Making Composites from Mixing Limestone with Addition of Latex

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Abstract: Research on mixing of limestone with the addition of latex in making composites have been done. This study uses limestone originating from Sidikalang where crushed limestone using ball mill then calcined at 900°C for 2 hours then tested using XRD Pan Analytical X'pert Powder PW 30/40 to determine the presence of CaO compounds with the greatest intensity of 3036 at θ=37.3556 and particle size tested using PSA Shimadzu SALD-2300 is obtained an average of 731.7 nm. Then limestone powder (LSP) is used as a filler with various variations on latex composites. Analyzing of the morphology using SEM FEI Inspect-S50 can be seen from the photo that limestone powder can be evenly distributed on compsites 800 g of latex + 200 g of limestone powder while in the next variation the limestone powder is not evenly distributed because the addition of the filler increases but the addition of the matrix decreases. The mechanical properties of latex composites with limestone powder were tested using ASTM D-412 where optimum tensile strength was found in 800 g of latex + 200 g of limestone powder is 1.279 MPa and optimum elongation of 1000 g latex is 4.328 mm/mm. Thermal properties were tested using TGA ASTM E1131 obtained by decomposition thermal at 300-400°C because in the sample 1000 g of latex has thermal stability at 377.39°C and in the sample 800 g of latex + 200 g of limestone powder has thermal stability at 378.21°C.

1 INTRODUCTION

Natural latex is a substance obtained from rubber latex (Havea Brasiliensis). Natural latex is composed of hydrocarbons and contains small amounts of non-rubber parts, such as fat, glycolipid, phosphorus, proteins, and other organic materials (Kohjiya et al., 2014).

Natural rubber is one of the important agricultural products because it holds a role in improving human living standards and increasing foreign exchange. The consumption of natural rubber and world synthetic rubber in 2004 only reached 20.03 million tons, among them 11.5 million tons were natural rubber. However, as the largest landowner in fact processing latex into finished or processed goods, Indonesia is only able to control the market 10% which is still far below Thailand with land that is narrower than Indonesia. Where Thailand was able to dominate the market by 29% and Malaysia by 59%. One of the factors that caused this weakness was the high production costs in processing rubber finished goods (Sinaga, 2015). This is what encourages researchers to conduct research in suppressing production costs in processing latex by carrying out special treatment for latex raw materials namely by adding fillers.

Indonesia as a country that is rich in natural products has abundance in various sectors. One of the natural resources produced is limestone with a large $CaCO_3$ content (Lukman et al., 2012).

Most forms of calcium in limestone are found as calcium carbonate ($CaCO_3$). Limestone has a density of 2.6-2.8 g/cm^3 and is pure in the form of calcite crystals consisting of $CaCO_3$. $CaCO_3$ content in limestone reaches more than 90% and the rest are other substances (Oates, 1998).

The potential for limestone production in Indonesia is very large and almost evenly distributed throughout Indonesia, mainly used as industrial excavation (Shubri et al., 2014). In general, the amount of limestone in Indonesia reached 28.678 billion tons. Statistics show that the industrial sector for the use of limestone tends to increase at 10.45% every year (Rumengan et al., 2017). The distribution of limestone deposits are almost evenly distributed throughout Indonesia, but the largest deposits are in West Sumatra (Mukarrom, 2017).

(Keliat, 2015) Has investigated the role of limestone addition to the mechanical properties and

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thermal resistance of HDPE-g-MA composites where the results of the mechanical properties of HDPE-g-MA composites showed that the optimum tensile strength was 15.51 MPa and optimum elongation was 62% while the thermal properties using TGA on HDPE-g-MA + limestone composites have a decomposition point by 498,42^{o}C.

Saputra (2016) has investigated the research on making composites using fillers namely black carbon and matrix namely natural rubber which aims to determine the tensile test properties of composites where the addition of black carbon at 20, 25 and 30% results in an average of the highest tensile strength occurred in the natural black carbon composites of 20% at 1.18 MPa, at 25% at 0.91 MPa, at 30% at 0.56 MPa while at the addition of black carbon 20, 25 and 30% yielded the average value of natural rubber composite strain 20% black carbon for 42.53%, at 25% at 25.19%, at 30% at 6.69%, while the addition of black carbon 20%, 25% and 30% yielded the highest average modulus of elasticity occurs in natural rubber composites-black carbon at 20% at 2.21 x 10^{-2} MPa, at 25% at 2.16 x 10^{-2} MPa, at 30% at 1.91 x 10^{-2} MPa.

Sinaga (2015) has investigated research on the making and characterization of composite matrix polymers where filler namely silica rice husk and matrix, which is concentrated latex where aims to determine the nature of mechanical tests where the optimal composition for modulus of elasticity for making latex-silica based composites is 0.221 MPa. In this study we will develop composites by mixing limestone as filler and latex as a matrix. Where in limestone generally there is calcium carbonate ($CaCO_3$). The choice of limestone as a filler in the manufacture of composites is based on the properties of limestone which can improve the mechanical properties of rubber and the amount is very abundant in Indonesia so can reduce the emphasis on processing rubber goods. Where the character of limestone is plastic, it can harden quickly so that it gives the strength of the binder, easy to do, produces a good bond for plastering. Therefore, the researchers hope that this research will be able to reduce production costs in processing latex and add value to limestone. In addition, composites can be produced with better durability.

2 MATERIALS AND METHODS

2.1 Materials

The tools used in this study include: ball mill, 170 mesh sieve, analytical balance, mechanical mixer, x-ray diffraction, particle size analyzer, oven, furnace stove, thermogravimetric analysis, scanning electron microscopy, tensile test tools, glass tools, two roll mill, limestone, latex, aquadest.

2.2 Procedure

2.2.1 Sample Preparation

2000 g of limestone is taken from the mountains of Sidikalang, Dairi, North Sumatra and then cleaned using aquadest and then dried in the oven for 6 hours at 110□.

2.2.2 Making Limestone Powder using Ball Mill

Limestone and seven metal balls (balls change) are inserted into the grinding jar. Then put into grinding station. Close the engine cover, then set the rotation speed to 250 rpm and set the playback time to 60 minutes and press the start button. Next, limestone powder was obtained and then filtered using a 170 mesh sieve. Then the limestone powder that has escaped from the sieve is weighed as much as 100 grams, then calcined 100 grams of limestone powder from the sieve at 900□ for 2 hours. Furthermore, it was characterized using x-ray diffraction or X-Ray Diffraction (XRD).

2.2.3 XRD Characterization of Limestone Powder

Determine the structure of limestone powder material was carried out using x-ray diffraction brand Pan Analytical X'Pert Powder PW 30/40. 2 g of ball mill limestone powder is put into a 2x2 cm2 holder. Then the holder containing the sample is connected to the diffractometer. Set the sample name, initial angle, final angle and speed of analysis on the computer and press the start button. From the XRD data it can be seen the composition of compounds from limestone powder.

2.2.4 Particle Size Analyzer

Determine the particle size of limestone powder was carried out using particle size analyzer brand Shimadzu SALD-2300. ± 1 ml of limestone powder solution is inserted into the PSA to test the particle size characteristics of the solution. The working principle of this PSA is to use a laser beam diffraction method that is fired on the liquid sample being tested, the particles in the sample solution undergo a movement called Brownian motion. The light source (laser) used in the PSA in this study uses the principle of Dynamic Light Scattering (DLS). Measuring with this PSA is done at room temperature. This temperature affects the movement of particles in a solution (Brownian motion) during measurement by a device. The higher the temperature, the more active the motion of the particles, this affects the accuracy of the measurement results. Furthermore, the results of the average particle size of limestone powder can be determined based on the graph obtained.

2.3 Preparation of Composites

200 g of calcined limestone powder at 900□ for 2 hours mixed with 800 g of latex using a mechanical mixer with a speed of 1000 rpm for 15 minutes. Then the same procedure was carried out for mixing 750 g of latex and 250 g of limestone powder; 700 g of latex and 300 g of limestone powder; 650 g of latex and 350 g of limestone powder; 600 g of latex and 400 g of limestone powder; 1000 g of latex, then milled using two roll mill and dried for 1 week. Composites obtained were ground back using a two roll mill. Furthermore, it was characterized by tensile test, TGA analysis, SEM analysis.

2.3.1 Test of Tensile and Extensibility

Tensile strength and elongation testing were carried out by tensile testing instruments brand Cometech Qc 502M1 on each specimen by means of dumbbell and specimen sizes based on ASTM D-412. Turn on the Torsee's Electronic System tool. Left for 1 hour. Clamped the sample using giff. Set voltage, strain, and unit. Turn on recorder (ON). Installed note ink. Set the x axis (strain) and y axis (voltage) and set the unit. Installed sample. Press the start button. Rated load and stroke values. Judging by the numbers in load (stress) and stroke (strain), if the sample has broken up. Note the load and stroke values of the sample.

2.3.2 Analysis of Thermal Properties with Thermogravimetric Analysis

Analysis of thermal properties was carried out using the thermogravimetric analysis brand ASTM E1131. Weighed a sample 10 mg and then put it into an aluminum cell, then press it. The cell that has been pressed is placed in a position adjacent to the reference cell, where after the device is in equilibrium, then the analysis device is operated with a temperature of 30^{o}C to 600^{o}C with a speed of heating increase of 10^{o}C / minute and the gas used is nitrogen. The results obtained in the form of a graph of flow of heat flow to temperature and mass graphs that are lost to temperature.

2.3.3 Analysis of Morphological Properties by SEM

Analysis of morphological properties was carried out using the scanning electron microscopy brand FEI Inspect-S50. The microscopic observation process using SEM begins with glueing the sample with a stick made of older metal specimens. Then after the sample is cleaned with a blower, the sample is coated with gold and palladium with a dionspater machine pressurized 1492x10^{-2} atm. The sample is then put into a special room and then irradiated with 10 Kvolt electron beam so that the sample emits secondary electrons and bounced electrons which can be detected by scientor detectors which are then amplified by an electrical circuit which causes Chatode Ray Tube images. Shooting is done after selecting a particular part of the object (sample) and the desired magnification so that a good and clear photo is obtained.

3 RESULTS AND DISCUSSION

3.1 Characterization of Limestone Powder

The results of the characterization of limestone powder produce a diffractogram as shown in Figure 4.1 by matching the results of the limestone diffractogram with the diffractogram that contained JCPDS (Join Committee On Difraction) so that limestone constituents can be identified.

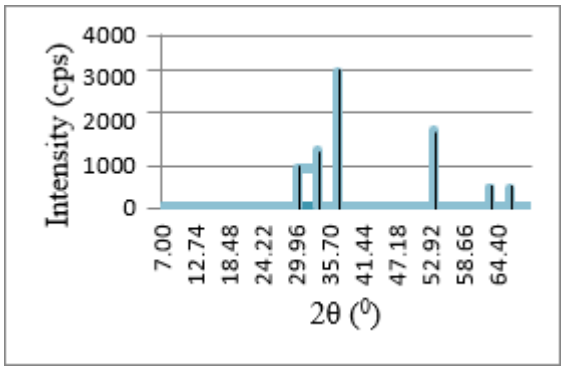

Figure 1: Diffractogram of limestone powder from the Sidikalang mountains, Dairi, North Sumatra

Identification results showed that limestone from the Sidikalang mountain range, Dairi, North Sumatra contained CaO minerals which were characterized by the presence of peaks at 2θ = 32.20280°, 37.35560° and 53.86320° (Table 4.1). Similar results were also reported by Husin (2013), CaO constituent minerals with peak characteristics at 2θ = 32.290°, 37.40° and 53.870°.

Table 1: Angle Value 2θ of CaO

Standard (JCPDS)	Sidikalang Mountain
32.29	32.20
37.40	37.35
53.87	53.86

The results of the characterization of limestone that has become limestone powder showed that the particles of limestone powder had an average particle size of 731.7 nm.

3.2 Composite Characterization

Composite characterization was carried out to determine the quality produced by mixing a polymeric material, where characterization was carried out, namely characterization of mechanical properties by tensile strength test, elongation and elastic modulus, characterization of thermal properties using TGA (Thermogravimetric Analysis) and characterization of morphological properties by using SEM (Scanning Electron Microscopy).

3.2.1 Tensile Test

Tensile strength testing is carried out to determine the tensile strength of the test object against the pull and the extent to which the material increases in length. This test uses the ASTM D-412 standard.

From the results of the tensile strength measurements of latex composite specimens with the use of limestone powder as fillers showed a better value than without using fillers. The highest tensile strength measurements were 1.279 MPa (800 g latex + 200 g limestone powder) and the lowest tensile strength was 0.272 MPa (1000 g latex). For full results can be seen in Table 2 below.

For the measurement of tensile strength obtained that the value of tensile strength increases lower when the addition of filler increases. This is because if the number of fillers increases and the number of matrices decreases, the spread of fillers will be uneven in the latex compound, resulting in agglomeration of fillers which decreases the effectiveness of the tensile force between the filler particles and the matrix which decreases the tensile strength 2012; Fang et al., 2014).

Table 2: Testing Results of Mechanical Properties of Tensile Test of Latex Composite Specimens

Sample (Ltx : LSP)	Wide (mm)	Thick (mm)	Stress (MPa)
10:0	14	4.9	0.272
8:2	10.5	3	1.279
7:3	10.2	3.2	1.109
6:4	10.7	3.4	0.910
6.5:3.5	10.4	3.1	0.814
6:4	10.6	3.3	0.718

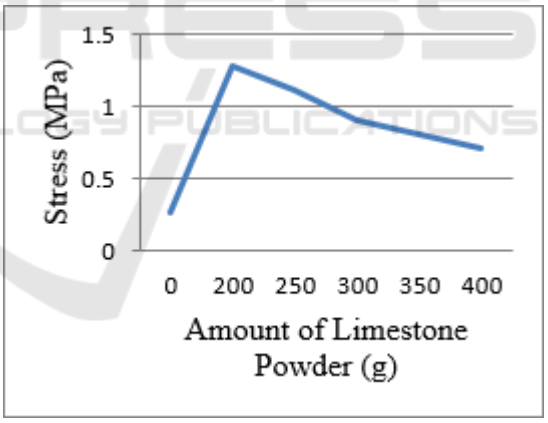

Figure 2: Graph of Stress vs Amount of Limestone Powder

3.2.2 Extensibility

For the measurement of strain (strain or elongation) the highest was 4.328 mm / mm (1000 g latex) and the lowest elongation was 2.915 mm / mm (600 g latex + 400 limestone powder). For full results, see Table 3 below.

Table 3: Testing Results of Mechanical Properties of Strain of Latex Composite Specimens

Sample (Ltx : LSP)	Gauge (mm)	Δ l(mm)	Strain (mm/mm)
10:0	69	298.64	4.32
8:2	64	242.30	3.78
7:3	71	253.21	3.56
6:4	63	215.76	3.42
6.5:3.5	54	169.22	3.13
6:4	47	137.02	2.92

For the measurement of elongation, it is obtained that the value of elongation increases when the addition of filler increases. This is because rubber has elastic properties but because of the increasing number of fillers and the addition of fewer matrices, the elastic properties of the rubber (as a matrix) decrease. In addition, increasing increments of fillers tend to form agglomerations which cause composite mixtures to become brittle so that they break more easily which causes the value of elongation to decrease (Veronika et al., 2013).

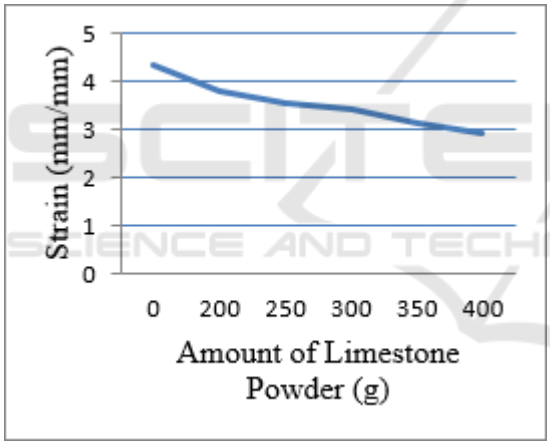

Figure 3: Graph of Strain vs Amount of Limestone Powder

3.2.3 Modulus of Elasticity

The highest young modulus measurement is 0.337 MPa (800 g latex + 200 g limestone powder) while the lowest modulus is 0.063 MPa (1000 g latex). For full results can be seen in Table 4 below.

Table 4: Testing Results of Young Modulus of Latex Composite Specimens

Sample (Ltx : LSP)	Stress (MPa)	Strain (mm/mm)	MoE (MPa)
10:0	0.272	4.328	0.063
8:2	1.279	3.785	0.337
7:3	1.109	3.566	0.311
6:4	0.910	3.424	0.265
6.5:3.5	0.814	3.133	0.259
6:4	0.718	2.915	0.246

For the measurement of young modulus it was found that the value of young modulus decreases when the addition of filler increases. If more and more fillers were added there will be an uneven distribution of latex compounds, thereby reducing the value of young modulus (Veronika et al.,2013).

Tensile strength indicates the maximum force required to decide on the lime powder latex composite. Observation results of tensile strength are expressed in the form of stress curves, namely the ratio of loads to cross-sectional area = F / A, to the extension of the material (strain / strain), namely the length increase divided by the initial length of material, expressed in stress-strain curves.

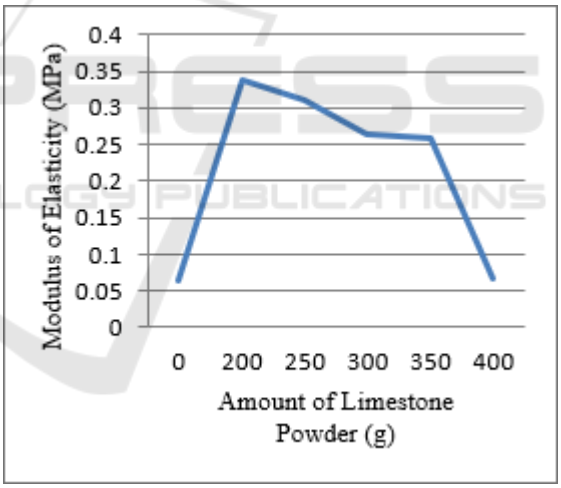

Figure 4: Graph of Modulus of Elasticity vs Amount of Limestone Powder

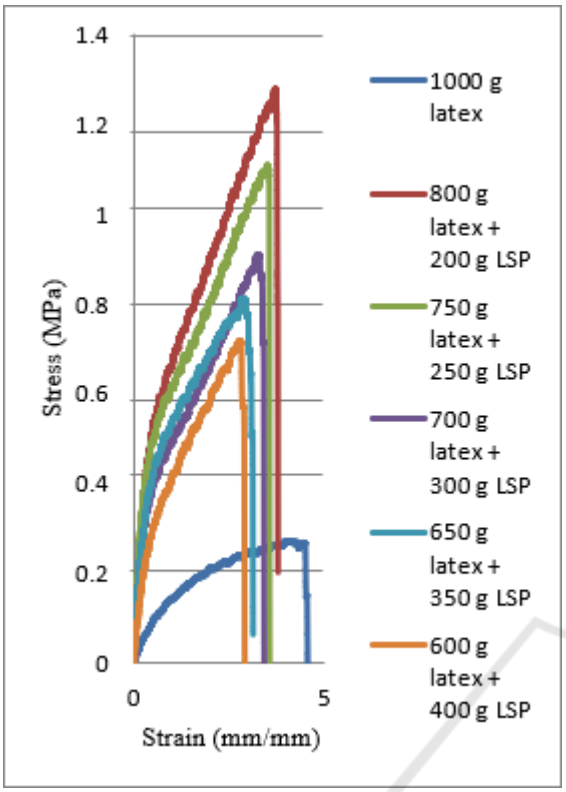

Figure 5: Curve of stress *vs* strain

From the graph above, it can be seen that the highest stress is shown in 800 g of latex + 200 g of limestone powder which is 1.279 MPa because in subsequent variations the addition of filler increases and the number of matrices decreases, spread of filler will be uneven in the latex compound resulting in agglomeration of fillers which decreases the effectiveness of tensile forces between particles of fillers and matrices which then decreases tensile strength (Nuraya et al., 2012; Fang et al., 2014) while the highest strain is shown in 1000 g of latex which is 4.328 mm / mm because rubber has properties the elastic but in the next variation the increase in filler increases but the addition of the matrix decreases, causing the elastic properties of rubber (as a matrix) to decrease and the increase in filler tends to form agglomeration which causes the composite mixture to become brittle so that it breaks easily causing a value elongation s decreases (Veronika et al., 2013).

3.2.4 Morphological Analysis of SEM

Characterization using SEM was carried out to look at the morphology of latex composites with fillers of limestone powder, where the morphological results that appeared could show which combination of fillers and matrices in this case to see the dispersion (distribution) of filler particles into the polymer matrix. The results of particle board morphology analysis can be shown in Figure 6 and Figure 7 as below.

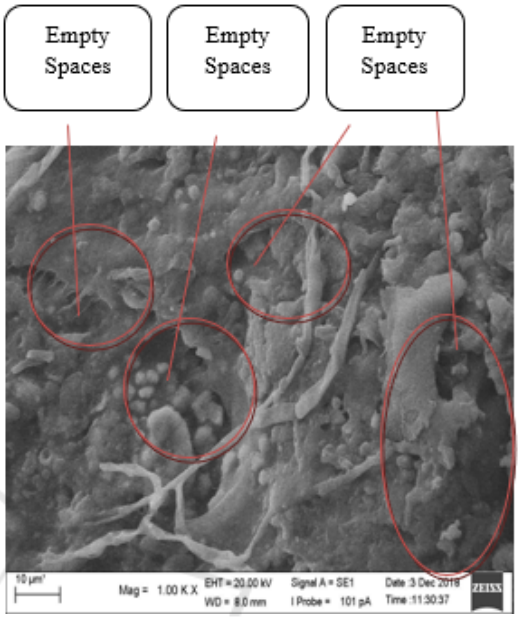

Figure 6: SEM photos of latex

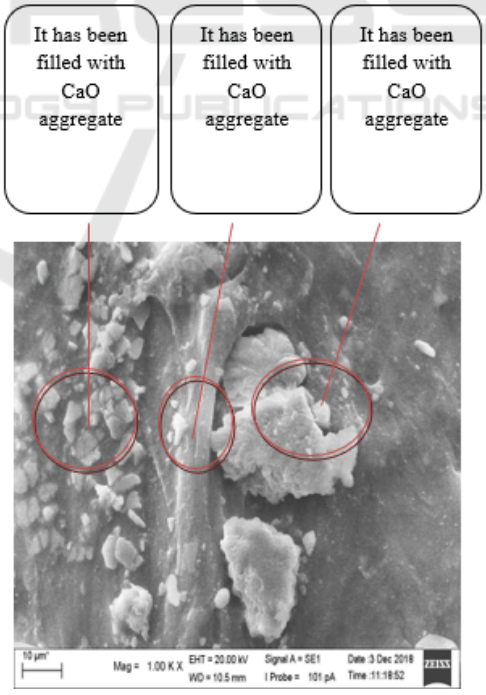

Figure 7: SEM photo of Latex/LSP (8:2)

In Figure 6, there are blank spaces in the latex matrix while in Figure 7 it appears that the empty

spaces have been filled with CaO aggregates and distributed almost evenly to the latex matrix. This is due to the small size of the particles from the filler which allows limestone to be able to combine with latex. The more amount of CaO added in the latex compound, the rubber matrix will be increasingly filled by the dispersion of the filler material so that it will cause agglomeration of latex and decrease mechanical properties (Dewi et al., 2014).

3.2.5 Characterization of Thermal Properties

The characterization of thermal properties in latex composites with limestone powder fillers using Thermogravimetry Analysis (TGA) is a technique to measure changes in thermal transition or material heat to the function of temperature or time in a controlled atmosphere.

Thermogravimetry Analysis (TGA) is a test performed on a sample to determine changes in weight (loss) due to changes in temperature. Analysis provides information on the point at which the lost mass is seen most clearly with respect to temperature changes so that the resulting data can be used to predict thermal stability.

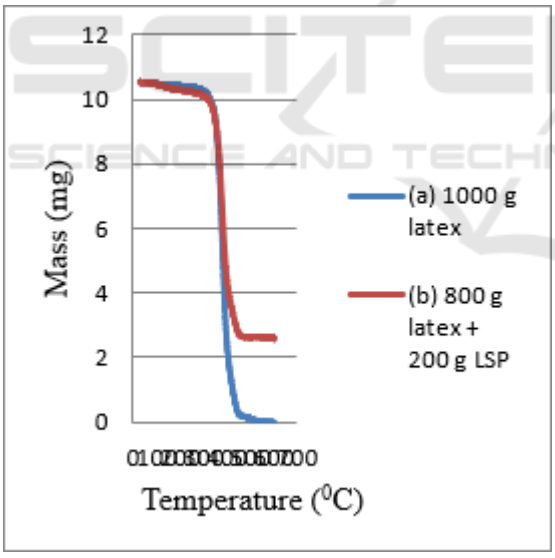

Figure 8:TGA curve (a) Latex and (b) Latex/LSP (8:2)

On the curve shown in Figure 4.8 it can be seen that the sample of 1000 g of latex has thermal stability at 377.39ºC while a sample of 800 g of latex + 200 g of limestone powder has thermal stability at 378.210ºC. This shows that the composite experienced thermal decomposition at a temperature of 300-400ºC. Because after passing the 400ºC temperature both curves are not at the same point (not coincide again) and separated into 2 curves.

In Figure 4.8 it can be seen that in a sample of 800 g of latex + 200 g of limestone powder which is at a temperature of 100ºC mass is obtained 10.4727 mg; at a temperature of 200ºC a mass of 10.2941 mg is obtained; at a temperature of 300ºC mass is obtained 10.1138 mg; at a temperature of 4000 0C a mass of 4.30106 mg was obtained; at a temperature of 500ºC a mass of 2.60660 mg is obtained; at 600ºC a mass of 2.56472 mg was obtained. Whereas the sample of 1000 g of latex at a temperature of 100ºC obtained a mass of 10.4583 mg; at a temperature of 200ºC a mass of 10.3953 mg is obtained; at a temperature of 300ºC a mass of 10.2337 mg was obtained; at a temperature of 400ºC a mass of 2.26365 mg is obtained; at a temperature of 500ºC the mass is 0.0882131 mg; at 600ºC the mass is 0.0300798 mg.

From the above data it can be concluded that in the sample of 1000 g of latex at a temperature of 600ºC the mass of 0.0300798 mg was obtained while in the sample 800 g of latex + 200 g of lime powder at a temperature of 600ºC a mass of 2,56472 mg was obtained. The mass in the sample of 800 g of latex + 200 g of limestone powder was greater than in the sample of 1000 g of latex. This is because limestone generally has a melting temperature of 900-1000ºC so it cannot decompose thoroughly and produce a greater amount of mass (Keliat, 2015).

4 CONCLUSIONS

The morphological results of SEM on a mixture of 1000 g latex show that there are empty spaces in the latex matrix whereas in a mixture of 800 g latex + 200 g limestone powder it appears that the empty spaces have been filled with CaO aggregates and are evenly distributed on the latex matrix.

The results of the characterization of the mechanical properties of latex composites found that the optimum tensile strength was found in a composite of 800 g of latex + 200 g of limestone powder of 1.279 MPa, optimum elongation was in 1000 g of latex of 4.328 mm / mm and optimum young modulus was in composite 800 g latex + 200 g limestone powder of 0.337 MPa.

The results of the characterization of thermal properties with TGA on latex composites is that the composite decomposes at 300-400ºC because in the sample 1000 g of latex has thermal stability at 377.390ºC and in the sample 800 g of latex + 200 g

of limestone powder has thermal stability at temperature of 378,210ºC.

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