

Investigation of Polyurethane Primer Coating Paint Based on Local Palm Oil with Antimicrobial and Anticorrosion Agent Formula Bentonite-Chitosan

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ABSTRACT

Paint is a product that is really needed to coat iron or steel materials such as industrial pipes, medical equipment, and so on. The sample formulation used was the addition of chitosan bentonite filler 2, 4, 6, 8% w/w. In TGA testing, the sample underwent single decomposition and showed the best results in the Polyurethane/Bentonite/Chitosan 8:8% w/w sample where the sample began to degrade at a temperature of 416.85 (°C). The bacterial test results showed the best results in the Polyurethane/Bentonite/Chitosan 8:8% w/w sample which had the widest inhibition zone with a value of 6.9 mm for E.Coli bacteria. The sample with the composition Polyurethane/Bentonite/Chitosan 8:8% w/w was the best sample where the sample experienced the smallest corrosion rate, namely 5.08 mpy. The application of this paint is to strengthen the durability of the building so that corrosion does not occur in the Hagu Barat Laut of Lhokseumawe because it is close to sea waters which have air levels that are quite susceptible to corrosion.

Keywords: Biodegradable, Paint, Polyurethane, Bentonite, Chitosan

INTRODUCTION

Aceh is one of the provinces which is currently engaged in several fields including medical, construction, food and large and small scale industry. Based on data from the Central Statistics Agency (BPS) for 2022, it shows that the human development index in the Aceh region continues to increase with a percentage increase of 5.09% points for the period 2010 to 2022. Increasing construction figures results in higher market demand for material development needs. Paint is a product that is really needed to coat iron or steel materials such as industrial pipes, medical equipment, and others. Polyurethane paint is a paint product that currently has great interest among the public.

The problem of paint products which currently has a large amount of interest in the community. Problems that are currently felt by the public and industry regarding paint products include paint peeling on the coated material. This is caused by hot/high temperatures. High temperatures can increase the kinetic energy of particles, causing corrosion of the material. Corrosion is a condition that is highly avoided, especially in the factory and medical industries because it can trigger damage to equipment and the emergence of bacterial activity that is harmful to health. Various renewable resources such as palm oil, soybeans, castor oil and sunflower oil are used as ingredients to produce polyurethane. made from vegetable oil[1]. Vegetable oil-based polyurethane resins have attracted attention in recent decades, due to their biocompatibility, ease of synthesis and use as coating materials.[2].

The properties of polyurethane show good resistance to ultraviolet light, weathering, good surface adhesion, high flexibility, but in some cases polyurethane shows inadequate corrosion resistance. This is because water and ions can penetrate the polyurethane layer. The degradation process occurs when the corrosive electrolyte encounters the polyurethane coating[3]. This causes corrosion to occur more quickly. To increase the corrosion resistance of polyurethane, bentonite and chitosan are added as fillers.

Research Urgency

Bentonite has become so popular in the polymer industry that previous research has shown that it can improve the mechanical and temperature properties of polyurethane[4]. Clay coated silicate can play an important role in terms of providing barrier and heat resistant properties to coating systems. The main content of bentonite is the mineral montmorillonite (80%) with the chemical formula $(Al_2O_3 \cdot 4SiO_2 \cdot X H_2O)$. It has color variations ranging from white to yellow, to olive green, deposited in various fresh waters and marine basins, characterized by moderate environmental and climatic conditions. The bentonite

sheet structure consists of 2 tetrahedral layers composed of the main element Silica (O and OH) flanking one octahedral layer composed of the element M (O and OH) (M= Al, Mg, Fe)[5].

The addition of chitosan as an alloy in modifying polyurethane can provide anti-bacterial properties to polyurethane[6]. According to research results which show that intercalation of chitosan through a cation exchange process can increase its antimicrobial activity. Chitosan is a multifunctional chemical in the form of fibers and is a copolymer in the form of thin sheets, pink or yellow in color, odorless[7]. Chitosan is a poly - (2-amino-2-deoxy- β -(1-4)-D-glucopyranose) with the molecular formula (C₆H₁₁NO₄) which can be obtained from complete or partial deacetylation of chitin[8]. Based on previous research, it shows that the addition of montmorillonite (MMT) can increase the adhesion of polyurethane paint coating applications and also increase the gloss index of the paint layer surface.[9]. Synthesis of polyurethane with the addition of bentonite filler, chitosan as heat resistant properties in the coating material obtained a temperature of 500°C while with the addition of B:K 2.5:2.5% w/w it was obtained at a temperature of 580°C[10].

In this research, polyurethane will be made using oleic acid based on palm oil with the addition of bentonite-chitosan filler in order to increase its heat-resistant and anti-bacterial properties. Oleic acid in palm oil will be used as a source of polyol to produce polyurethane. The characteristics carried out are assessed in terms of functional group analysis, bacterial activity analysis, and heat resistance analysis and corrosion tests.

Research Objectives, Benefits And Limitations

This research was carried out to reduce the problems caused by corrosion problems, so that renewable resource-based coating paint is produced which has anti-bacterial and anti-corrosion properties by using palm oil-based polyurethane polymers which are strengthened by the addition of Bentonite and Chitosan replacing petrochemical polymers in the paint industry so that can be applied to the manufacture of coating paints which currently use petroleum which is becoming scarce and not environmentally friendly as raw materials. The use of palm oil-based polyurethane polymer reinforced with the addition of Bentonite and Chitosan has shown many improved properties in previous polymer studies, especially increased thermal and anti-bacterial properties. The coating paint industry requires good thermal properties to prevent corrosion at high temperatures and good anti-bacterial properties to prevent the growth of bacteria that can be harmful to health. So the coating paint produced from this research has better conventional value than the coating paint previously available.

METHOD

3.1 Materials

The materials used in this research consisted of 3 types, namely polyurethane based on palm oil, bentonite and chitosan.

3.2 Methodology

3.2.1 Purification and Opening of Bentonite Interlayer

Weighed 20 grams of bentonite and then ground it using a crusher. Filtering was carried out using a 100 size sivetrayum. A total of 18.2 grams of cetyl trimethyl ammonim bromide (CTAB) was dissolved in 250 mL of distilled water, the solution was heated at 80 °C for 1 hour. A total of 20 grams of bentonite was dissolved in 250 mL of distilled water.

The bentonite solution dispersion was added to the CTAB solution and stirred for 1 hour. The bentonite is filtered and washed with distilled water several times until there is no more bromide. The filtrate was tested by dropping 1 M AgNO₃ until no white precipitate formed, indicating that the bromide had been removed. The bentonite is put into the oven at 60°C to dry.

3.2.2 Polyol Synthesis

The polyol synthesis process goes through two process stages, namely the epoxidation and hydroxylation processes. Polyol synthesis was carried out in a 350 mL 3 neck flask equipped with a mechanical stirrer and cooling system. 60 mL of glacial CH₃COOH and 30 mL of 30% H₂O₂ were slowly added into the reactor while stirring. Through a dropper funnel, 2 mL of concentrated H₂SO₄ was added and stirred slowly at a temperature of 30°C for 1 hour. Then, through a dropper funnel, oleic acid was added slowly. 100 mL of palm oil. The temperature was maintained at 30°C and continued to stir for 3 hours.

The reaction result is an oleic acid epoxidation compound, which is cooled to room temperature and the oil phase is separated as epoxidized oil which will then be used in the hydroxylation process. In the hydroxylation stage, 100 mL of methanol was added with 50 mL of glycerin, 2 mL of concentrated H₂SO₄ catalyst and 5 mL of water into a 350 mL three-neck flask, heated to a temperature of 40°C. The epoxidized oil solution was added to the mixture into a three-neck flask, stirred at 50°C for 2 hours. Next, it was cooled to room temperature and transferred to a separating flask to separate the polyol formed and then stored in a glass bottle.

3.2.3 Chitosan Synthesis

A total of 4.25 grams of chitosan was dissolved in 100 mL of 2% glacial acetic acid solution while stirring at a speed of 500 rpm for 2 hours with a pH of 4.0 until a chitosan suspension was obtained. Next, 50 mL of 0.1 N NaOH was dropped slowly into the chitosan suspension. Next, the chitosan suspension was

rinsed using 150 mL of distilled water or until the pH was neutral and dried in an oven at a temperature of 60°C, then the chitosan was analyzed.

3.3.3 Production of PU/Bentonite/Chitosan Coating Paint

Polyol, TDI, bentonite and chitosan were mixed in a glass beaker using a magnetic stirrer at 200 rpm for 1 hour. In this procedure, amounts of bentonite and chitosan are used, respectively 2,4,6,8, and 10 percent by weight (wt%). The resulting polyurethane is then cooled to room temperature. Next, the chemical structure of polyurethane, chitosan and bentonite paint was analyzed using FTIR. Paint heat resistance analysisThe coating was analyzed using TGA and surface shape analysis using SEM.

3.3 Sample Preparation

Mix polyol, bentonite, chitosan and then TDI into a glass beaker and stir using a magnetic stirrer at a speed of 200 rpm for 1 hour. In this procedure, amounts of bentonite and chitosan are used at 1, 3, 5, 7, and 9 percent by weight (%wt). The resulting polyurethane was then cooled to room temperature. Next, the chemical structure of polyurethane paint, bentonite and chitosan was analyzed using FTIR and the heat resistance analysis of the coating was analyzed using TGA and surface shape analysis using SEM.

3.4 Functional group analysis using Fourier Transform Infra Red (FTIR)

FTIR is used to analyze the characterization of polymer materials and functional group analysis. The synthesized polyurethane samples were crushed using mortar equipment. Samples were crushed with KBr using a Shimadzu FTIR spectrophotometer. Spectra were obtained in the mid-infrared region (4000-400 cm⁻¹) at room temperature.

3.5 Analysis of thermal properties using thermogravimetric analysis (TGA)

In principle, this method measures the reduction in material mass when heated from room temperature to high temperature which is usually around 900°C with a heating rate of 20°C/minute. The TGA tool is equipped with a micro balance in it so that the sample weight can automatically be recorded and presented in a graphic display.

3.6 Surface scanning electron microscopy (SEM) morphological analysis

A tool that forms a microscopic image of the surface of a specimen. An electron beam with a diameter of 5-10 nm is directed at the specimen. The SEM technique is basically an examination and analysis of the surface of a specimen, the data or appearance obtained is data from the surface or layer which has a thickness of around 20 μm from the surface. The surface image obtained is a photograph of all the protrusions, indentations and holes on the surface.

3.7 Bacterial Activity Test

To determine the effect of adding chitosan as an antibacterial, it was analyzed using the halo zone method by cultivating *S.Aureus* and *E.Coli* bacteria in the media. Then each plate that had been smeared with polyurethane was placed on the surface of the media, the samples were incubated for 8 hours at 37 °C. Observations were made by measuring the diameter of the clear area formed around the membrane to determine its antibacterial properties.

3.8 Corrosion Test

This was done to determine the effect of adding anti-corrosion paint to steel plates. Steel plates that are not coated with coating paint and steel plates that are coated with coating paint will be soaked in sea water containing ions for ± 1 month. 48 Iron samples were observed in the 3rd week to determine changes that occurred in the steel plate.

RESULTS AND DISCUSSION

4.1 Characterization of Fourier Transform Infra Red (FT-IR)

Fourier Transform Infrared(FT-IR) is used to determine the functional group bonds contained in polyurethane paint that has been modified with the addition of chitosan bentonite filler. The purpose of FT-IR analysis on polyurethane paint samples is to determine the wavelength and characteristic peaks in the sample. Each functional group has a specific characteristic peak. The FT-IR spectrum is presented in figures 3.1, 3.2 and 3.3.

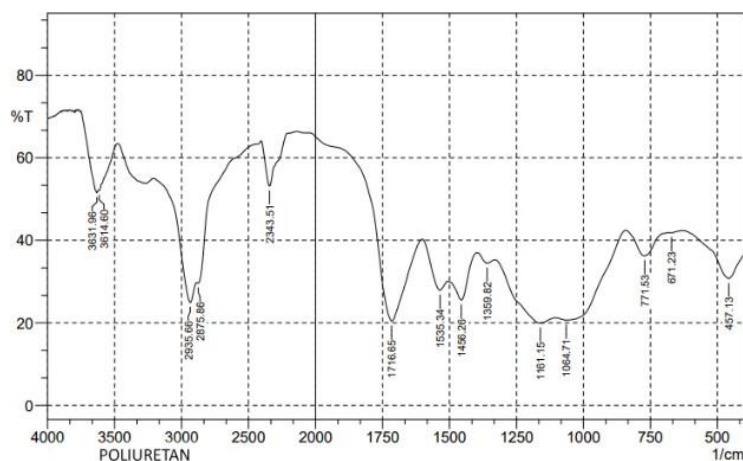


Figure 1. FT-IR Spectrum of Polyurethane

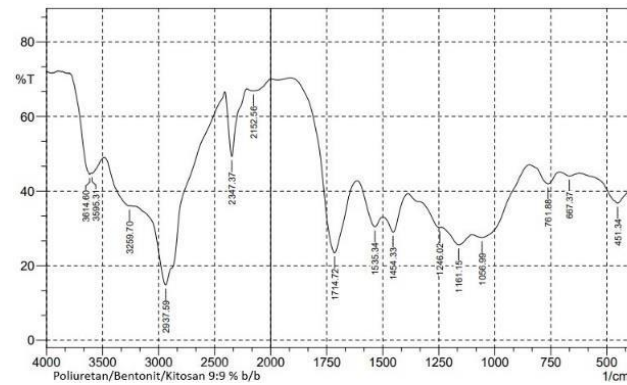


Figure 2. Polyurethane/Bentonite/Chitosan FT-IR Spectrum 9:9 % w/w

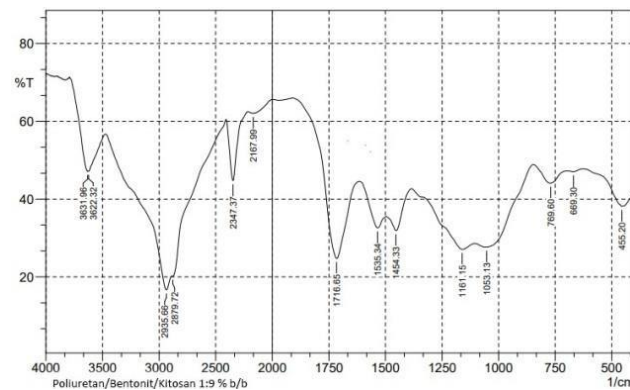


Figure 3. FT-IR Spectrum of Polyurethane/Bentonite/Chitosan 1:9 % w/w

The results of FT-IR analysis show that the formation of NH urethane groups in polyurethane compounds is indicated by the wide NH absorption wave number at 3630.95 cm^{-1} . The wave absorption of 2937.66 cm^{-1} , 1716.65 cm^{-1} shows the CH and C=O groups. In polyurethane that has been modified with the addition of 8% bentonite and 8% chitosan and the results of FT-IR analysis show the formation of NH urethane groups at an absorption wave number of 3612.60 cm^{-1} . The CH wave absorption at 2935.66 cm^{-1} and 1716.65 cm^{-1} shows the C=O wave absorption. In polyurethane that has been modified with the addition of 4% Bentonite: 4% Chitosan, NH urethane groups are formed at an absorption wave number of 3612.58 cm^{-1} . The CH wave absorption at 2932.58 cm^{-1} and 1716.55 cm^{-1} shows the C=O wave number absorption. In Figure 3.3 with a comparison sample of polyurethane that has been modified with the addition of 2% bentonite and 8% chitosan, it shows the characteristics of the number area. waves 3611.96 cm^{-1} , 2935.66 cm^{-1} and 1716.65 cm^{-1} indicate the NH, CH and C=O groups. The detected functional groups represent the functional groups of polyurethane (-NHCOO-).

The analysis results show that the functional groups of polyurethane and polyurethane that have been modified by adding bentonite-chitosan do not change the functional groups. Mixing polyurethane with bentonite and chitosan

does not change or affect the absorption of the functional group wavelengths of the polyurethane. This is because the mixing process takes place without changes in chemical bonds.

4.2 Thermogravimetric Analysis (TGA) Analysis Results

Thermogravimetric Analysis(TGA) is a tool used to quantitatively determine the thermal stability and mass loss of polyurethane samples that have been modified with the addition of bentonite and chitosan. Thermal stability is an important parameter for the selection and use of materials. The results of characterizing the properties of polyurethane paint using Thermogravimetric Analysis (TGA) can be seen in the following table:

Sample (%)	Onset (°C)	Endset (°C)	Weight loss (%)
8:8	416.85	498.94	-32,159
4:6	352.78	401.63	-45,738
6:6	346.82	378.59	-49,659
2:2	319.21	392.06	-55,736

The table above shows that all samples experience single decomposition where on set and end set only occur once. Of the four best samples tested using Thermogravimetric Analysis (TGA), it shows that the Polyurethane sample with modified bentonite 8: chitosan 8 (%) has the best thermal stability among the other samples, where the sample begins to degrade (onset) at a temperature of 416.85 (°C) and stopped degrading (endset) at a temperature of 498.94 (°C) with a weight loss of -32.159%. Polyurethane samples with modified bentonite 2: chitosan 2 (%) have lower thermal stability where the sample starts to degrade (onset) 319.21 (°C) and stops degrading (endset) at a temperature of 392.06 (°C) with weight loss -55.736%.

This shows that the addition of bentonite and chitosan can increase thermal stability. Based on the principle of Thermogravimetric Analysis (TGA), the sample with the highest degradation (onset) and degradable (endset) temperatures is the sample with the best thermal stability[4]. In this research, Polyurethane/Bentoni 8: Chitosan 8 (%) is the sample that has the best thermal stability because it has the highest degradation temperature (onset) and degradation temperature (endset).

4.3 Morphological Structure Test Results using Scanning Electron Microscopy (SEM)

SEM testing aims to support the best sample results which aims to support the best sample results taken from the TGA test. The samples tested were the best samples with variations of 8% bentonite: 8% chitosan.

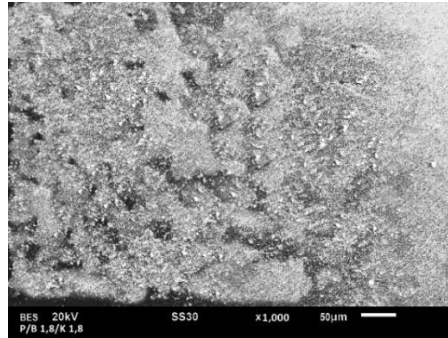


Figure 4. SEM Test Results for Polyurethane/Bentonite/Chitosan 8:8 % w/w

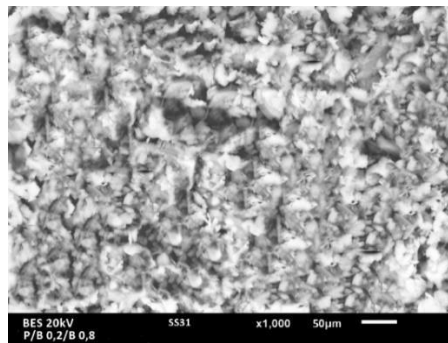


Figure 5. SEM Test Results for Polyurethane/Bentonite/Chitosan 2:8 % w/w

Tests using SEM showed that the chitosan chains were well dispersed into the bentonite interlayer. The formation of composite flocculation can be attributed to the interaction of the hydroxylated edges of the silicate layer between the hydroxylated edge groups of the silicate and the chitosan chains. (Rihayat et al., 2019). Figure 3.9 shows the surface structure of a sample where the chitosan contains a mixture of bentonite and chitosan. The darker surface is an iron plate that has been applied with polyurethane with the addition of bentonite-chitosan filler. Figure 3.9 shows that the sample is homogeneous and no agglomeration occurs. In samples with a Polyurethane/Bentonite/Chitosan ratio of 2:8% w/w, it shows that the sample surface is not homogeneous, where agglomeration occurs in the sample so that the sample surface appears uneven.

4.4 Anti-Bacterial Test

Table 1. Results of Anti-Bacterial Test

Sample (%)	Type of Bacteria	Clear Zone Vertical (mm)	Clear Zone Horizontal (mm)	P. Steel Plate (mm)	L. Steel Plate (mm)	Obstacle Zone (mm)
02:02	S. Aureus	18.3	11	17	10	1.8
02:02	E. Coli	18.7	11.3	17	10	2.35
02:04	S. Aureus	18.9	11.5	17	10	2.65
02:04	E. Coli	19.6	11.8	17	10	3.5
02:08	S. Aureus	20	12.2	17	10	4.1
02:08	E. Coli	21.7	13	17	10	6.2
04:02	S. Aureus	22.3	13.7	17	10	7.15
04:02	E. Coli	23	14.1	17	10	8.05
04:04	S. Aureus	23.2	13.8	17	10	8.1
04:04	E. Coli	25	16	17	10	11
04:06	S. Aureus	24.5	15.5	17	10	10.25
04:06	E. Coli	26.3	16.9	17	10	12.75
04:08	S. Aureus	24.9	16	17	10	10.9
04:08	E. Coli	26.7	17.3	17	10	13.35
06:02	S. Aureus	25.2	16.6	17	10	11.5
06:02	E. Coli	27.5	18.3	17	10	14.65
06:04	S. Aureus	25	17	17	10	11.5
06:04	E. Coli	27.9	21.1	17	10	16.45
06:06	S. Aureus	25.7	17.4	17	10	12.4
06:06	E. Coli	32	21.9	17	10	20.95
06:08	S. Aureus	25.9	18.2	17	10	13
06:08	E. Coli	32.5	22.5	17	10	21.75
08:02	S. Aureus	26.4	18.7	17	10	13.75
08:02	E. Coli	32.7	23.2	17	10	22.3
08:04	S. Aureus	27	19.2	17	10	14.6
08:04	E. Coli	33	24	17	10	23
08:06	S. Aureus	27.1	20.6	17	10	15.4
08:06	E. Coli	33.4	24.8	17	10	23.8
08:08	S. Aureus	28	21.3	17	10	16.65
08:08	E. Coli	33.8	25.9	17	10	24.75

Anti-bacterial testing aims to produce paint products that are able to prevent disease caused by bacteria and corrosion caused by bacteria. The sample to be tested is placed on agar media on which bacteria have been cultured. On the agar medium a clear zone will form and the diameter of the clear zone formed will be measured. Based on the results of anti-bacterial testing, it shows that the percentage of 8:8% w/w bentonite:chitosan has the widest resistance zone with a

value of 6.9 mm for bacteria. From the results of the bacterial test it can be concluded that the polycationic properties of chitosan which can disrupt bacterial metabolism work more on bacteria that have a negative charge such as E.Coli bacteria.

4.5 Corrosion Test

Corrosion is the main thing to be reviewed in this research. Corrosion tests were carried out to determine whether variations in the amount of bentonite and chitosan were able to minimize the occurrence of corrosion. Corrosion testing in this research uses the mass loss method to calculate the amount of mass lost due to corrosion. In this research, steel plates that are not coated with coating paint and steel plates that are coated with coating paint will be soaked in sea water containing ions for ± 1 month. 17 Iron samples were observed in the 3rd week to determine changes that occurred in the steel plate. The following is a graph of the comparative effect of variations in the amount of bentonite-chitosan on the corrosion rate.

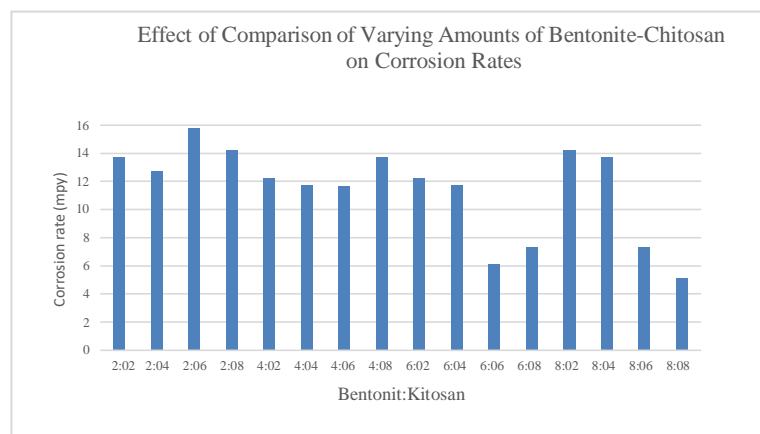


Figure 6. Effect of Comparison of Varying Amounts of Bentonite-Chitosan on Corrosion Rates

From the graph above it can be concluded that the amount of bentonite-chitosan contained in polyurethane paint affects the corrosion rate. This is because the addition of bentonite and chitosan fillers can act as reinforcing materials thereby increasing the mechanical and physical properties of polyurethane, which based on the graph above shows that the greater the amount of bentonite and chitosan can reduce the corrosion rate. In this study, the largest mass loss was the Bentonite - Chitosan ratio of 2:2 %w/w with a corrosion rate of 13.73 mpy. The sample with a Bentonite-Chitosan ratio of 8:8 %w/w experienced mass loss of at least a corrosion rate of 5.08 mpy. This shows that the greater the amount of bentonite and chitosan contained in polyurethane is able to reduce the

rate of corrosion on steel plates. Based on the corrosion resistance level table, all samples tested had a good level of corrosion resistance, where the corrosion rate for each sample was 5-50 mpy.

CONCLUSION

The best results were obtained from the composition Polyurethane/Bentonite/Chitosan 8:8 % w/w. This shows that increasing the amount of bentonite and chitosan can increase the reinforcement, thermal stability and mechanical properties of the coating material. FT-IR testing shows the presence of NH, CH, C=O groups which represent the functional groups of polyurethane (NHCOO-). The FTIR test results show that mixing polyurethane and bentonite does not have an impact on the wavelength absorption of the polyurethane functional groups, this is because the mixing process only occurs physical changes. In the Thermogravimetric Analysis (TGA) test, the sample experienced single decomposition and showed the best results for the Polyurethane/Bentonite/Chitosan 8:8 % w/w sample where the sample began to degrade at a temperature of 416.85(°C). SEM test shows that the chitosan chains are well dispersed into the bentonite interlayer. The results of the bacterial test showed the best results on the samples Polyurethane/Bentonite/Chitosan 8:8 % w/w has the widest zone of inhibition with a value of 6.9 mm for E.Coli bacteria.

Corrosion tests show that the addition of bentonite and chitosan affects the corrosion rate, the greater the composition of bentonite and chitosan can reduce the corrosion rate. The sample with a Polyurethane/Bentonite/Chitosan composition of 8:8 % w/w was the best sample where the sample experienced the smallest corrosion rate, namely 5.08 mpy.

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