# MECHANICAL PROPERTIES AND CHARACTERIZATION OF NATURAL RUBBER CHITOSAN COMPOSITES

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#### Abstract

The present research work focuses on the preparation and characterization of natural rubber composite by incorporation of chitosan as filler into the rubber matrix. In the preparation of natural rubber chitosan composites, the chitosan powder loading were 0 %, 2.5 %, 5 %, 7.5 % and 10 %. The rubber chitosan composite compounding was prepared by using two roll mills and then molded by compression molding method. The mechanical properties of natural rubber chitosan composites such as specific gravity, tensile modulus (MPa), tensile strength (MPa), elongation at break (%) and tear strength (kN/m) were determined by standard rubber testing methods (ASTM). The result suggested that increasing the chitosan powder loading, specific gravity and tensile modulus of the composites were increased and tensile strength, elongation at break and tear strength of the composites were decreased. The prepared natural rubber chitosan composites were characterized by SEM technique.

Keywords: natural rubber, chitosan, mechanical properties, natural rubber chitosan composites

# Introduction

Natural rubber is a naturally occurring elastomeric polymer of isoprene (2-methyl-1, 3-butadiene). Natural rubber is included in at least 40,000 products including food packing materials, medical devices, tires and non-automotive mechanical products. Natural rubber is essential in all these different demanding applications due to its outstanding properties, such as resilience, elasticity, abrasion and impact resistance, efficient heat dispersion and malleable at cold temperatures, which cannot be matched by synthetic rubber (Cornish 2001).

Particulate materials (fillers) have been used for decades for the reinforcement or dilution of natural rubber and other elastomers. The addition of fillers in natural rubber is necessary in order to achieve the mechanical properties required. The efficiency of the fillers depands on parameters that are related to the fillers characteristics, including the particle size, structure and functionality and also parameters related to the rubber and processing conditions. The reinforcing effect of fillers is achieved due to physical and chemical interactions between the rubber and the fillers that allow these two components to produce one material with the inherent properties of both (Ahmadi and Shojaei, 2013).

Natural components are currently being manufacture with carbon black and silica, the former being the most widely used filler in industry. Nevertheless, carbon black is derived from petroleum and so is a non-renewable material. In the last decades some research on alternative filler material has explore bio-based sources (Valodkar and Thakor, 2012).

Chitin is a structural polysaccharide and is the second abundant natural polymer after cellulose. Chitin is found in shells of arthropods (crabs, lobsters, as well as mollusks (eg., squid pens). Chitosan is a linear copolymer d-glucosamine obtained by partial chitin deacetylation normally found in the form of granules, sheets, or powders (Ravi Khumar, 2000). There are currently a variety of applications for chitosan in the biomedical, food, and chemical industries

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owing to its interesting properties, such as biodegradability, biocompatibility, low toxicity, and biological activity (Domard, 2011).

This study focuses on incorporating chitosan to be successful employed as filler is ensuring good interaction with the rubber at a reasonable loading level. Then, the effects of chitosan loading on the mechanical properties, morphology of chitosan-filled natural rubber are reported.

# **Materials and Methods**

All chemicals used in this research were procured from British Drug House (BDH), England. The chemicals were as used as received unless state otherwise. All specific chemicals used were cited detail in each experimental section. The apparatus consist of conventional lab wares, glass wares and modern equipment.

# Sampling

Natural rubber sheet was procured from local market, Mawlamyine Township, Mon State. Commercial chitosan purchased from Ever Green Co. Ltd. (Yangon) was used in the present work.

#### **Preparation of Chitosan Sample**

The purchased chitosan sample was ground into powder by grinding machine and dried in oven (70  $^{\circ}$ C, 3 h) prior to use expel moisture.

# **Preparation of Natural Rubber Chitosan Composites**

The vulcanization of natural rubber chitosan composites were carried out for efficient vulcanization system. Composition of the weights of the ingredients used was shown in Table 1.

Natural rubber was first rolled about 7 min by a roller to break out the fibrous bond of rubber polymer chain. This process is called mastication. Stearic acid, zinc oxide and titanium oxide (TiO<sub>2</sub>) were added. Simultaneously and continuously rolled about 5 min. Then, mercapto benzadiazole (MBT), zinc diethyl dithiocarbamate (ZDEC) and butylated hydroxytoluene (BHT) and jelly were added in order to make acceleration to prevent oxidized and vulcanite harder. And then, the chitosan powders were added as filler in the compounding. It was then rolled continuously for about 10 min with sulphur to obtain a two millimeter thickness sheet. The total mixing time was approximately 20 min.

Composite A is natural rubber composite by mixing natural rubber (100 g) and other natural rubber chitosan composites were prepared by loading chitosan filler such as 2.5, 5, 7.5 and 10 g.

Ingredients	Composition of ingredients in various natural rubber chitosan composites (g)							
	A <sub>0</sub>	A <sub>2.5</sub>	$A_5$	A <sub>7.5</sub>	A <sub>10</sub>			
Natural Rubber	100	100	100	100	100			
Zinc oxide (ZnO)	0.75	0.75	0.75	0.75	0.75			
Stearic acid	1.0	1.0	1.0	1.0	1.0			
TiO <sub>2</sub>	1.0	1.0	1.0	1.0	1.0			
ZDEC	1.0	1.0	1.0	1.0	1.0			
BHT	0.5	0.5	0.5	0.5	0.5			
MBT	2.0	2.0	2.0	2.0	2.0			
Jelly	1.0	1.0	1.0	1.0	1.0			
Sulphur (S)	1.2	1.2	1.2	1.2	1.2			
Chitosan	0	2.5	5	7.5	10			

Table 1 Composition of Ingredients in the Prepared Natural Rubber Chitosan Composites

 $A_0$ -  $A_{10}$  = natural rubber composites with various loading of chitosan 0 to 10 (g)

 $TiO_2$  = titanium oxide

ZDEC = zinc diethyldithiocarbamate

#### **Determination of Mechanical Properties of Natural Rubber Chitosan Composites**

Mechanical properties of natural rubber chitosan composites are those physical properties that related to strength, toughness and durability. The properties were determined at Rubber Research and Development Center in Ministry of Agriculture, Livestock and Irrigation Department of Agriculture (Yangon).

#### **Determination of specific gravity**

The specific gravity of the composites was measured by Wallace direct reading specific gravity balance. The test piece was suspended on a needle form at one end of the beam which was zeroed by means of quickly adjustable sliding weights. The test piece was then immersed in water contained in a glass beaker locked on a frication-clamped platform. This platform can be raised and lowered easily and remained in position without on additional clamping. When the test piece was immersed, the specific gravity was calculated. The results are shown in Figure 2 and Table 2.

#### Determination of tensile strength, elongation at break and tensile modulus(M<sub>300</sub>, M<sub>400</sub>, M<sub>500</sub>)

The tensile test was done according to H.5000 F tensile testing machine. The test was performed to determine the capability of a material to resist the deformation during stretch. The important data obtained from tensile test were tensile modulus at 300 % ( $M_{300}$ ), modulus at 400 % ( $M_{400}$ ), modulus at 500 % ( $M_{500}$ ), tensile strength and elongation at break. The prepared composites were cut off according to JISK 7127. The both ends of the test pieces were firmly clamped in the jaw of tensile strength testing machine. One jaw was fixed and other was movable. The movable jaw moved at the rate of 10 mm min<sup>-1</sup>. The resultant data were shown

at the recorder. This procedure was repeated three times for each composite. The results are presented in Figures 3, 4, 5 and Table 2.

#### **Determination of tear strength**

The tear strength was measured by tear strength testing machine. The specimen to be tested was cut out by the die from the above sheets. Specimen was cut with a single nick (0.05 mm) at the entire of the inner concave edge by a special cutting device using a razor blade. The clamping of the specimen in the jaw of test machine aligned with travel direction of the grip at the rate of 100 mm min<sup>-1</sup>. The recorder of the machine showed the highest force to tear from a specimen nicked. The procedure was repeated three times for each result. The results are presented in Figure 6 and Table 2.

# Characterization of the Prepared Natural Rubber Chitosan Composites by SEM Modern Technique

The prepared composites were investigated by SEM for surface morphology was performed at West Yangon University, Yangon.

# **Results and Discussion**

This research work focuses on the investigation of the effect of chitosan loading on rubber compounding. There were 5 types of natural rubber chitosan composites prepared as shown in Figure 1. As the result, the changing of the mechanical properties of the prepared composites was observed with increasing filler loading on rubber matrix (Table 2). And then the characterization of the prepared composites by using SEM modern technique was also performed.

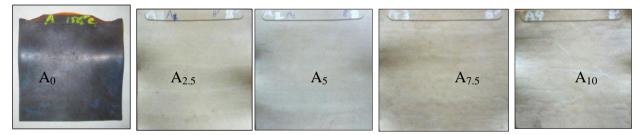


Figure 1 Natural rubber chitosan composites A<sub>0</sub>, A<sub>2.5</sub>, A<sub>5</sub>, A<sub>7.5</sub> and A<sub>10</sub>

# Table 2 Mechanical Properties of the Prepared Natural Rubber Chitosan Composites

No.	Test	Resultant data of various natural rubber chitosan composites						
		A <sub>0</sub>	A <sub>2.5</sub>	$\overline{A}_5$	A <sub>7.5</sub>	A <sub>10</sub>		
1.	Specific gravity	0.93	0.95	0.95	0.96	0.96		
2.	Tensile Strength (MPa)	5.9	3.9	3.3	1.1	1.1		
3.	Elongation at Break (%)	940	883	832	665	667		
4.	Tensile modulus at 300% (M <sub>300</sub> )	0.6	0.6	0.7	0.7	0.8		
5.	Tensile Modulus at 400% ( $M_{400}$ )	0.9	0.9	1.0	1.1	1.1		
6.	Tensile Modulus at 500% (M <sub>500</sub> )	1.2	1.3	1.3	1.4	1.5		
7.	Tear strength (kN/m)	22.9	11.3	8.2	6.1	5.6		

#### Physicomechanical Properties of the Prepared Natural Rubber Chitosan Composites

# Specific gravity

From the experiment data, the sample (without filler) had the lowest specific gravity (0.93). As the filler loading increased, the gravity of the composites also increased. In this experiment data, the composite with 10g chitosan filler loading had the highest density as shown in Figure 2 and Table 2.

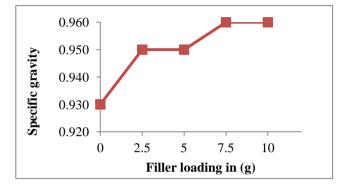


Figure 2 Specific gravity of the prepared natural rubber chitosan composites (A<sub>0</sub>-A<sub>10</sub>)

# **Tensile strength**

The tensile strength is the maximum tensile stress reached in stretching a test piece to its breaking point. The effect of chitosan loading on the tensile properties of chitosan filled rubber composites is shown in Figure 3 and Table 2. It can be observed that the tensile strength of the composites decrease with increasing chitosan loading. This may be due to the size and geometrical factor of the filler, whereby irregular shaped fillers tend to decrease the strength of composites due to the inability of the filler to support the stress transferred from the matrix. Large particle size filler provides a smaller surface area, which give rise to a weaker interaction between the filler and rubber matrix.

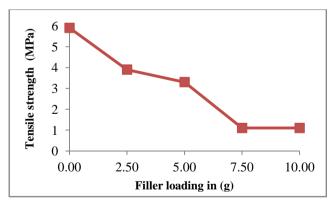


Figure 3 Tensile strength of the prepared natural rubber chitosan composites (A<sub>0</sub>-A<sub>10</sub>)

# **Elongation at break**

According to the result data as shown in Figure 4 and Table 2, it revealed that a falling trend of elongation, as the filler loading increased, when the sample (without chitosan filler) had the highest elongation. This is so because rubber is highly elastic and as the filler loading is

increased, there is an adherence of the filler to the polymer phase which results in the reduction of intermolecular bonds between the rubber chains stiffening of the rubber chain and thus leads to resistance to stretching.

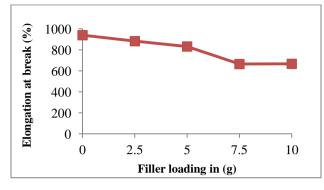
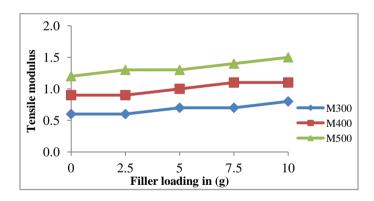


Figure 4 Elongation at break of the prepared natural rubber chitosan composites (A<sub>0</sub>-A<sub>10</sub>)

#### **Tensile modulus**

Modulus is a measure of the fitness of a material that is its resistance to extension. The higher modulus of a material, the less its extend for a given force. From the result data shown in Figure 5 and Table 2, the modulus ( $M_{300}$ ,  $M_{400}$  and  $M_{500}$ ) increase as the filler loading increases. This behavior can be by the fact that adhesion occurred between the filler and the rubber which led to increase in stiffness, rigidity and strength.



**Figure 5** Tensile modulus of the prepared natural rubber chitosan composites  $(A_0-A_{10})$ 

## **Tear strength**

Tear strength used to characterize the tear resistance is one of the important mechanical parameters of rubber products. The experimental data of the tear strength of rubber (without filler) and rubber chitosan composites ( $A_{2.5}$ - $A_{10}$ ) are shown in Figure 6 and Table 2. With increasing chitosan loading, it can be revealed that the tear strength was decreased due to the poor dispersion of chitosan in rubber matrix.

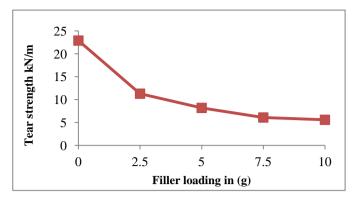


Figure 6 Tear strength of the prepared natural rubber chitosan composites (A<sub>0</sub>-A<sub>10</sub>)

#### **Characterization of the Prepared Natural Rubber Chitosan Composites**

The SEM micrographs show the fracture surfaces of chitosan filled rubber composites without chitosan ( $A_0$ ) and with chitosan of  $A_{2.5}$ ,  $A_5$ ,  $A_{7.5}$  and  $A_{10}$  as shown in Figure 7. With increasing filler at higher 10 % of chitosan loading, the fructured surfaces show poor dispersion and adhesion of chitosan filler whereby there were aggregations and significant detachment of chitosan from the filled compound, this cause major failure in tensile, elogation at break and tear strength properties of the composites.

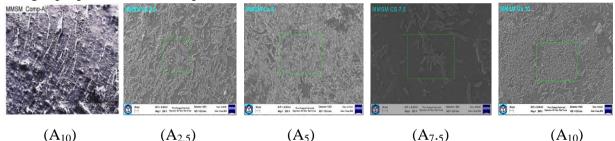


Figure 7 Scanning electron micrographs of the prepared natural rubber chitosan composites  $(A_0-A_{10})$ 

#### Conclusion

From this study, the following conclusion can be drawn.

The physicochemical properties, such as specific gravity, tensile strength, elongation at break, tensile modulus ( $M_{300}$ ,  $M_{400}$  and  $M_{500}$ ) and tear strength of composites with various chitosan loading 0 %, 2.5 %, 5 %, 7.5 % and 10 % were examined. As for the mechanical properties result, the tensile strength, elongation at break and tear strength of rubber chitosan composites decreased with increasing chitosan loading whereas modulus ( $M_{300}$ ,  $M_{400}$  and  $M_{500}$ ) and specific gravity increased. Because of an increase in chitosan loading reduces the elasticity of the prepared composites resulting in more stiff and rigid. The stiffness of the prepared composites increased with increasing chitosan loading due to the ability of chitosan impart greater stiffness to prepared composites. Increase in chitosan loading contributed to an increased in mass of chitosan in the prepared composites, thus the density of the prepared composites increased with increasing chitosan loading. And morphological studies of the tensile fractured surfaces of the vulcanisates indicated that chitosan increased in loading interacts less well. Due to increase of rigid and stiffness of the prepared rubber composites, rubber can be filled with chitosan and can be used for commercial application.

# Acknowledgement

The authors would like to express their profound gratitude to the Department of Higher Education (Yangon Office), Ministry of Education, Yangon, Myanmar, for provision of opportunity to do this research and Myanmar Academy of Arts and Science for allowing to present this paper.

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