

**EFFECT OF COMBINATION ULTRASONIC AND
BALL MILLING TECHNIQUES OF FILLERS
DISPERSION ON MECHANICAL AND
PHYSICAL PROPERTIES OF NATURAL
RUBBER LATEX FILMS**

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by

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LIST OF ABBREVIATIONS

ASTM	American Standard for Testing Materials
CaCO ₃	Calcium Carbonate
CaNO ₃	Calcium nitrate
CB	Carbon Black
EB	Elongation at break
HA	High Ammonia
ISO	International Organization for Standardization
KOH	Potassium hydroxide
M	Modulus
MEA	Monoethanolamine
MPa	Mega Pascal
MST	Mechanical Stability Time
N/mm	Newton per millimetre
NH ₃	Ammonia
NR	Natural Rubber
pH	Hydrogen concentration
POM	Polarised Optical Microscopy
Si	Silica
TiO ₂	Titanium Dioxide
TMTD	Tetra Methyl Thiuram Disulphate
TSC	Total Solid Content
ZDEC	Zinc diethyl carbamate

LIST OF SYMBOLS

°C	Degree Celsius
cm ³	Centric cubic
m/m	Mass per mass
min	minute
ml	milliliter
mm	millimeter
wt/wt	weight over weight
g	gram
hrs	hours
phr	Part per hundred rubber
rpm	Revolution per minute
s	second
µm	Micrometre
m _o	The initial mass
m _i	The final mass of the dried latex test sample
nm	nanometre
µm	Micrometer

**KESAN KOMBINASI KAEDAH ULTRASONIK DENGAN PENGILING
BEBOLA BAGI MENYEDIAKAN SERAKAN PENGISI TERHADAP SIFAT
MEKANIKAL DAN FIZIKAL FILEM LATEKS GETAH ASLI**

ABSTRAK

Penambahan pengisi komersial seperti kalsium karbonate, titanium dioksida, kaolin, karbon hitam dan silika dalam sebatian lateks getah asli telah mengurangkan sifat mekanikal filem lateks getah asli ini disebabkan kesan ketidakstabilan dalam sebatian lateks getah asli yang dikawal oleh kualiti serakan, saiz zarah dan juga pH serakan itu sendiri. Kaedah pengiling bebola adalah kaedah konvensional bagi penyediaan penyebaran dan kaedah ultrasonik telah berjaya digunakan dalam penyediaan pengisi nano seperti tiub karbon nano (CNT). Dalam kajian ini, parameter pengiling bebola (durasi masa dan kelajuan), kesan parameter ultrasonik (durasi masa, kelajuan, dan kepekatan), dan kaedah kombinasi telah dijalankan. Gabungan kaedah ultrasonik dan pengiling bebola telah menunjukkan pengurangan saiz partikel serakan pengisi yang dihasilkan. Pengujian saiz partikel, mikroskopi elektron penghantaran (TEM) dan ujian mikroskopi elektron (SEM) telah dijalankan untuk mengkaji sifat serakan pengisi. Ujian tegangan dan cabikan juga dijalankan untuk mengkaji sifat-sifat mekanikal filem lateks getah asli. Penyebaran CaCO_3 dan TiO_2 digunakan untuk mengkaji kesan pembebanan pengisi (0, 5, 10, 15 dan 20 bsg) dalam filem lateks getah asli. Berdasarkan keputusan sifat mekanikal, pembebanan pengisi yang optimum diperolehi pada 10 bsg. Serakan CaCO_3 digunakan dalam sebatian pembersihan lateks getah asli untuk meningkatkan keberkesanan untuk menarik keluar kotoran semasa proses pembersihan. Keseluruhannya, kajian ini memberikan kaedah penyediaan serakan pengisi yang sesuai bagi menghasilkan saiz partikel pengisi yang kecil supaya serasi dengan saiz partikel lateks getah asli.

**EFFECT OF COMBINATION ULTRASONIC AND BALL MILLING
TECHNIQUES OF FILLERS DISPERSION PREPARATION ON
MECHANICAL AND PHYSICAL PROPERTIES OF NATURAL RUBBER
LATEX FILMS**

ABSTRACT

The addition of commercial fillers like calcium carbonate, titanium dioxide, kaolin, carbon black and silica in Natural Rubber (NR) latex compounds has reduced the mechanical properties of NR latex films due to the destabilization effect in the NR latex compounds which govern by the dispersion quality, particle size and also the pH of the dispersion itself. The ball milling methods were a conventional method of preparation of dispersions and ultrasonic method has successfully used in preparation of nano fillers such as carbon nano tube (CNT). In this study, ball milling parameters (duration, speed), effect of ultrasonic parameters (duration, speed, and concentration), and combination methods were carried out. The combination of ultrasonic and ball milling methods has shown the reduction in particle size of the resulted fillers dispersion. The particle size analyzer, transmission electron microscopy (TEM) and scanning electron microscopy (SEM) analysis were carried out to investigate the properties of fillers dispersions. The tensile and tear test were also carried out to investigate the mechanical properties of the NR latex films. The CaCO_3 and TiO_2 dispersions were used to investigate the effect of filler loading (0, 5, 10, 15 and 20 phr) in NR latex films. Based on the mechanical properties results, the optimum filler loading obtained was at 10 phr. The dispersion of CaCO_3 was used in NR latex cleaning compound to improve the effectiveness to pull out the dirt during cleaning process. Overall, this study has specified suitable fillers dispersion preparation methods to obtain small particle size of fillers which compatible with the size of NR latex particles.

CHAPTER ONE

INTRODUCTION

1.0 Introduction

Natural rubber (NR) latex was tapped naturally from the *Hevea brasiliensis* tree, which was a species of rubber tree indigenous to Brazil. Physically, it was a cloudy, white liquid, similar in appearance to cow's milk with a density of 0.980 g/cm³ or thereabouts, and containing, one-part rubber particles with non-rubber particles suspended in water. NR latex tapped from rubber trees needs to undergo a process known as vulcanization to convert it to useful product. In NR latex, vulcanization or crosslinking occurs between the rubbers of these particles converting, from temperature sensitive material to technologically useful elastic material (Hashim, 2010).

Commercial fillers and pigments were added to latex compounds for various reasons, the principal of which are to stiffen the product obtained from the latex, to cheapen it, and color it. The presence of significant levels of filler particles in a latex compound was also likely to affect the flow behavior, partly because the overall volume fraction of the disperse phase in the compound was altered (usually increased) and partly because colloid equilibria may be disturbed, thereby allowing structures to develop between the various dispersed particles (Blackley, 1997a). Fillers such as carbon black, kaolin, calcium carbonate (CaCO₃), silica and titanium dioxide (TiO₂) were commonly used in the rubber compound. Carbon black and silica are known as their reinforcing role, whereas calcium carbonates are known to reduce the cost. Titanium dioxide and kaolin were normally added to the NR latex products for

whitening purposes (Ochigbo & Luyt 2012). In general, conventional CaCO₃ and TiO₂ are used as non-reinforcing fillers in NR latex compounds to improve the cost-performance ratio of the dipped products. It is known that the addition of these non-reinforcing fillers reduces the mechanical properties, especially the strength of NR latex films (Cai et. al., 2003). Besides that, the mechanical properties reduce due to the destabilization effect in the NR latex compound (Mat Suki et al., 2015).

In addition, higher loading levels of these inorganic particulate fillers impart stiffness to the NR latex films and as a result the flexibility which one of the most important properties of NR latex gloves (Amarasiri et. al., 2013). According to the free volume concept, the higher stiffness will restrict chain mobility

The methods of dispersions preparation play a role in the reduction of the particle size of the prepared dispersion. In the NR latex compounding, the fillers need to be dispersed before adding in latex compounding to ensure the homogeneous distribution of the dispersions in the compound. The ball milling method was widely used for preparing filler dispersion for latex compounding ingredients.

The ball milling was a mechanical method to reduce crystalline region and modify the particle size of fillers with a number of potential advantages such as reduction of the particle size due to shear with high possibilities of the fillers reduction in the size. However, the process operates at room temperatures, thus increasing energy the consumption. The high degree of agitation helping in breaking of any agglomerated material and no gaseous emissions or liquid effluents has produced (Franta, 2012). The basic processing parameter includes ball milling time and ball rotation speed.

On the other hand, ultrasonic method has been used by some research and industries to prepare dispersion together with ball milling method. Ultrasonic method was widely used in preparation of nano fillers dispersion such as carbon nanotubes (CNTs) (Zhong et al., 2012). The cavitation during the ultrasonic process generates high shear forces that break particle agglomerates into single dispersed particles. Duration for ultrasonic plays important role in dispersing powders into liquids. Zhong et al. (2014), proved that an excellent time to prepare dispersion when the ultrasonic power and frequency were fixed.

1.2 Problem Statement

Fillers have been widely used in many applications including in rubber or latex industries for the past decades. These filler function as reinforcing agents, which increase certain mechanical properties, such as tensile strength, tear strength, and abrasion resistance and also as cheapener to reduce the cost (Blackley, 1997a and Roslim et al., 2010).

However, owing to the high surface energy, particles of fillers tend to agglomerate during the dispersion preparation. The particle agglomerations are detrimental to the properties in NR latex film to the microstructure heterogeneity (Vaßen et al., 1999). At present, a number of methods were used for the dispersion of particle which includes ball mill and ultrasonic as the most common methods (Hussain et al., 2006, Chen et al., 2007 and Bitmann et al., 2011).

Extensive studies have been carried out on commercial filler (reinforcing and non-reinforcing) particles for the preparation of the high performance product in the last two decades, as they possess many excellent characteristics depending on the particle size (Zhang et al., 2012 and Arumugam et al., 2010). But there are few reports on the

method for the dispersion of commercial filler particle, which restrain their potential benefits and applications. For this reason, it was important to investigate the dispersion method to decrease particle size of commercial filler dispersions. Currently, the single dispersion method was commonly used for preparing filler dispersion, especially ball milling and ultrasonic methods as mentioned above. In the ball milling method, when the fillers are milled the particles will have a large specific surface area with high energy where their hydrophilic tendency to absorb water and agglomerates. Granularity and evenness in size also were the main problem in the milling process (Ding et al., 2008 and Shahabuddin et al., 2011).

The problem occurred in ultrasonic method was the dispersion ability of the ultrasonic was lower as the size of particles decreases. Few agglomerates can be broken up when they attained a critical size range with the increased of ultrasonic time up to a certain value. Excessive increased of ultrasonic time cannot result in the more homogeneous dispersion of the powder. Furthermore, the thermo-effect caused by ultrasonic might result in the increased of the temperature, which increased the steam pressure of the bubble formation (Zhong et al., 2014). The transducers send and receive ultrasonic pulses through the steam. The mass flow is then calculated from temperature and pressure inputs and built- in steam tables.

The aim of this research was to find an effective dispersion preparation method for preparing fillers (CaCO_3 and TiO_2) dispersion. A comparative investigation was carried out to study the dispersion effects based on a different dispersion method which include ball milling, ultrasonic, ultrasonic /ball milling and ball milling / ultrasonic methods on CaCO_3 and TiO_2 dispersions. Their dispersion preparation on the particle size was discussed. Investigation was further made by applying the optimum processing dispersion preparation method of CaCO_3 and TiO_2 on reinforcing filler

carbon black and silica on mechanical properties of NR latex film. Then, the best dispersion preparation method was used to investigate the effect of filler loading of CaCO_3 and TiO_2 on the properties of NR latex films. The smallest CaCO_3 dispersion prepared by ultrasonic/ ball milling method was used NR latex cleaning compound application. The CaCO_3 was added in NR latex and waste latex cleaning compound in order to increase the effectiveness to pull out the dirt during cleaning application.

1.3 Research Objectives

The effect of combination ultrasonic and ball milling methods of commercial filler dispersion on mechanical properties of NR latex films was carried out with the specific objectives include:

1. To determine the optimum processing time for ball milling, ultrasonic methods and combine of both methods of the filler (CaCO_3 , TiO_2 , kaolin, carbon black and silica).
2. To investigate the effect of CaCO_3 , TiO_2 , kaolin, carbon black and silica prepared by combine method filler dispersion preparation method (ultrasonic/ball milling) with optimum processing time of fillers on mechanical properties of NR latex films
3. To examine the effect of filler loading of CaCO_3 and TiO_2 fillers on the mechanical properties such as tensile strength and tear strength of NR latex films
4. To compare the untreated CaCO_3 and treated CaCO_3 by ultrasonic and ball milling inside NR latex cleaning and investigated their tensile strength and cleaning effectiveness of the NR latex films.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

This chapter discusses the literature review about Natural rubber (NR) latex, methods to produce the latex compound, the preparation of filler dispersion, the properties of commercial filler, different dispersion preparation methods, filler loading and function of cleaning agent on NR latex film.

2.2 Natural Rubber (NR) Latex

The source of natural rubber is the latex tapped from rubber trees of the species *hevea brasiliensis*. In the modern polymer field, the term 'latex' denotes stable colloidal dispersion of a polymeric substance in an aqueous medium. NR latex, being the most widely known plant latex, was a stable colloidal dispersion of cis-1,4-polyisoprene with high molecular mass in an aqueous medium comprises of two phases, a dispersion medium and dispersed phase. The dispersion medium was also referred to as the 'serum' of the latex, and denoted as the aqueous fluid which exudes from the latex when colloidal destabilization and phase separation has occurred. The other phase, known as the dispersed phase of discontinuous phase with a composition of spherical particles of the high molecular weight polymer with particles sizes ranging from 100 nm to 1000 nm (Bateman,1963).

For NR latex to be industrial useful, the colloidal stability of the NR latex should remain unchanged and stable for longer period of time. The NR latex must also be capable of being colloidal destabilized by appropriate conditions to produce latex products. The stability of the field NR latex should be maintained after the NR latex

was collected from the rubber tree through process concentrated commonly done by centrifugation to increase the dry rubber content to 60% and then preserved with ammonia (Premamoy, 2002).

Immediately after the tapping, the latex is usually mixed with ammonia solution to preserve it against bacterial attack and also to prevent solidification (coagulation). High ammonia (HA) latex is concentrated latex that contains about 0.6 – 0.8% m/m of ammonia. The addition of ammonia increased the pH of the latex to approximately 11 to 12. Thus, apart from being effective as a bactericide, ammoniation also increased the colloidal stability of NR latex both for short-term and long-term storage. Meanwhile, 0.2 % m/m of ammonia on the whole latex was sufficient for short-term preservation and it is called low ammonia (LA) latex (Blackley, 1997a).

2.3 Natural Rubber (NR) Latex Compounding

Compounding was a process of incorporating chemical reagent/curative agents to achieve desired properties. In NR latex compounding, the first step of compounding was the preparation of the solution (water soluble ingredients), dispersion (solid ingredients), and emulsions (water insoluble ingredients). There are three important rules for the preparation of solutions, dispersion and emulsion which are;

I. The particle size must be compatible with size of latex dispersion. Difference in particle size during compounding could increase the possibility of formation of coagulations. This is due to the kinetic energy exerted by smaller particles which may surpass the energy potential barrier easily and formation of instantaneous coagulation (Wiese and Healy, 1970).

II. Colloidal stability of dispersion and emulsion should be compatible Stability of the colloidal particles depends on the potential energy barrier which should be bigger than the average kinetic energy of the particles. DLVO theory is the aggregation of aqueous dispersions quantitatively and describes the force between charged surfaces interacting through a liquid medium. The well-known DLVO theory was established by Derjaguin, Landau, Verwey, and Overbeek in the 1940s (Derjaguin and Landau 1941, Verwey and Overbeek 1948). It is theory a dispersion stabilizing theory in which zero potential was used to explain how two particles approach one and another wherein the ionic atmospheres begin to overlap and repulsion force was developed. As predicted by DVLO's theory, small particles will exhibit more rapid coagulation than larger particles (Jan, 2007).

III. pH of the solution must be like the pH of the latex. Incorporation of highly acidic or alkali compound into NR latex system which leads to an instantaneous coagulation. This was due to the modification of colloidal particle interaction with the new medium that eventually altered their potential energy barrier, thus leading into colloidal destabilization (Joseph and Alexander, 2010).

Raw NR latex must be compounded with several additives to enhance its properties and being able to be utilized commercially. The additives used can be divided into three main categories which are surface active agent, liquid phase modifier and elastomer phase modifier (Blackley, 1997a);

2.3.1 Surface Active Agent

Surface active agent or surfactant was used to change the water insoluble materials into water compatible system to avoid the undesired change in colloidal stability of latex system. There are three classifications of Surface Active Agent.