CHAPTER 1

INTRODUCTION

1.1. Project Background

Raw elastomer or natural rubber rubber (NR) originally from *Hevea Braziliensis* tree shown in figure 1.0 have poor physical and mechanical properties such as low in tensile strength and low oil resistance. (Ciesielski, 1999). Develop the standard level of properties that fix their own application, NR usually will be mix with various compounding ingredients, shaped and cured. There are three main components; mixing, shaping and testing.

Natural rubber latex has 25% - 40% of rubber contents. This content may be differing due to the type of tree, the tapping method, soil condition and the season. Rubber has particles size of 0.01µm – 5µm as highlighted in Gazely et al, 1990, p.63.

Figure 1.0: *Hevea Braziliensis* tree

Nowadays, there are many achievements in rubber industry; one of the achievements is the ability to improve the physical and mechanical properties of raw elastomer with additive such as silica sand that can be used in tyres, belts and seals. Adding the additives is to control the rubber vulcanisates property and improve some of rubber properties which are strength, performance and service life (Ansarifar, Shiah, and Bennet, 2005).

Silica Sand: Foundry Requirements and Classification and Ismail et al (1994) stated that silica sand is the most essential raw material and importance for its thermal resistance and availability. Silica sand have special feature which is it is a very polar material.

In line with the advanced technology and demand nowadays, collaboration LGM and UTP is to invent a derivation process of new lignin from Malaysian rubber tree. The project is looking on the possibilities of using the patented material composites applications. The composites of NR-RWL will take the advantages of silica sand and the rubber to produces or create a newly formed material.

1.2. Problem Statement

To investigate the good property of rubber wood lignin (RWL) and silica sand by produce a newly improved rubber.

A derivation process of new lignin from Malaysian rubber tree was invented. There are no establishment on relevant physical and mechanical properties of the rubber wood lignin composites with silica sand addition.

1.3. Objectives

1. To study and establish the physical and mechanical properties of the RWL composites.
2. To evaluate the effect of RWL composites with SiO_2 addition on oil resistance.

1.4. Scope of study

The project covers the understanding of composites advantages. Student will enhance the knowledge about reinforcement and binder good properties mixing together will formed newly improvise material.

In this project, silica will act as additive in the natural rubber-lignin composites to enhance its physical and mechanical properties such as tensile, stress at break and oil resistance.

1.5. Relevancy of The Project

Composites study is relevant to my field because advance engineering material is one of the Mechanical Engineering areas.

Applying several Mechanical Engineering courses while conducting the project such as Introduction to Material Science, Engineering Material and Advance Engineering Material.

1.6. Feasibility of Study

This project is the collaboration with Rubber Research Institute of Malaysia (RRIM) and it is in Final Year Project scope and time frame.

CHAPTER 2

LITERATURE REVIEW

2.1. Natural Rubber

Natural rubber is widely used elastomer in automotive application or industrial. Special features of natural rubber is their self-reinforcement character, originate from strain induced crystallization. Addition of additive like silica can improvise the rubber properties by increase its rigidity, its abrasion resistance, tear strength, and stress at break. The improvement is due to the complex interplay between the polymer and the additive good properties. Introduction of silica additive will increases the local strain. Silica contributes to crack growth resistance in rubber. (Chenal et al. 2007)

According to Mars and Fateni (2004), rubber has the ability to withstand very large strain without permanent deformation. Several applications are vibration insulator, belts and impact bumper. Fatigue testing is used to determine how many cycles will the composite can withstand before it goes to failures. It will base on fracture mechanics, focusing on predicting the growth of particular crack.

When the rubber latex is left for a week or more, this will lead to the creaming effect. The amount of rubber that can pass through the filter is to be defined as filterability before clogging happen.

Drying of rubber latex which is consisting of almost 40% water. There are three stages of drying processes; first is free evaporation of water from the film surface, second, irreversible contact at volume fraction when the particles are forced, the evaporation will decreases as particles coalesce under interfacial tension. The third stages is small exponential drying rate when water diffuses through capillary channel or through the polymer itself.

After completing the mixing and the latex is stored for several days which is ranging from one to seven days is called maturation. The aim is to destroyed air bubbles in rubber latex. (Gazely et al.)

2.2. Lignin

Lignin was second abundance natural aromatic polymer in the world, it provide mechanical strength to the plant because it act as matrix component of cellulose and hemicellulose in plant cell wall. (Lora, and Glasser 2001)

Gregorova, kosikova and Moravcik (2006) explained that lignin consist of three dimensional amorphous natural polymer and happen in the plant and function as stabilizer against mechanical, biochemical and environmental stresses. Nowadays, lignin gets more attention due to its properties; crosslinked structure with tetra-branched points, stabilizing effect, reinforcement effect and biodegradability. Lignin also a complex phenolic polymers of intermediate molecular weight. Figure 2.0 illustrates example of several types of hydroxyl groups in lignin.

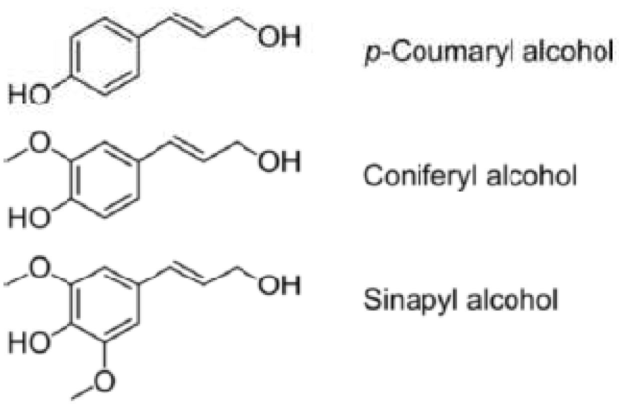

Figure 2.0: Types of hydroxyl group in lignin

2.3. Silica Sand

Silica sand is abundantly available in the world and commonly used in ceramic applications. Silica sand have several good properties as; high hardness, high strength, high chemical resistance, high temperature resistance, and good insulation.

Silica sand used for this study has the size of less than 45µm which is produces by using low speed ball milling having speed of 90rpm and grinding with zirconia ball.

2.4. Rubber-Lignin Composites with Silica Sand as Filler

Mixing process is the most important part in determining the properties of rubber. There are four stages. At stage two and three, zinc oxide which act as activator were added. At the final stages, there will be adding sulphur act as curing agent and antioxidant. (Ansarifar et al. 2005)

Arun et al. (2010) found that the tensile strength will slightly increase initially, and decreases continuously with the amount of additive. This is maybe due to poor latex interaction and non-uniform distribution shown in Figure 2.1.

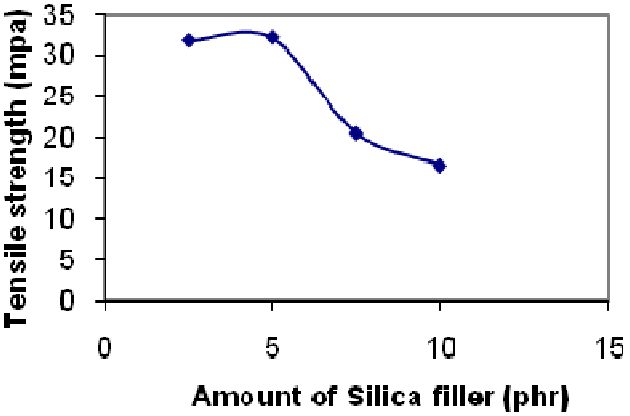

Figure 2.1: Variation of tensile strength of latex-silica composites with the amount of silica

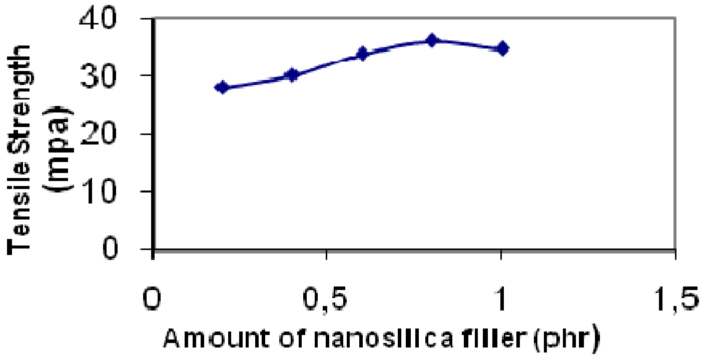

Figure 2.2: Variation of tensile sstrength of latex-slica nanocomposites with the amount of nanosilica

Figure 2.2 indicates that the tensile increase gradually comparing with the silica additive. This behaviour due to higher homogenous distribution of nanosilica in the latex compound.

There are also a study indicate that different mixing time will affect the silica agglomerate as shown in Figure 2.3 (a) and Figure 2.3 (b) for mixing time 4min and 11min when silica having the particle size of larger than 40µm. The mixing plays a big role in determining the quality of silica disperse because during mixing the rubber break. (Ansarifar et al. 2005).

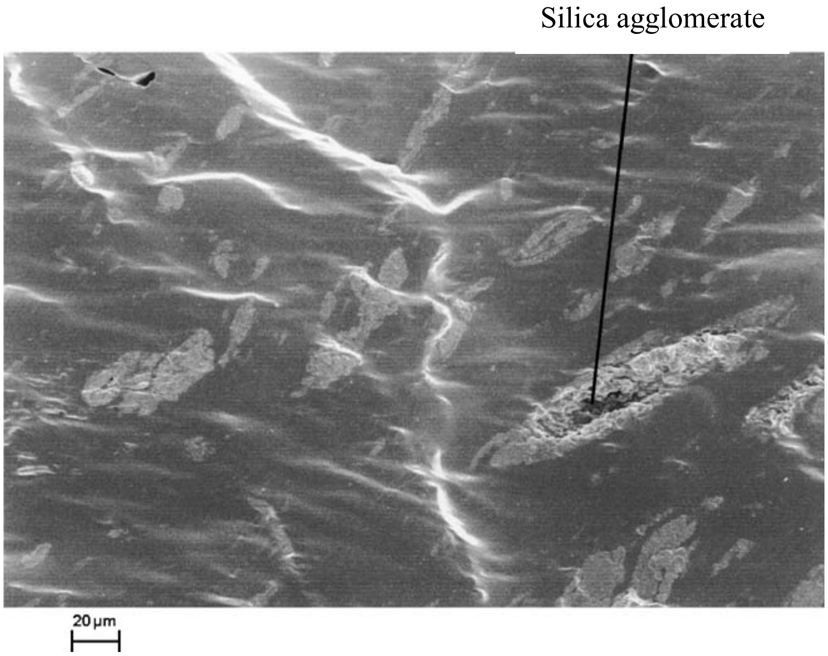

Figure 2.3 (a): SEM micrograph showing inadequate silica dispersion in rubber. Mixing time 4min

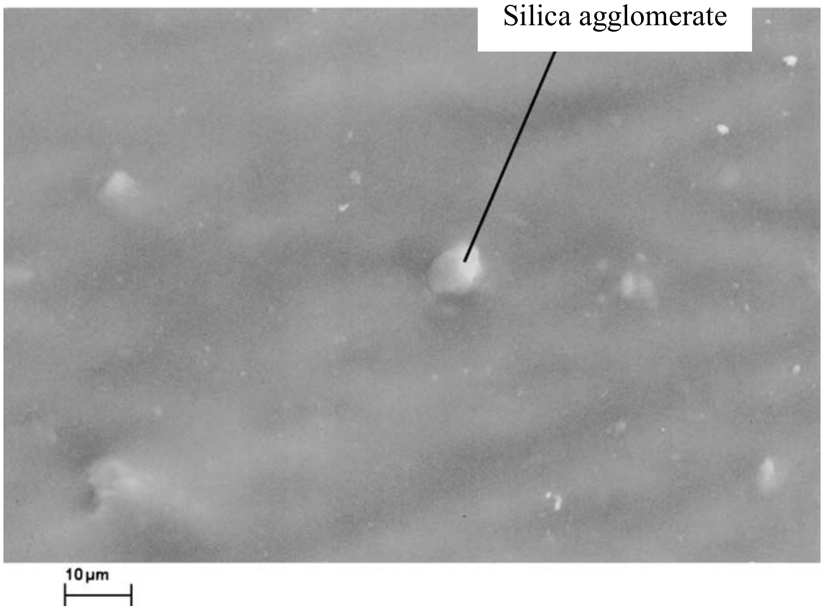

Figure 2.3 (a): SEM micrograph showing silica agglomerate in rubber. Mixing time 11min

CHAPTER 3
METHODOLOGY/ PROJECT WORKS

3.1. Project Activities

Figure 3.0 shown briefly the project flow from preparing the silica sand until discussion of the result obtain after mechanical testing. Mechanical testing consist of tensile testing, oil swelling testing and SEM analysis.

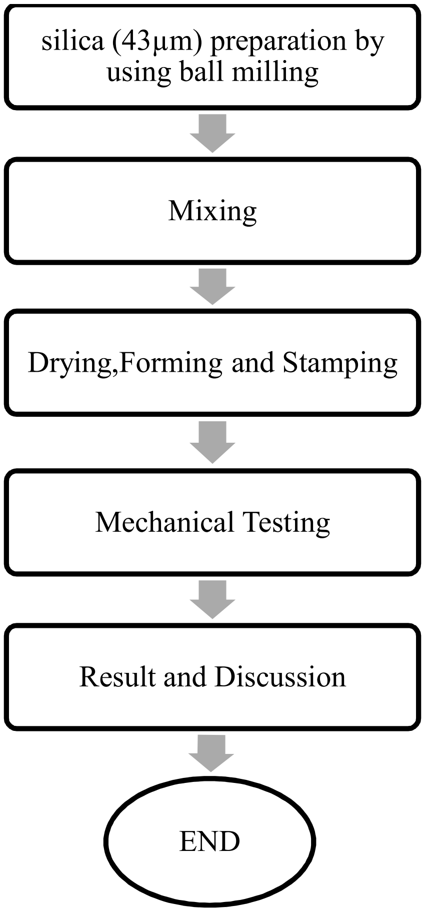

Figure 3.0: Project Flow

3.2. Material

The material get from *Lembaga Getah Malaysia* is raw rubber latex in Figure 3.1 (a) and RWL in Figure 3.1 (b). The reinforcement is silica sand around Tronoh (Universiti Teknologi PETRONAS, UTP) shown in Figure 3.1(c).

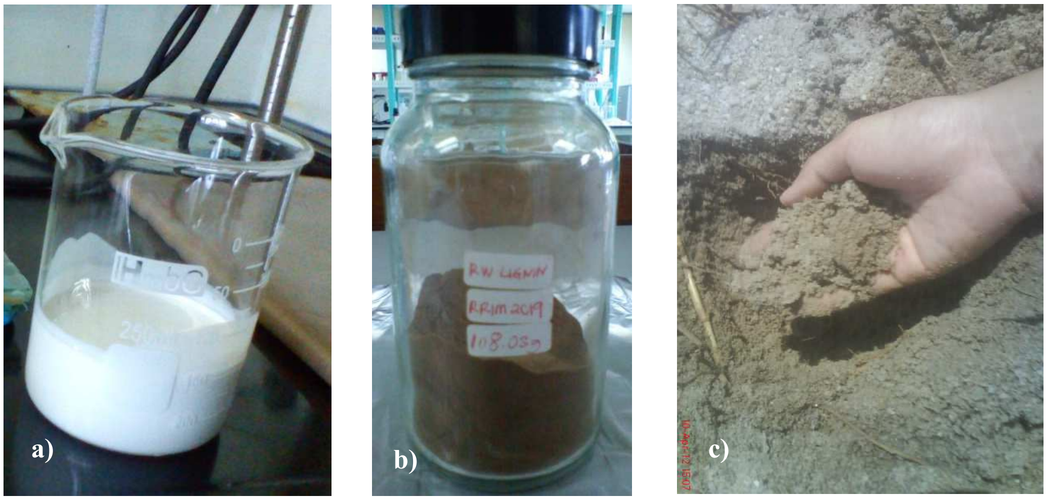

Figure3.1: a) Rubber latex, b) Rubber wood lignin, c) Silica sand

3.3. Silica Sand Preparation Procedure

The equipment used in silica sand ball milling process are Ball Mill (US stone ware) in Figure 3.2(a), ceramic jar in Figure 3.2(b), zirconia ball for grinding in Figure 3.2(c) and siever pan in Figure 3.2 (d).

Figure 3.2(a): Ball Mill (US stoneware)

Figure 3.2(b): Ceramic jar

Figure 3.4(c): Zirconia ball

Figure 3.2(d): Siever pan

Figure 3.3 illustrates the process flow for the production of silica sand fine particles.

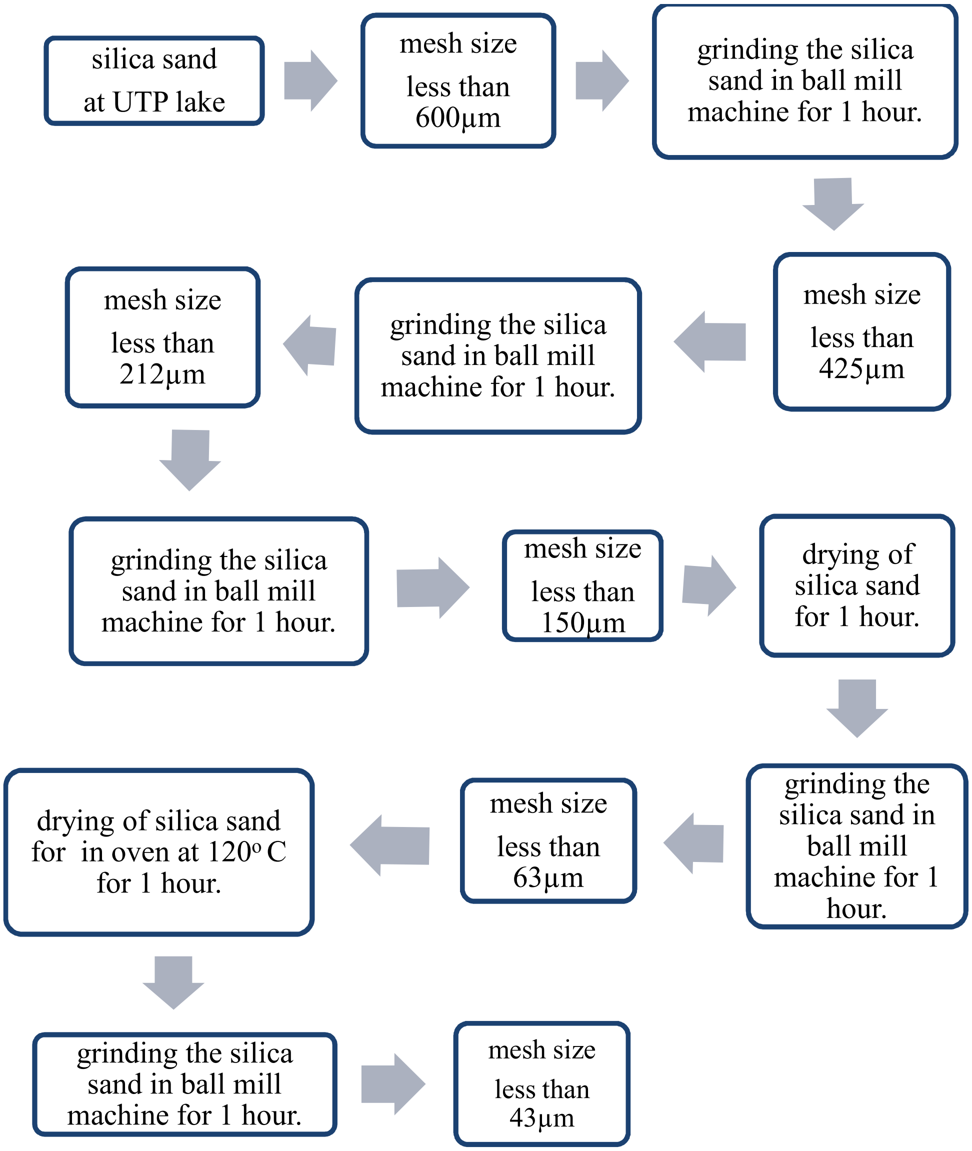

Figure 3.3: Production of silica sand process flow

3.4. Rubber-Lignin Composite with Sio₂ Addition Procedure

Total volume of rubber latex used for one batch l is 200 ml rubber latex (30% rubber content). This batch contains 60g rubber by weight (200 ml x 30%). Vary silica loading according to Table 3.0 with RWL maintained at 10% loading per rubber weight.

Table 3.0: Silica with Varying Weight Percent Loading

	Silica (%) per rubber content	RWL (%) per rubber content	Weight of silica (g)	Weight of RWL (g)
Film batch 1(control)	0	0	0	0
Film batch 2	6	0	3.6	0
Film batch 3	12	0	7.2	0
Film batch 4	6	10	3.6	6.0
Film batch 5	12	10	7.2	6.0

Sulphur and zinc oxide were added at 1% and 0.5% per dry rubber weight respectively. RWL is mixed with silica and the mixture is then added together with natural rubber latex in a suitable vessel. The mixture is stirred using high shear impeller at 280 rpm. Glass mould was used to obtain films of approximately 0.5 mm in thickness. Drying is carried out at ambient temperature until sufficient dryness and followed by leaching and heating at 100°C for 30 min.

Process flow of mixing the silica sand, RWL is shown in Figure 3.9. This whole procedure takes place in Rubber Research Institute of Malaysia (RRIM), Sungai Buloh, Selangor.

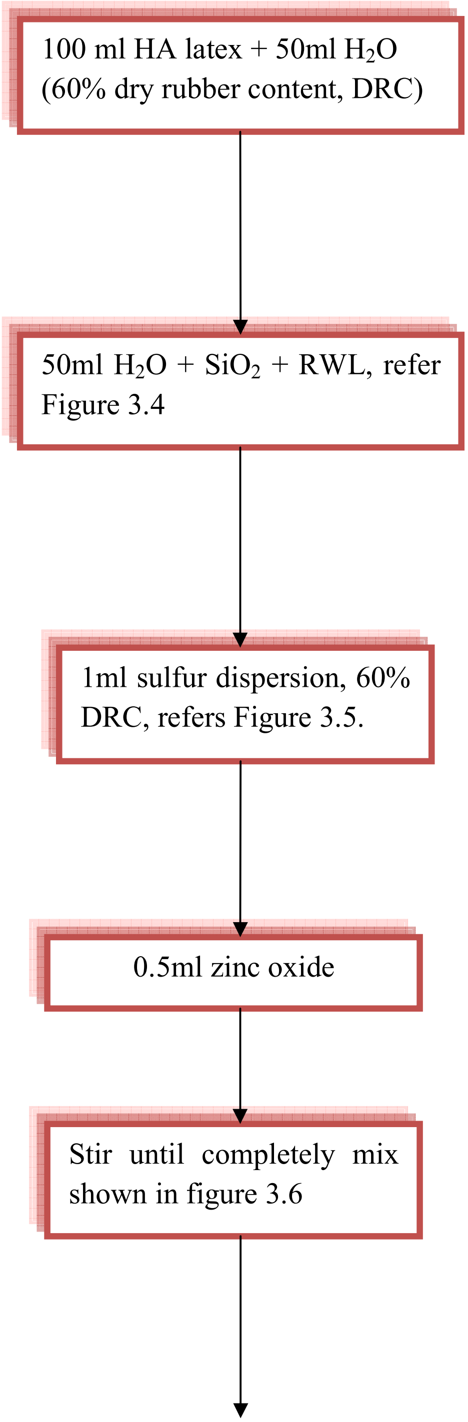

Figure3.4: mixing HA latex, H_2O, SiO_2 and RWL

Figure 3.5: sulphur dispersion

Figure 3.6: stirring until completely mix

Figure 3.7: Maturation

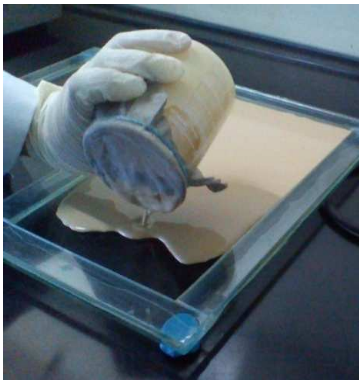

Figure 3.9: Filtering and casting

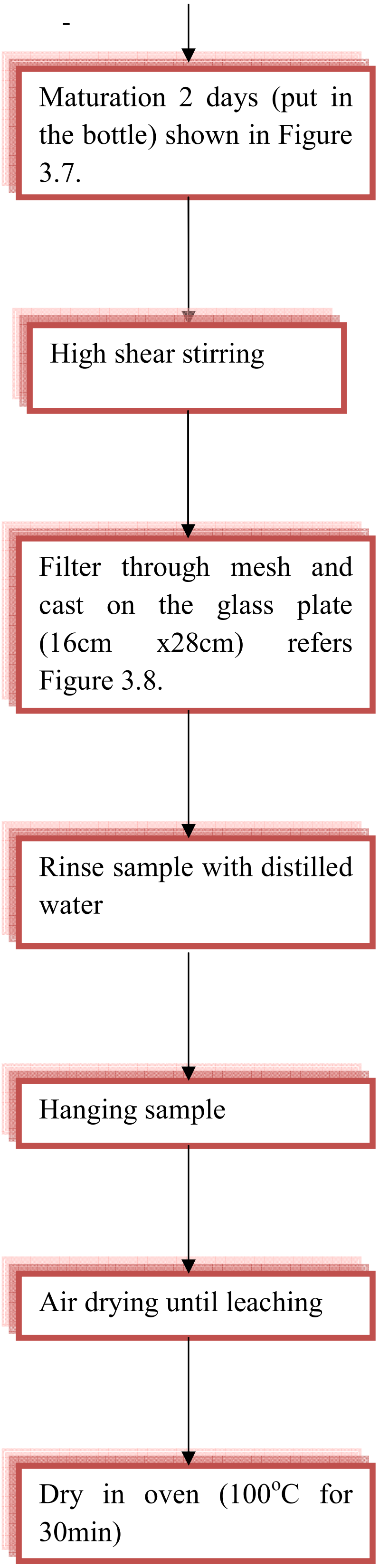

Figure 3.9: Rubber production mixing process flow.

Shaping

Shaping the rubber sheet into the dumbbell shape in Figure 3.9 is need for cutting or stamping. It requires dumbbell sample puncher as shown in Figure 3.10 that only have the die and a press. The cutter must be very shape and free from nicks or unevenness in the cutting edges.

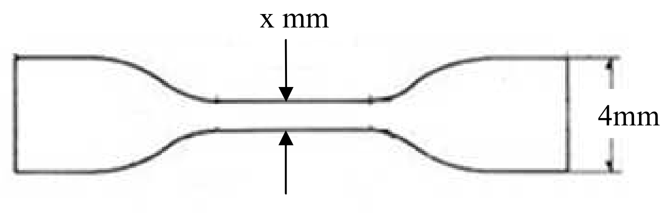

Figure 3.10: Dumbbell shape

Figure 3.11: Dumbbell sample puncher

Tensile testing (ASTM D412)

According to the Maker of Material Testing System, stress-strain measured carried out by stretching dumbbell shaped specimen. Record the initial length, thickness, and width. This testing is performing at the room temperature.

1. Put the specimen into the tensile gripper shown in Figure 3.12
2. Extensometer is attach to the sample
3. Set the speed to 500mm/min to separation the tensile grips.
4. Test end when the specimen break(rupture)

From the test, several analyses we can obtain are:

1. Tensile Yield Stress
2. Tensile Yield Strain
3. Tensile Strain at Rupture
4. Elongation at Rupture

Figure 3.12: Tensile Testing Machine

Oil Swelling Testing (ASTM D471)

Refer to ASTM international, Standard Test Method for Rubber Property-Effect of Liquid, swelling test is a method for evaluate the comparative ability of rubber and rubber-like composition to withstand the effect of liquid. In this standard procedure we used IRM 903 shown in Figure 3.13 as standard test liquid. This testing will be the influence of liquids to the specimens. The testing is conduct under room temperature, 23±2 °C and immersion for 24hr.

The test specimen is in circular shape with diameter of 22mm in Figure 3.14. We used 6 specimens for each batch. Weight each sample for every batch before and after immersion. Put each sample into the 30ml glass bottle as in Figure 3.15 and left it for 24hr.

Figure 3.13: IRM 903 oil

Figure 3.14: Sample cut in circular shape

Figure 3.15: Sample immerse in IRM 903

Calculation for percent change in mass as in equation (1):

$$\Delta M\% = \frac{(M_2 - M_1)}{M_1} \times 100 \tag{1}$$

Where:
M_1 = mass before immersion (g)
M_2 = mass after immersion (g)

3.5. Gantt Chart

Gantt chart for activities planned along for this final year project I (FYP I) is shown in Table 3.1 and final year project II (FYP II) is shown in Table 3.2 and suggested milestones.

Table 3.1: FYP I Gantt chart and Milestone

No	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
									Mid-Semester Break							
1	Selection of Project Topic	🟦	🟦													
2	Preliminary Research Work		🟦	🟦	🟦	🟦										
3	Submission of Extended Proposal						●									
4	Oral Proposal Defence									🟦	🟦					
5	Project Work Continues											🟦	🟦	🟦		
6	Submission of Interim Draft Report														●	
7	Submission of Finalized Interim Report															●

Table 3.2: FYP II Gantt chart and Milestone

No	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
									Mid-Semester Break								
1	Project Work Continues	■	■	■	■	■	■	■									
2	Submission of Progress Report									●							
3	Project Work Continues									■	■	■	■	■			
4	Pre-SIDEX												●				
5	Submission of Draft Report													●			
6	Submission of Dissertation (soft bound)														●		
7	Submission of Technical Paper														●		
8	Oral Presentation															●	
9	Submission of Project Dissertation (hard bound)																●

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Result

4.1.1. Silica Sand

The natural silica sand taken near the UTP Lake was mesh into less than 600µm shown in Figure 4.0. The final product after go through grinding with low speed ball mill with zirconia ball at speed of 90rpm shown in Figure 4.1.

Figure 4.0: Silica sand (less than 600µm)

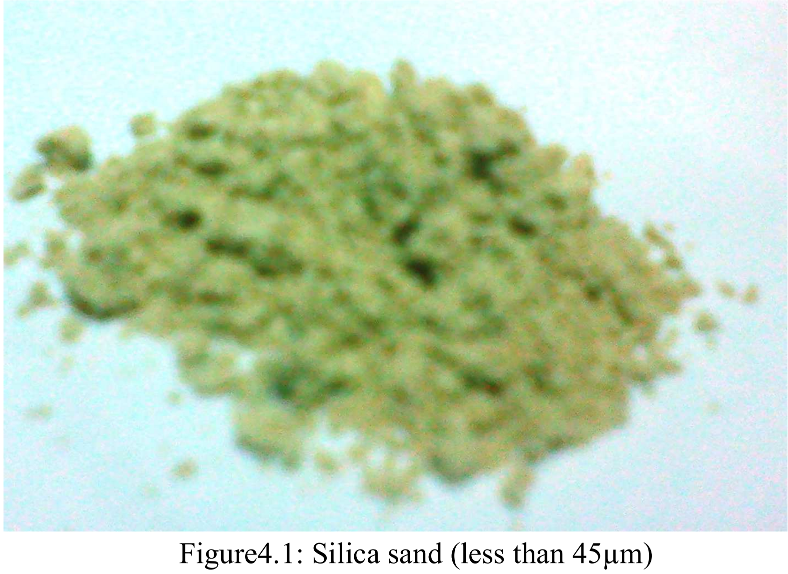

Figure4.1: Silica sand (less than 45µm)

4.1.2. Tensile Testing for Rubber Latex, Rubber Latex with Silica and Rubber latex with RWL and Silica.

Table 4.0 indicate the silica (%) per rubber content and RWL (%) per rubber content for each batch that we used in the testing.

Table 4.0: Silica with Varying Weight Percent Loading

	Silica (%) per rubber content	RWL (%) per rubber content	Weight of silica (g)	Weight of lignin (g)
Film batch 1(control)	0	0	0	0
Film batch 2	6	0	3.6	0
Film batch 3	12	0	7.2	0
Film batch 4	6	10	3.6	6.0
Film batch 5	12	10	7.2	6.0

Stress-strain curve for tensile testing result for control batch which have no RWL and silica is shown in Figure 4.2.

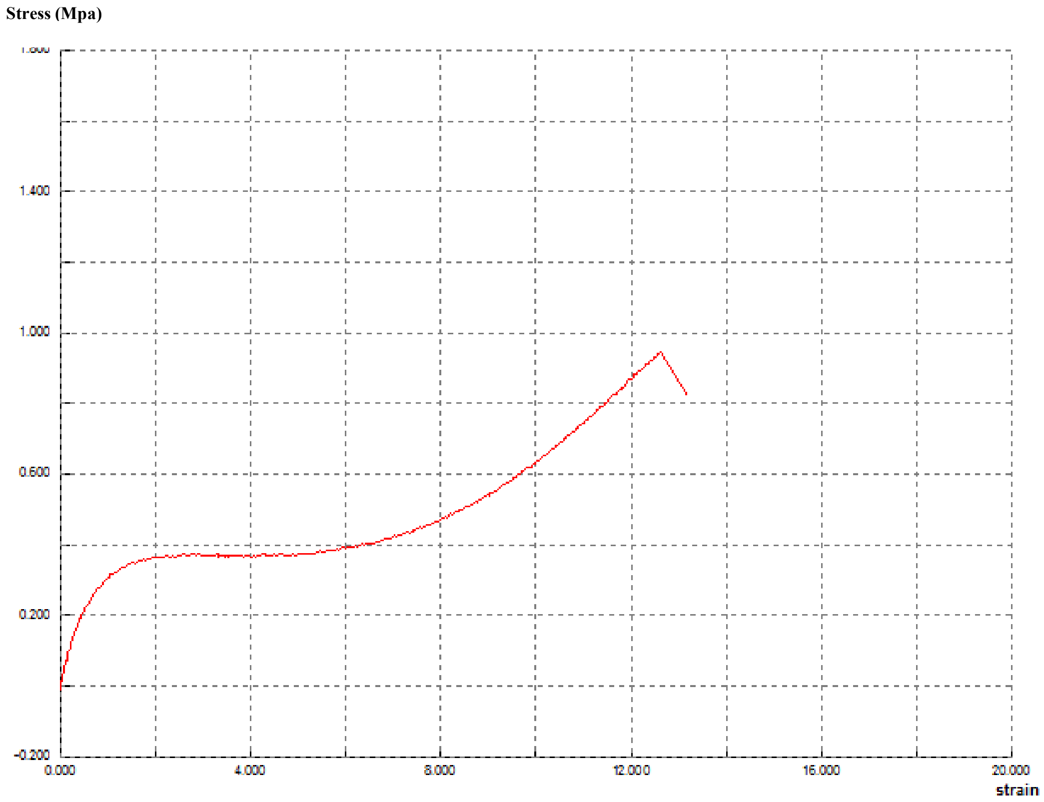

Figure 4.2: stress-strain curve for latex Batch 1 (control)

Figure 4.3 and Figure 4.4 have the silica in the rubber latex which are 6 (%) silica per rubber content and 12 (%) silica per rubber content. Figure 3.5 and Figure 3.6 illustrates the stress-strain curve when 10 (%) RWL and silica is added. Figure 3.5 have 6 (%) silica per rubber content and Figure 3.6 have 12 (%) silica per rubber content. The difference of their individual stress-strain curve can be observed.

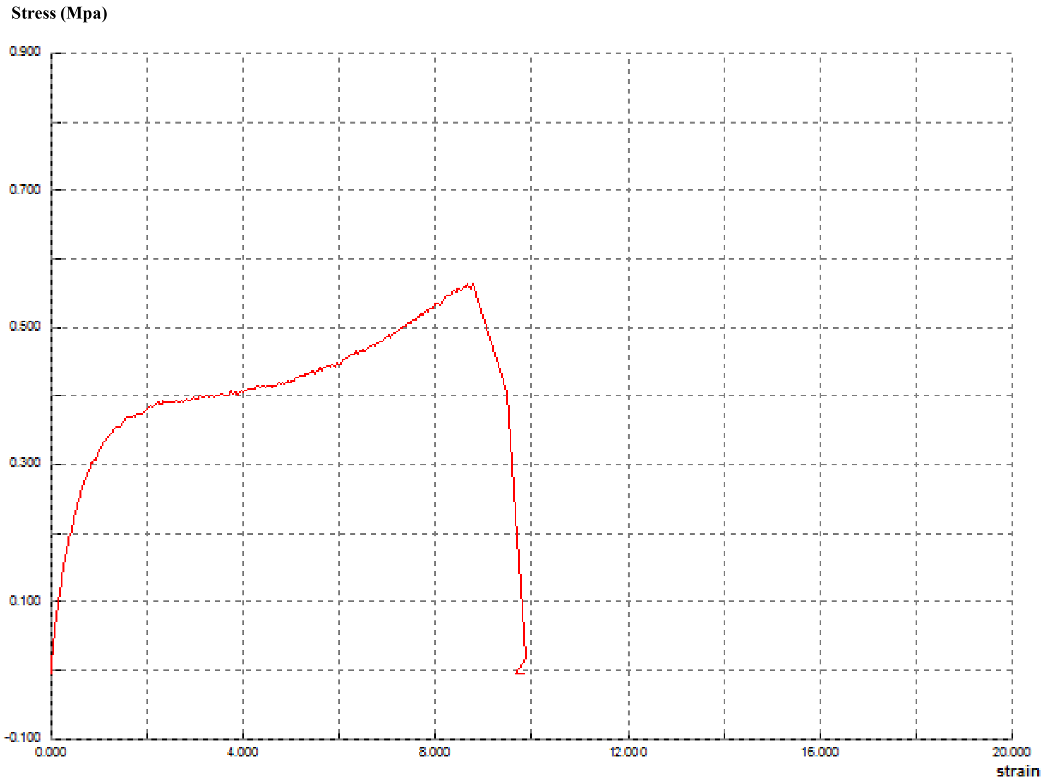

Figure 4.3: stress-strain curve for latex Batch 2 (silica 6%)

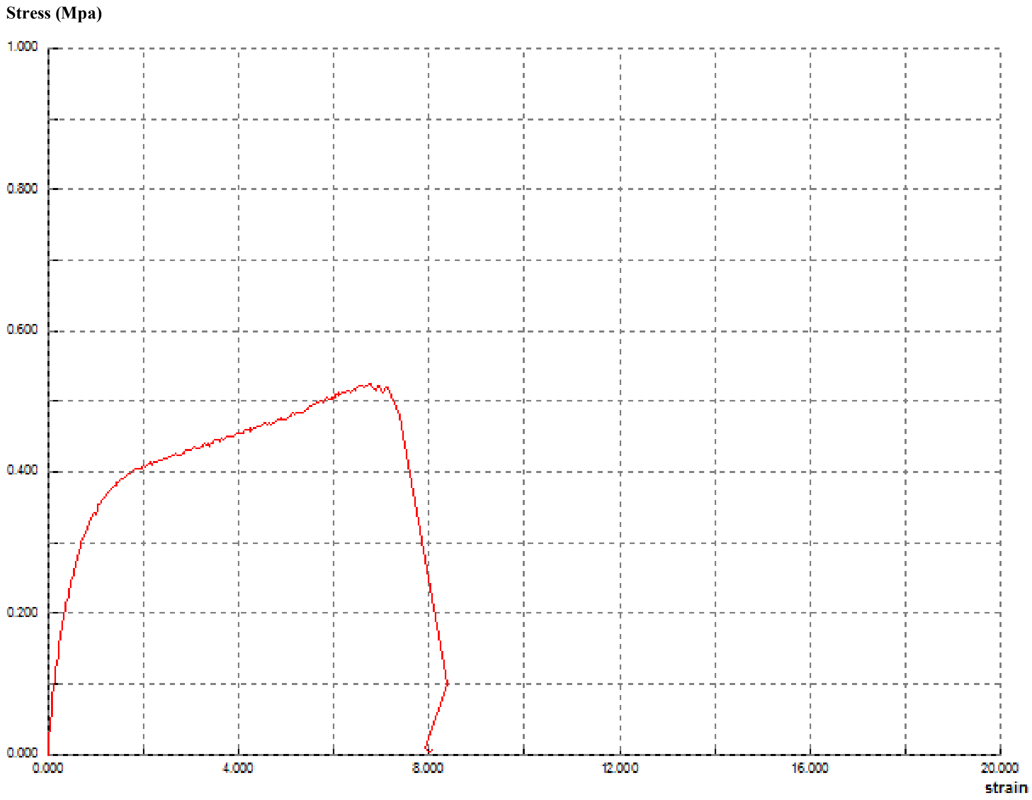

Figure 4.4: Stress-Strain curve for latex Batch 3 (silica 12%)

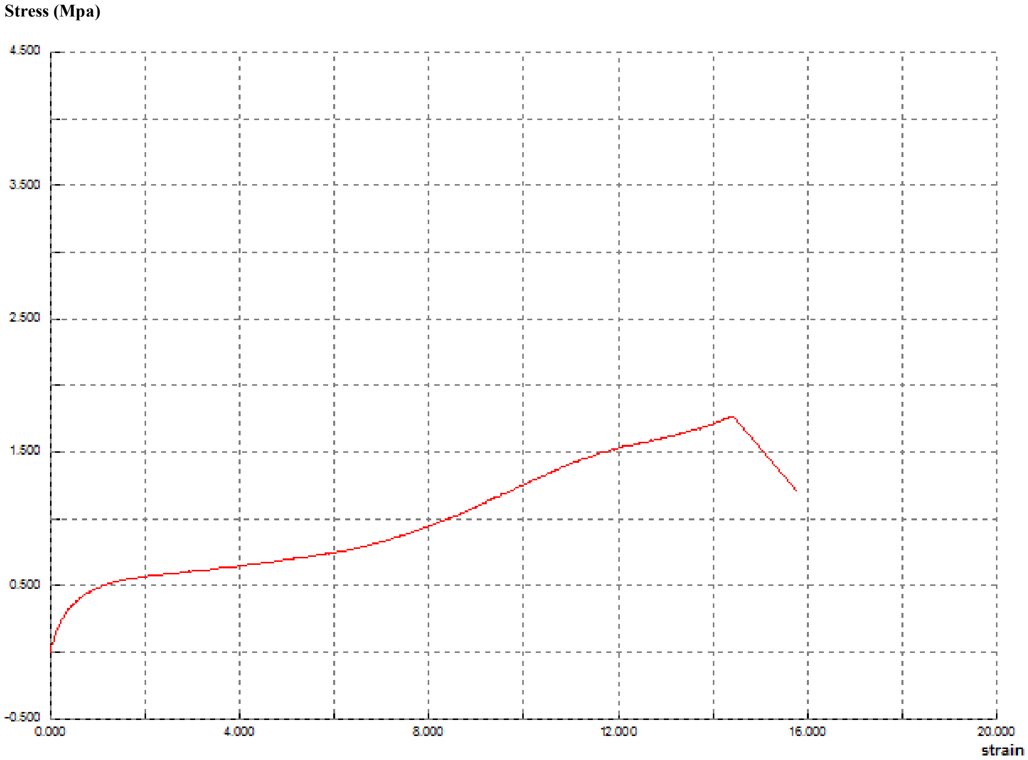

Figure 4.5: stress-strain curve for latex batch 4 (silica 6%, lignin 10%)

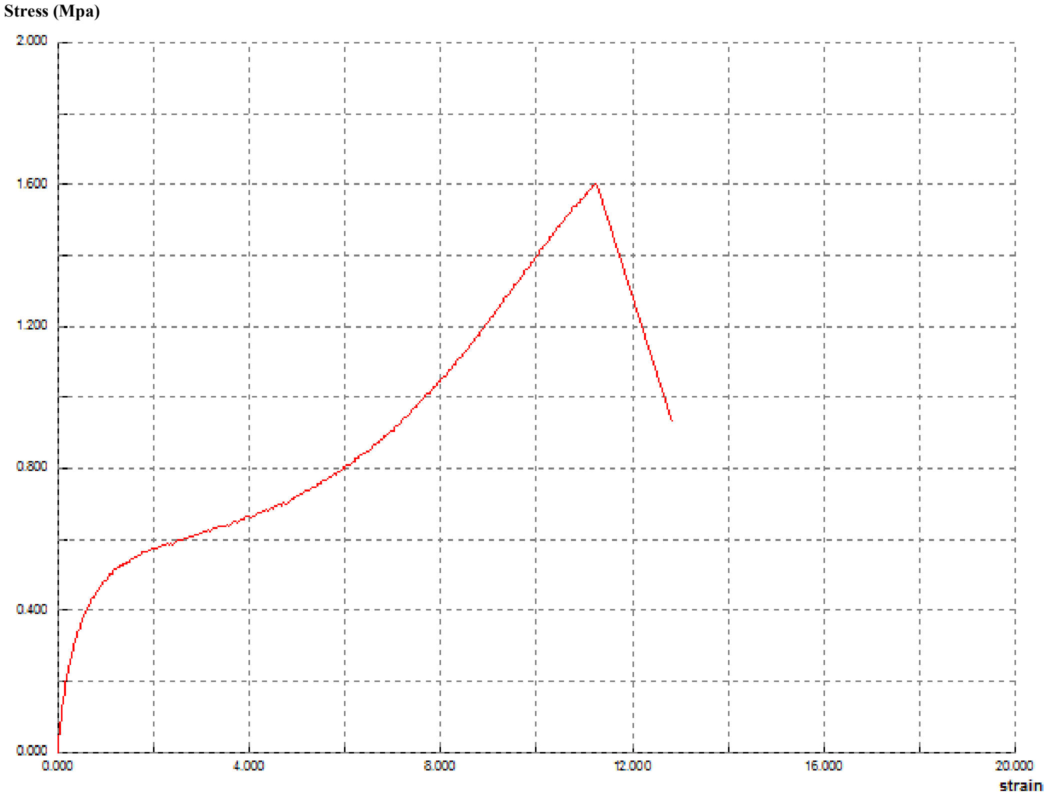

Figure 4.6: stress-strain curve for latex batch 5 (silica 12%, lignin 10%)

Table 4.1 indicated the summary result obtain from the stress-strain curve for all rubber batches. From this table we can see clearly the difference n value for batches. Observe the pattern in Figure 4.7 for stress-strain at peak for every batch and Figure 4.8 for elastic modulus for each batch.

Table 4.1: Result obtains from stress- strain curve of all rubber latex batches

Batch no.	Maximum force (N)	Stress at Peak (MPa)	Strain at Peak	Energy to Break (kJ)	Elastic Modulus (MPa)	Elong at Peak
1	5.37	0.961	12.94	0.189	0.043	1294.053
2	3.209	0.575	8.622	0.116	0.095	862.186
3	2.81	0.507	6.846	0.088	0.142	684.588
4	8.968	1.77	14.401	0.41	0.085	1440.071
5	8.379	1.498	11.364	0.298	0.100	1136.383

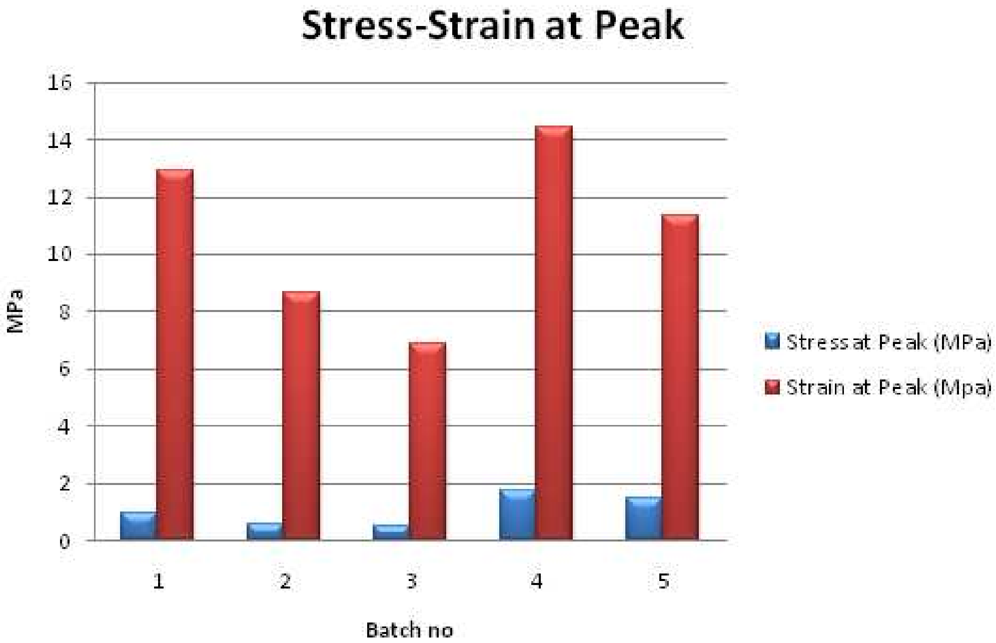

Figure 4.7: Stress-strain at peak

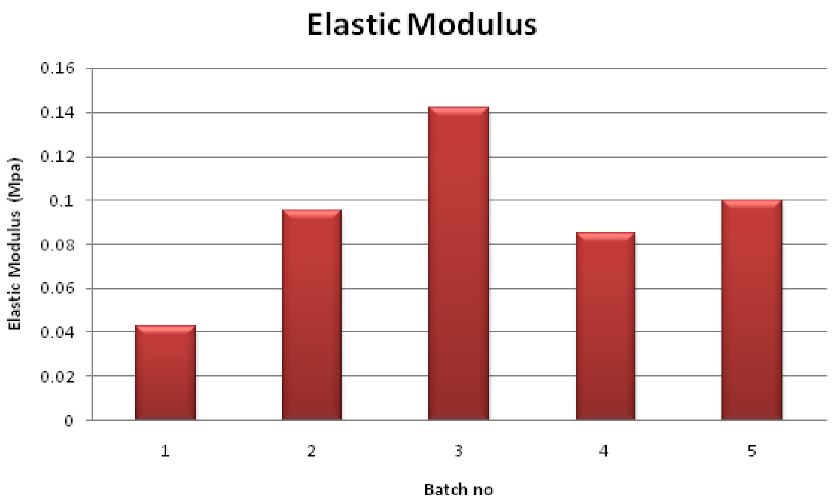

Figure 4.8: Elastic Modulus for each batch

4.1.3. Oil Swelling Testing

Oil swelling test result is summarize in Table 4.2 and the difference in percentage oil swelling can be seen clearly in Figure 4.8.

Table 4.2: Oil swelling test result

sample batch	RWL (%) per rubber content	Silica (%) per rubber content	Average weight before swelling (g)	Average weight after swelling (g)	% oil swelling
1.	0	0	0.5248	1.1845	125.7
2.	0	6	0.4699	1.1359	141.7
3.	0	12	0.5216	1.1039	111.6
4.	10	6	0.4757	1.0488	120.4
5.	10	12	0.5343	1.0262	92.1

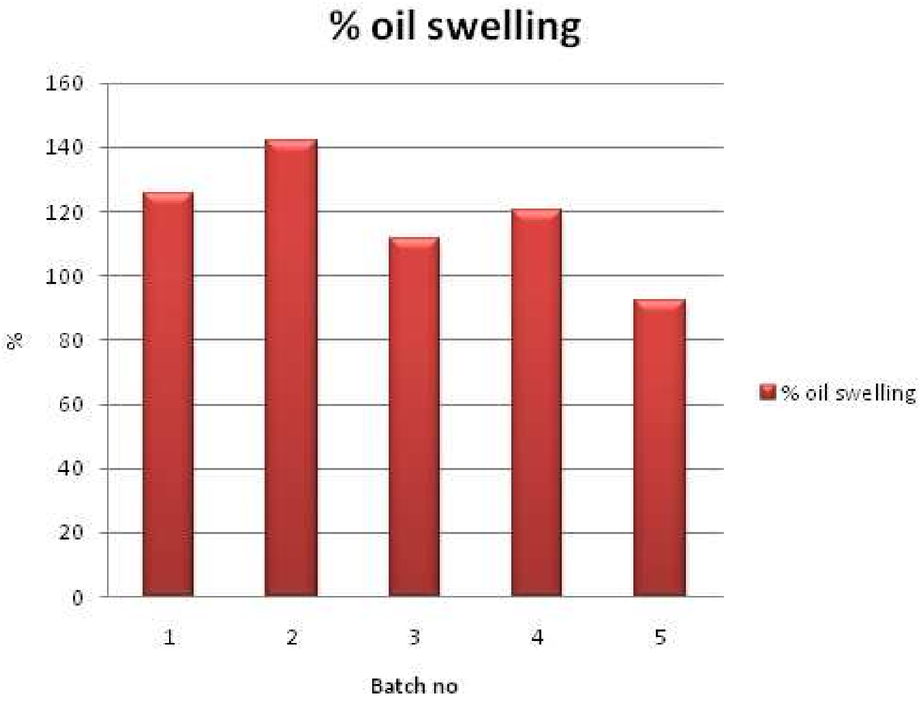

Figure 4.9: Percentage oil swelling

4.1.4. Field Emission Scanning Electron Microscope (FESEM) analysis

FESEM provides narrower probing beam at low as well as high electroenergy, it will give better result resolution and minimize sample changing and damage. Figure 4.9 shown FESEM micrograph with 100X magnification of control batch (Batch 1). Figure 4.10 illustrate FESEM micrograph with 1.00 K X magnification of Batch 2 which have 6 (%) silica per rubber content and Figure 4.11(a) and (b) indicated FESEM micrograph with 1.00 K X and 100X magnification of Batch 4 which have 6 (%) silica per rubber content and 10 (%) RWL per rubber content.

Figure 4.10: FESEM for Batch 1 (control)

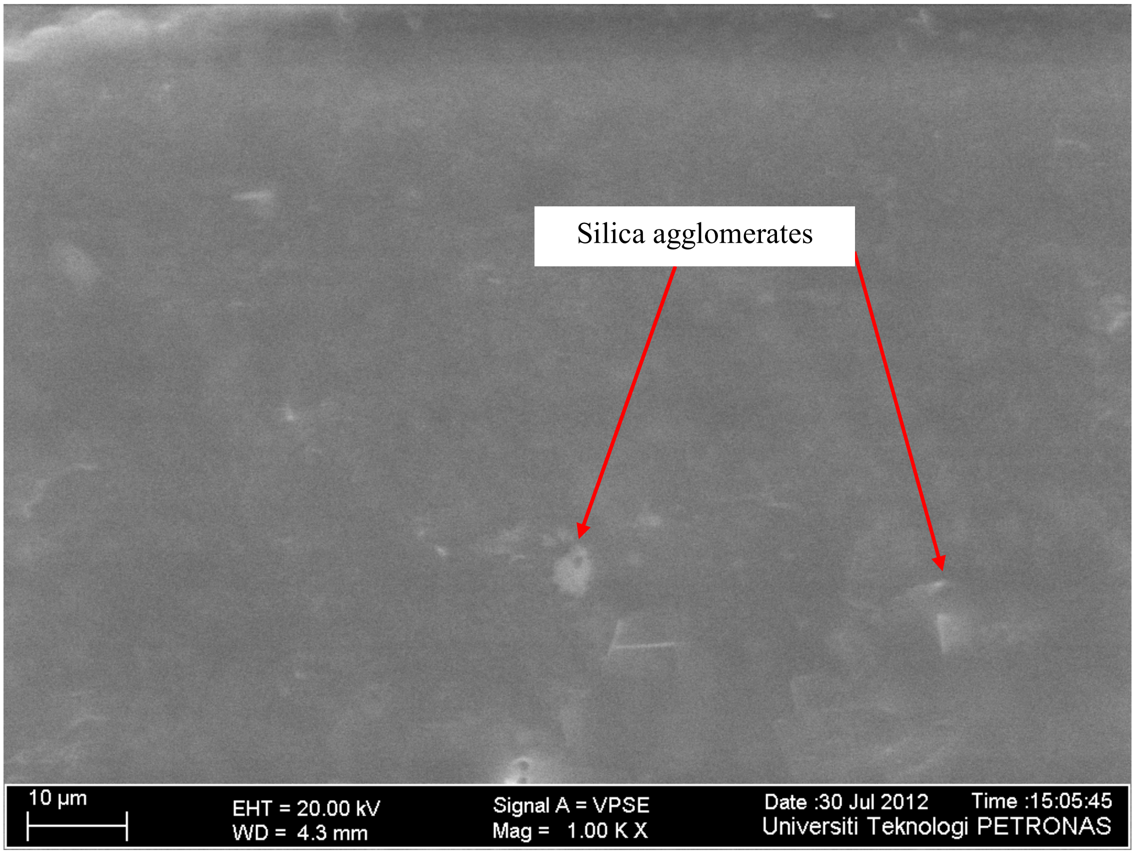
Silica agglomerates

Figure 4.11: FESEM for Batch 2

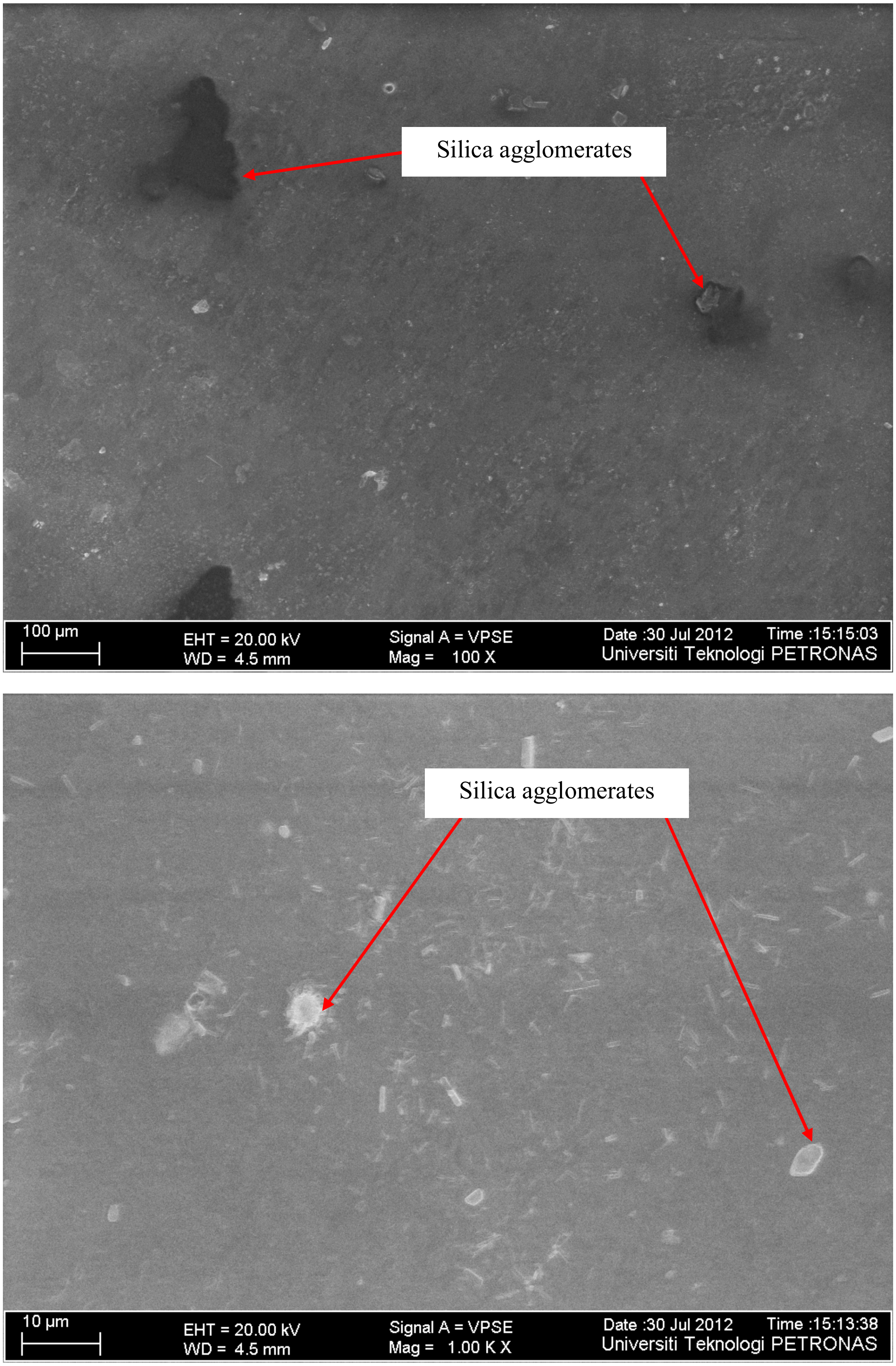

Figure 4.12(a) and (b): FESEM for Batch 4

4.2. Discussion

4.2.1. Tensile Testing for Rubber Latex, Rubber Latex with Silica and Rubber latex with RWL and Silica.

Comparing Figure 4.2 with Figure 4.3 and Figure 4.4, we can notice that the graph was sharp increase and then suddenly break as we add more silica sand into the sample. Now, compare it with Figure 4.5 and Figure 4.6 the graph shows the samples breaks after the graph gradually increased with addition of lignin. This shown that amount of the silica give huge effect on the sample. These two behaviours due to the poor silica distribution cause by the particles size of silica are bigger than the particles size of rubber latex. The stress-strain at peak for each batch can be observed clearly on Figure 4.7. Elastic modulus in Figure 4.8 also can be obtained by using equation (2).

$$\text{Elastic modulus, E} = \frac{\text{Tensile stress,}\sigma}{\text{Tensile strain,}\epsilon} \tag{2}$$

Based on Table 4.1, rubber latex (control) has maximum force of 5.37MPa compare to rubber latex with silica sand itself, the maximum force was decreasing with increasing of percentage of silica sand (Batch 2 and Batch 3). In the presence of lignin in the natural rubber latex, it show increasing in the maximum force from 5.37MPa for batch 1 increased until 8.968MPa in Batch 4 but decrease with the increasing in silica sand to 8.379Mpa in Batch 5. Kosikova, Gregorova, Osvald and Krajcovocova (2006) claims that lignin have softly influenced the natural rubber cure characteristic.

Now, we comparing elastic modulus of these sample, stiffer the slope of the graph, the higher elastic modulus. Higher elastic modulus means the material have lower tendencies to deflect. As we can see in Table 4.1, the elastic modulus increases from Batch 1 until Batch 3. The same pattern can be observed for Batch 4 and Batch 5.

The decreasing in maximum force for rubber latex-silica sand may due to poor interaction between NR latex and silica and can also caused by non-uniform in filler distribution. The elongation at break is also found to be decreased continuously due to the poor NR latex-filler interaction and non-uniform distribution during the mixing and dispersion process. With RWL as additives in the NR latex and its good properties in provide mechanical strength to plant it increasing the elongation before rupture and high maximum force need to apply to the specimen before it failed.

4.2.2. Oil Swelling Testing

Oil swelling testing done for evaluate the characteristic of rubber when immersed in oil. This result is good in the application of vehicle gasket and conveyor belt. As a result of this testing was Batch 1 (control) have 125% was less resistance to oil due to the unsaturated polymer which is degrades in oil. Oil resistance was far worse in when the percentage of silica only 6%, 141.7% for Batch 2 and 120.4% for Batch 4. When percentage loading of silica increase to 12 %, the oil resistance was improved to 111.6% for Batch 3 and 92.1% for Batch 5 as compare to Batch 2 and Batch 4. This result can been observed clearly in Table 4.2 and Figure 4.9.

4.2.3. FESEM Analysis

FESEM analysis was done in order to analyze the distribution of silica in natural rubber. Figure 4.10 is a control batch or reference batch to be compare with Batch 2 and Batch 4. Batch 2 showing less silica agglomerate as in Figure 4.11 than silica agglomerate in Batch 4 as shown in Figure 4.12(a) and (b). This is proved that silica is not well distributed when mixing with rubber compound. www.rtvanderblit.com/ Non-Black Filler for Rubber highlighted that the size particles of the filler in the rubber shoulde be in the range of 1-10µm are primaryly used but the most recommended range is 0.01-0.1µm in this recommended range they are believe can reinforcing and improve the rubber property as shown in Figure 4.13.

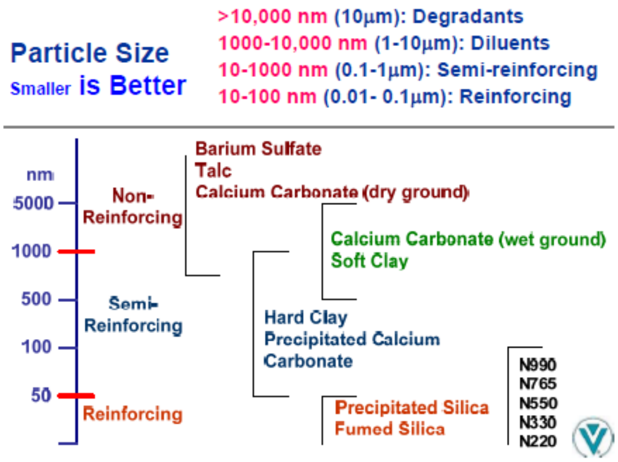

Figure 4.13: Particle size ranging in rubber(www.rtvanderblit.com/Non-Black Filler for Rubber, August 12,2012)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1. Conclusion

As the conclusion, the study is relevance to the objectives which are to develop the physical and mechanical properties of the rubber wood lignin composites. This project will able us to comparing the stress-strain curve of rubber lignin with stress-strain curve of rubber lignin with silica addition. When compare elastic modulus of these sample, stiffer the slope of the graph, the higher elastic modulus. The elastic modulus increasing from 0.043MPa for Batch 1 to 0.095MPa for Batch 2 and 0.142MPa for Batch 3. The same pattern can be observed for Batch 4 which has a value of 0.085MPa and 0.100 for Batch 5. This is indicate that mechanical properties in elastic modulus for rubber-lignin with SiO_2 addition show decreasing which means material behave more like brittle material. The presences of silica contribute to the decreasing in properties of rubber caused by the silica particles size that had been used which is bigger than the rubber particles size. It happens to be poor interaction between rubber latex-silica. The silica sizes that can give improve to the rubber properties is nano size particles which are 0.01-0.1µm. Analysis the silica dispersion in the rubber by using FESEM show us that silica have non-permanent structure which can form cluster and these may loosely bond as agglomerates.

Based on the result obtained 125% for Batch 1 (control) was less resistance to oil due to unsaturated polymer which is degrades in oil. Oil resistance was far worse when the percentage of silica content was 6%, 141.7% for Batch 2 and 120.4% for Batch 4 as compared to 111.6% for Batch 3 and 92.1% for Batch 5 which had 12% of silica contents. Therefore, the good physical property in oil resistance was established in Batch 3 and Batch 5 will give huge contribution in industrial and automotive industry.

5.2. Recommendation

In the nutshell, for further study and research silica sand must have nano size of particles of 0.01-0.1µm in order to have good uniform distribution and reinforcing the properties of rubber compound. In UTP, we only manage to get until less than 45 µm which is not small enough in order to have uniform NR latex-filler interaction.

The further studies can be improved by adding more variation of silica loading to check at which silica percentage give biggest changes in improving the properties. Thermal testing for further understanding on rubber composite behaviour may be useful in many industrial and automotive applications.

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