn remove or restrict the potentially dermatitis chemicals from the rubber that cause contact dermatitis in people (Palosuo, Antoniadou, Gottrup, & Phillips, 2011)

Utilization of chlorine functional into rubber system has been broadly explored for various applications nowadays. Numerous systematic studies have been published previously (Ho & Khew, 1999a; Siriwat et al., 2009; Tyczkowski, Krawczyk, & Woźniak, 2003; Van Amerongen & Koningsberger, 1950) and had revealed some light on the chlorination of natural rubber in term of chemistry and