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To cite this article: T Rihayat *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **788** 012046

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Making Polyurethanes from castor oil with addition of Bentonite and Chitosan as coating paints on eco-friendly medical device applications

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Abstract. Polyurethane-based vegetable oil coatings have been used in the past few decades considering the use of petrochemical-based raw materials is a non-renewable material. Vegetable oils used such as soybean oil, palm oil, and castor oil. They have lower environmental impacts, easy availability and biodegradation. In this study, polyurethane synthesis was carried out using the prepolymer method using the reaction of TDI with polyols based on castor oil. To provide anti-microbial properties of polyurethane, a composite method of polyurethane with chitosan was carried out. Whereas to provide heat resistance properties in polyurethane bentonite is added to polyurethane. Polyurethane/bentonite/nanocomposite chitosan was analyzed using the Fourier Transform Infrared Spectra (FTIR) to determine the microstructure of chemical compounds, Thermal Gravimetric Analysis (TGA) for viewing polyurethane/bentonite/chitosan heat resistance, and Scanning Electron Microscope (SEM) to see the morphology of polyurethane/bentonite/chitosan. FTIR analysis have shown the formation of hydroxyl groups in the compound epoxide castor oil, the reaction lasts for 2.5 hours at 50 °C as evidenced by the absorption of OH wave numbers which widens at 3500 cm⁻¹, the hydroxy group formed is the hydroxy group on C atoms secondary, and bentonite NH 3450 cm⁻¹, chitosan cluster C = O urethane widened at 1772 cm⁻¹. TGA analysis have pure polyurethane begins to decrease in mass at 246 °C, while polyurethane with the addition of filler decreases mass at 342 °C.

Keywords. Polyurethane, Chitosan; Bentonite; Biomedical; Coating

1. Introduction

The consumption of polyurethane polymer materials increases every year. Especially in the field of medicine, polyurethane is used as a face shield, blood bag, and infusion hose. The main medical device used in the medical field is a stethoscope. However, in this case, some problems pose obstacles to health, namely the use of a stethoscope not only for one patient but many patients who use it resulting in the



increasingly easy transmission of bacteria. Therefore, it is necessary to modify polyurethane in anti-microbial medical devices.

Polyurethane is produced from the reaction between polyols and isocyanates. In general, polyurethanes are made from petroleum-based polyols. However, the current oil condition is running low. Environmental problems and risks are a major concern as a reduction in conventional petroleum reserves. Therefore, it is necessary to transfer the raw materials for making polyols, namely vegetable oils such as soybean oil, sunflower oil, canola oil, flaxseed oil and castor oil. Utilization of vegetable oils is currently under the spotlight of the chemical industry, because they are one of the most important renewable chemicals platforms because of their universal availability, biodegradability, low prices, and exceptional environmental reductions (ie, low environmental toxicity and low toxicity to humans) Now natural ingredients are taking advantage of research and development, with polymer / composite vegetable oils being used in a variety of applications including coatings, adhesives, and biomedicine [1]. Polyurethane is a polymeric material that has a characteristic feature of the urethane (-NHCOO-) functional group in the polymer main chain. The urethane function group is produced from a reaction between a compound containing a hydroxyl (-OH) group commonly called a polyol with a compound containing an isocyanate group (-NCO-). Polyurethanes are generally synthesized from vegetable oils which are converted into derivatives, for example, alkyd resins and based on alkyd polyols. Polyols are then reacted with different diisocyanates to obtain polyurethane coatings [2].

Polyurethane (PU) is a block copolymer containing hard and soft segments where semicrystalline hard segments are formed by the reaction of polyisocyanates and extender chains (diol/diisocyanate) and soft amorphous segments consisting of long polyol chains [3]. Polyurethane is a copolymer block formed from a polyaddition reaction between or polyisocyanates (toluene 2,4-diisocyanate [TDI], diphenylmethane diisocyanate, naphthylene 1,5-diisocyanate, hexamethylene diisocyanate, isophoreon diisocyanate) and (di- or polyhydric) alcohol or other compounds containing active hydrogen atoms, as raw materials for polyurethane. Isocyanates can react with hydroxy groups such as alcohol to form urethane, the mechanism of the reaction of isocyanates with hydroxyl assemblages of alcohol compounds is determined by the reactivity of various types of hydroxyl assemblies. Diol amides and polyols from vegetable oils have been used as the main ingredients for synthesizing polyurethane along with aliphatic and aromatic isocyanates [4]. The longer the reaction time for making polyurethane, the more bonds are formed, so the higher the temperature needed to break the bonds, in other words, the mechanical properties are also increasing [5].

Polyurethane (PU) coatings have excellent resistance in terms of toughness, low temperature, flexibility, corrosion resistance and chemical resistance, so they are used in various applications such as the maintenance industry for chemical resistance [6] and biomedical devices.

Previous study said that used of *Jatropha* oil had hydroxyl polyol numbers obtained through methylation giving the greatest value, which was 87.91 mg KOH / g at a ratio of 1: 9 to methanol to isopropanol and 1 hour reaction time while at for 2 hours the hydroxyl group obtained 157.69 mg KOH / g which can be used to make semi-flexible polyurethanes. The length of reaction time affects the increase in hydroxyl number, which is the longer the reaction time, the greater the hydroxyl number obtained.

In this study using castor oil-based polyols containing ricinoleic acid which represented 90% of the total fatty acid content [7] by the method of methoxylation and longer reaction time. To improve the mechanical properties of polyurethane products as coating paint in medical devices used fillers such as bentonite and chitosan. Chitosan has various applications in the biomedical field such as wound healing, antibacterial coatings, tissue engineering scaffolding, separation membranes, stent coatings, sensors and drug delivery systems [8].

Chitosan has the potential for a wide range of applications in biocompatibility (non-toxic), biodegradability, antimicrobial properties, and a friendly natural environment to provide good opportunities for future progress. One of the main disadvantages of coating vegetable oil-based polymers is that they are unable to withstand high temperatures [9]. Therefore, currently, the development of science about nanotechnology not only produces anti-bacterial material but also can withstand heat. So

in this study utilizing the use of nanocomposite bentonite as a filler material in polyurethane matrices as heat-resistant reinforcement and increased mechanical properties of coating materials [10].

This study will make castor oil-based polyurethane as a paint and coating material. To improve the physical and mechanical properties of polyurethanes such as having strength, heat resistance, corrosion resistance and chemicals as well as having resistance to bacteria, modifications were made with the addition of bentonite and chitosan with varying concentrations.

Currently, there are many uses of chitosan for the anti-microbial properties of materials because chitosan has very beneficial properties, namely biocompatible, biodegradable, non-toxic, and inexpensive. Also, chitosan acts as an antimicrobial agent in polyurethane which can prevent the development of harmful bacteria such as *Staphylococcus Aureus* [11]. Various studies and developments to provide anti-microbial properties of polyurethanes have been carried out by several researchers with various raw materials used to obtain polyurethanes including, toluene diisocyanate and polyol [11], hexamethylene diisocyanate and polyol [12]. While to provide anti-microbial properties in chitosan polyurethane materials have been used [13]. ZnO nanoparticles [14], and heparin as anti-adhesion [15]. From the results of research conducted by previous researchers that chitosan can reduce the number of bacterial colonies, bacterial adhesion is significantly reduced and increases the anti-bacterial properties of polyurethane materials [16].

While the largest percentage of inhibition of growth of gram-negative bacteria *E. coli* and gram-positive was obtained at 2.0% weight PU / ZnO NP concentration [17.]. For the results of subsequent studies with modification of chitosan and heparin on polyurethane showed a high decrease in bacterial adhesion and can kill all bacteria in the sample after 24 hours of inhibition of bacterial metabolism in polyurethane [17]. However, previous studies did not explain the optimal amount of chitosan use to obtain maximum anti-microbial properties, so in this study the addition of anti-microbial properties in the paint layer with variations in the concentration of chitosan. Besides, at present, the development of science about nanotechnology not only produces anti-bacterial material but can also withstand heat. So in this study utilizing the use of nanocomposite bentonite as a filler in the polyurethane matrix as a heat-retaining amplifier and improving the mechanical properties of coating materials [18].

2. Methods

2.1. Materials

Bentonite from Nisam, North Aceh with a size of 100 mesh, aquadest, aquabidest, Cetyl Trimethyl Ammonium Bromide (CTAB), 2% acetic acid and 100% glacial, chitosan, formic acid, glacial H₂SO₄ 96%, methanol, AgNO₃ Solution and Sodium Hexamethaphosphate (NaPO₃)₆, molecular sieve, nutrient agar and steel plate.

2.2. Polyol synthesis

Entered 30 ml of 30% H₂O₂ into the reactor then added 100% CH₃COOH as much as 50 ml and concentrated H₂SO₄ as much as 2 ml. stirred with a speed of 200 rpm and a temperature of 40°C for 1 hour, added 100 ml of castor oil and stirred at a speed of 200 rpm for 3 hours. After cold, then add 100 ml of methanol, 50 ml of glycerin, concentrated H₂SO₄ catalyst 2 ml and 5 ml of water and heated to a temperature of 40°C. The oil produced is cooled to room temperature and separated by a separating funnel to obtain polyol. Figure 1 shows the flow of polyol synthesis.

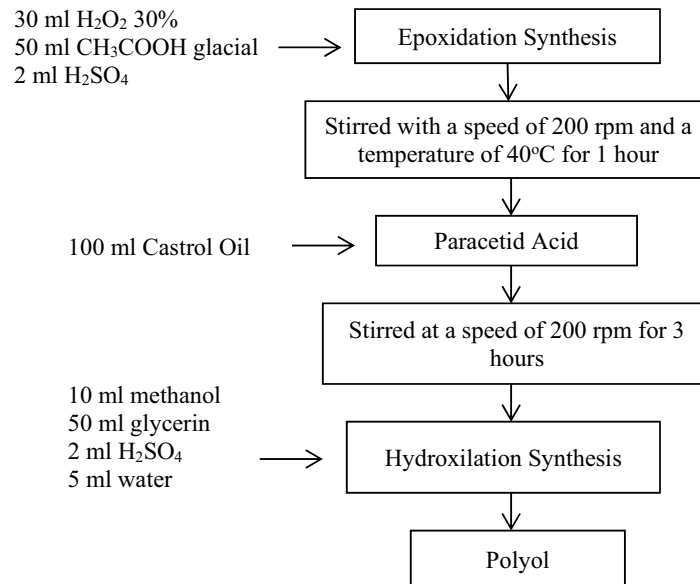


Figure 1. Flowchart of polyol synthesis.

2.3. Purification of bentonite

Order 18.2 grams of Cetyl Trimethyl Ammonium Bromide (CTAB) with 250 ml of distilled water into a 500 ml beaker glass. Then heated at 80 80C for 1 hour. Dissolved 20 grams of bentonite with 250 ml of distilled water into a 1000 ml glass beaker. Dispersion of bentonite solution was put into CTAB solution and stirred for 1 hour. Filtered, and bentonite washed with distilled water. It is dried at 60 °C. Figure 2 depicts the flow of purification of bentonite process.

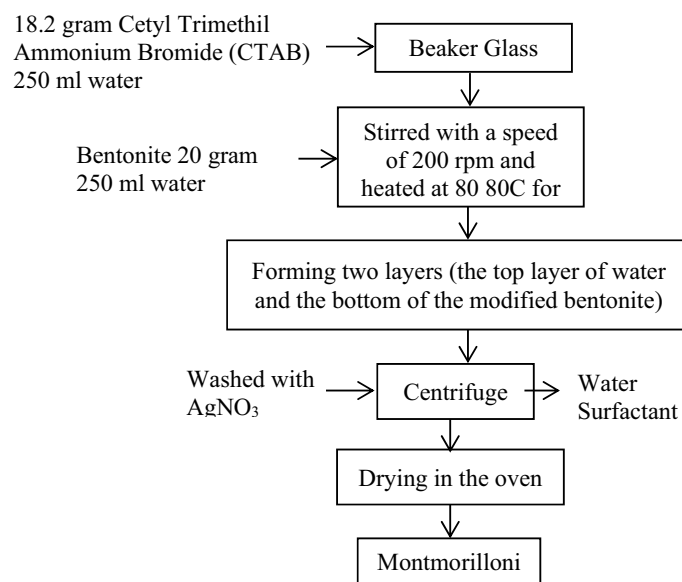


Figure 2. Flowchart of purification bentonite.

2.4. Chitosan processing

Dissolved 4.25 grams of chitosan into 2% acetic acid as much as 100 ml. Stirred at a speed of 500 rpm for 2 hours with a pH of 4.0 to obtain chitosan suspensions. Dropped 50 mL of 0.1 N NaOH slowly into the chitosan suspension. Rinse using 150 ml of distilled water or until neutral pH. Dried in an oven at a temperature of 60 °C. Figure 3 shows synthesis chitosan.

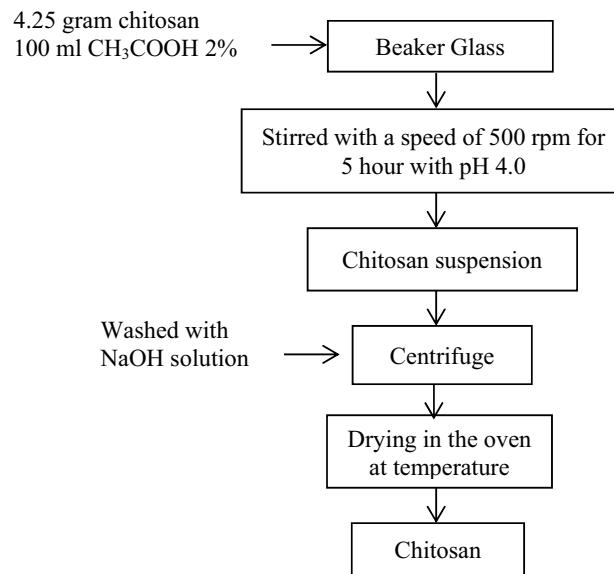


Figure 3. Flowchart of synthesis chitosan.

2.5. Polyurethane / Bentonite / Chitosan Paint Coating Synthesis

Mixed 4 ml of toluene into 16 grams of castor oil polyol. Montmorillonite and chitosan were added 2, 3, 4 per cent by weight (wt%) to beaker glass using a magnetic stirrer 600 rpm for 1 hour. The polyurethane produced is cooled at room temperature. Analysis of the chemical structure of polyurethane, bentonite, chitosan coatings using FTIR. Analysis of heat resistance of coating paint using TGA. Surface shape analysis using SEM. Bacterial susceptibility test with disc diffusion method. The overall process as illustrated in figure 4.

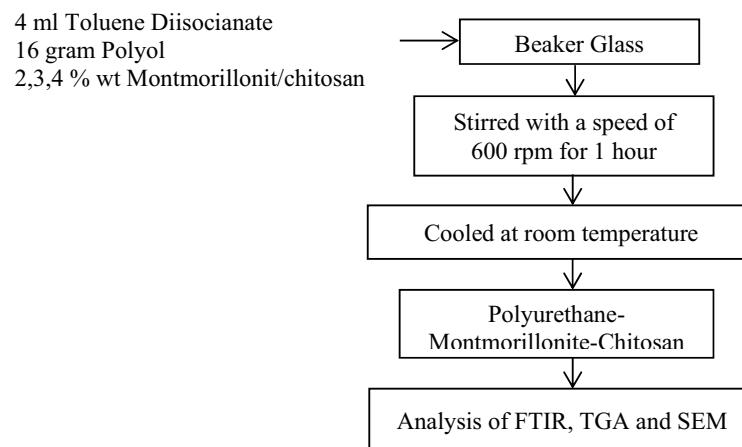


Figure 4. Flowchart of polyurethane / bentonite / chitosan paint coating synthesis.

2.6. Fourier Transform Infrared (FTIR) analysis

Infrared spectroscopy of nanocomposites obtained by pallet KBr using Shimadzu FTIR Spectrophotometer, to see the hydroxyl groups of the polyols produced. Spectra obtained in the mid infrared region (4000-400 cm^{-1}) at room temperature.

2.7. Thermogravimetric Analysis (TGA)

Thermogravimetry Analysis (TGA) is used to see the heat resistance of polyurethane paint with the addition of bentonite/chitosan. Thermal stability of the sample was determined by thermogravimetric analysis, using Perkin Elmer thermogravimetric analysis at a heating rate of $10^\circ\text{C} / \text{min}$ under a nitrogen atmosphere, from 50°C to 700°C . Approximately 2 mg of each sample was analyzed and consequently, the sample weight was determined.

2.8. Scanning Electron Microscope (SEM) analysis

An electron gun produces an electron beam and is obtained by an anode. The magnetic lens focuses electrons towards the sample. The focused electron beam scans the entire sample by being directed by the scanner coil. When electrons about the sample, the sample will issue a naew electron that will be received by the detector and sent to the monitor. SEM analysis to observe the surface of the object directly.

3. Result and Discussion

3.1. Polyol compounds from castor oil

Epoxide oil from castor oil is obtained from the reaction between peracetic acid and ricinoleic acid or castor oil, while peracetic acid is produced from the reaction between hydrogen peroxide and glacial acetic acid with the help of a concentrated sulfuric acid catalyst. Hydrogen peroxide is used to oxidize the double bond into an epoxide group in the epoxidation process. The results of the epoxy castor oil obtained have a lighter colour compared to the colour of oleic acid castor oil. Epoxide oil formed is an intermediate compound that can react further to form diol compounds, because it has two reactive places, namely carbonyl groups which can connect glycerides with fatty acids and epoxide groups. So that the process of forming a polyol will occur if the epoxide group reacts with alcohol. Epoxide compound formation reactions in vegetable oils have been carried from previous studies.

3.1.1. Fourier Transform Infrared (FTIR) analysis. X The FTIR test aims to look at the O-H group obtained from polyols produced through the synthesis of castor oil by adding other chemicals. The following are the results of the analysis produced.

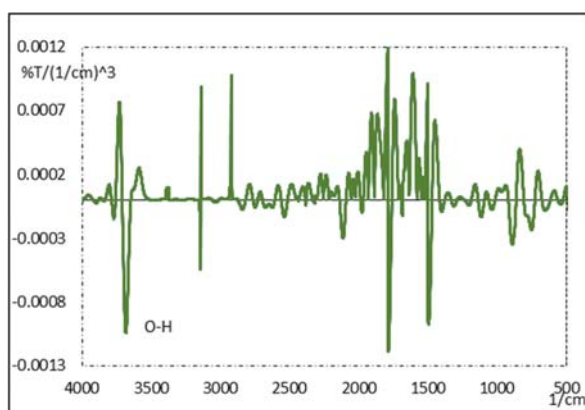


Figure 5. FTIR spectrum of ricinoleic acid polyol castor oil.

The results of FTIR analysis have shown in figure 5 where, the formation of hydroxyl groups in the compound epoxide castor oil, the reaction lasts for 2.5 hours at 50 °C as evidenced by the absorption of OH wave numbers which widens at 3500 cm⁻¹, the hydroxy group formed is the hydroxy group on C atoms secondary. The results of the hydroxyl group wavelength measurements in the previous study were 3412.38 cm⁻¹, 3461 cm⁻¹ and 3396 cm⁻¹.

3.2. *The characteristics of bentonite become montmorillonite*

Bentonite was taken in Teupin Reusep village, Muara Batu District, North Aceh district, bentonite was grey (figure 6) based on the results of chemical analysis, bentonite in Teupin Reusep village in North Aceh contained 51.6% montmorillonite.



Figure 6. North aceh bentonite.

Organoclay is a modification of bentonite with cetyl trimethyl ammonium bromide (CTAB) surfactant, which by adding surfactant can expand the distance of d-bentonite bentonite itself. The d-spacing layer is the area where polyurethane can enter into it when the composition is made. The matrix in this study is polyurethane and bentonite as fillers used to increase the temperature of polyurethane coating paint.

3.3. *Characteristics of polyurethane-montmorillonite-chitosan*

The manufacture of castor oil-based polyurethane coatings is to react the synthesized polyols with isocyanates. Isocyanates used toluene diisocyanate (TDI). The castor oil polyol produced is slightly yellowish in colour, after being reacted with TDI and applied to the specimen the material remains a yellowish colour.

3.3.1. Fourier Transform Infrared (FTIR) analysis. This FTIR test aims to see the urethane group of bentonite by adding surfactants to increase the interlayer of the bentonite and to become montmorillonite. Figure 7 shows the results of an analysis of surfactant modified bentonite (montmorillonite).

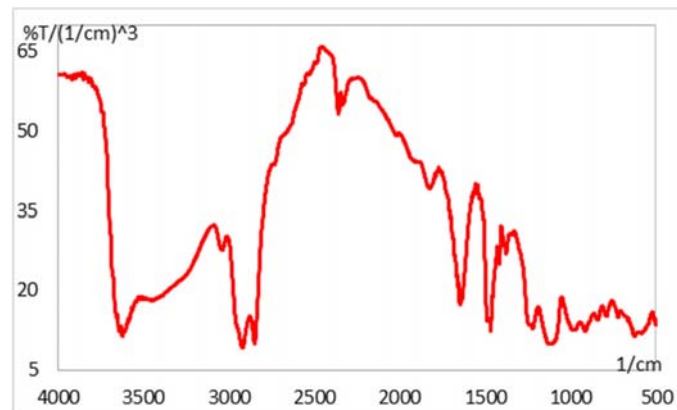


Figure 7. Polyurethane FTIR spectrum with Montmorillonite-Chitosan filler.

The results of FTIR analysis have shown the formation of NH urethane groups in castor oil polyurethane compounds, as evidenced by the absorption of NH wave numbers that is at wavelengths of 3450 cm^{-1} , whereas the results of the NH group wavelength measurements in the previous study were 3300 cm^{-1} , 3311 cm^{-1} , 3315 cm^{-1} , 3316 cm^{-1} . Cluster C = O urethane widened at 1772 cm^{-1} in a previous study of 1720 cm^{-1} (Kara et al, 2015), the CH group was an uptake of 2998 cm^{-1} and in the previous study 2924.10 cm^{-1} (Rohaeti Eli and Suyanta, 2011). From the results of the analysis show that the formation of polyurethane is seen from the presence of wavelength absorption in the functional groups N-H, C = O urethane, and C-H. The detected functional group represents the polyurethane functional group (-NHCOO-) in the form of wavelength absorption of separate functional group bonds.

3.3.2. Thermo Gravimetric Analysis (TGA). Thermal degradation testing using a TGA (Thermo Gravimetric Analysis) tool aims to qualitatively determine the thermal stability of polyurethane with the addition of fillers and compared to pure polyurethane without mixing. This test is based on changes in sample weight due to heating from room temperature to high temperatures usually hundreds of degrees Celsius so that the sample will experience a mass reduction (degraded) because it burns at a certain temperature. The process of mass loss occurs because of the decomposition process that is the termination of the chemical bond. The following is a figure 8 of the TGA test results.

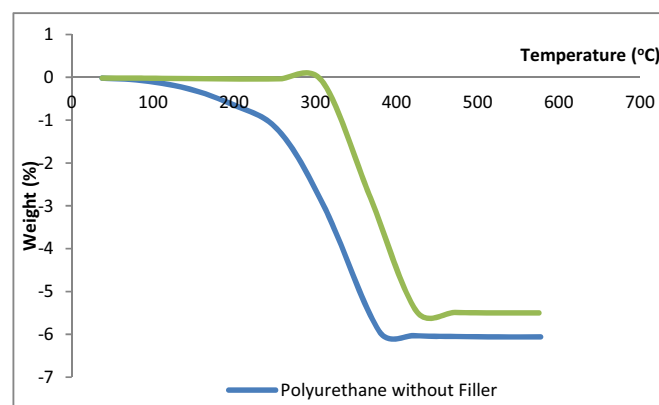


Figure 8. Thermogram Polyurethane without fillers and with the addition of fillers.

From figure 8 above, it is a plot of the decrease in mass on the y-axis and increases in temperature on the x-axis. This graph shows that in the nanocomposite polyurethane sample, there is a single

decomposition because onset and end set only occur once. Onset is the temperature at which the sample begins to be degraded thermally and the end set is the temperature at which the sample holds its mass from the combustion reaction. The degradation temperature of pure polyurethane and nanocomposite polyurethanes in this study ranged in the range 200-300°C. Based on graph 4, pure polyurethane begins to decrease in mass at 246 °C, while polyurethane with the addition of filler decreases mass at 342°C. These results indicate that bentonite in polyurethane polymers has succeeded in increasing thermal stability compared to pure polyurethane. The higher the filler concentration was added, the better the thermal stability of the material was marked by the increase in the degradation temperature.

3.3.3. Scanning Electron Microscope (SEM) analysis. Morphology of polyurethane nanocomposite with the addition of bentonite fillers which have been converted into montmorillonite and the addition of chitosan as an antibacterial agent from polyurethane paint produced. Figure 9 shows the SEM image of polyurethane-bentonite-chitosan coating plate.

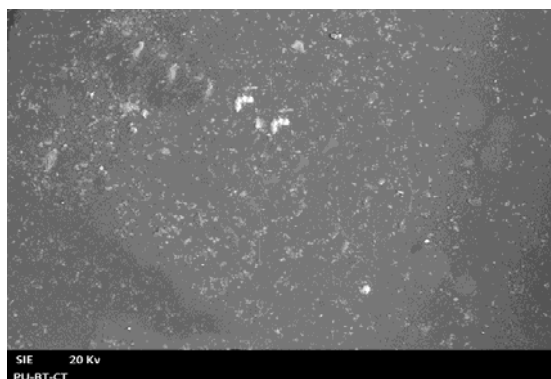


Figure 9. Morphology of polyurethane-bentonite-chitosan coating plate.

Surface results that experience white spots indicate bentonite-chitosan fillers dispersed into polyurethane paint, while darker-tended surfaces are iron plates which have been applied with polyurethane paint.

4. Conclusion

Polyurethane is made through castor oil polyols by reacting with TDI. Polyols are synthesized from castor oil through epoxidation and hydrodistillation reactions which can then be used as polyurethane coatings. From the results of FTIR analysis shows the formation of hydroxyl groups in epoxide castor oil compounds, the reaction lasts for 2.5 hours at 50°C, which widens at 3500 cm⁻¹, the hydroxy group formed is a hydroxy group on the secondary C atom, and NH 3450 bentonite cm⁻¹, chitosan cluster C=O urethane widens at 1772 cm⁻¹. TGA analysis has pure polyurethane starting to decrease mass at 246°C, while polyurethane with the addition of fillers decreases mass at 342°C.

Acknowledgement

The Author would like gratefully and acknowledgement thanks to Directorate of Student Affairs and Education Ministry of Research Technology and Higher Education of Indonesia and Politeknik Negeri Lhokseumawe.

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