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THE EFFECT OF CHITOSAN ADDITION ON ECO-FRIENDLY PLASTIC BASED ON PLA/PCL

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ABSTRACT

Research on making eco-friendly plastic has been developed because it is environmentally friendly and renewable. In this research, the manufacture of eco-friendly plastic from Polylactic Acid (PLA) and Polycaprolactone (PCL) with the addition of chitosan as a filler . The purpose of this study was to study and determine the effect of mixing variations in chitosan composition PLA on PCL the mechanical properties of eco-friendly plastics using a hot press at 200^0C for 1 hour. The variations of PLA/ PCL are 2/8 g, 3/7 g, 5/5 g, 7/3 g and 8/2 g while the composition of chitosan is 0,2 g, 0,3 g, 0,4 g, 0,5 gand 0,6 g. The characteristics of eco-friendly plastics can be seen from the biodegradation test, tensile strength test, elongation test, functional group test and plastic film morphology test. The results of the characterization of eco-friendly plastics with optimal performance were the composition PLA/PCLChitosan 8/2/0.6 gram yielded 38.8% for the biodegradation test, the tensile strength value was 42.53 MPa, the elongation percentage was 6.96%. While the functional groups contained are the N – H, C – H, C = O and C – O groups. The results of the identification of the functional groups show that no new functional groups are formed, but only a mixing process without any reaction on the constituent materials. Based on the morphological test results showed that the sample has a smooth surface. However, there is still insoluble chitosan because the mixing process is not homogeneous.

Keywords: Polylactic Acid (PLA), Polycaprolactone (PCL), Chitosan, eco friendly plastics and tensile strength tes

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INTRODUCTION

There has been concern for the development of bio-based plastics with reduced availability of fossil resources as well as increasing concentrations of carbon dioxide in the atmosphere. This type of plastic derived from petrochemical polymers is a very popular plastic used because it has several advantages. However, this plastic polymer cannot be destroyed naturally (non-biodegradable), causing environmental pollution. One of the biopolymers that has great potential to be developed as a substitute for conventional plastics is Polylactic Acid (PLA). PLA is a type of plastic produced from renewable natural materials such as starch through lactic acid fermentation. PLA is a biopolymer that is renewable and biodegradable. PLA polymer is strong, transparent and water resistant. In addition, the weakness of PLA is that it is stiff and has low permeability properties. To improve the biocomposite properties of PLA, several other polymers are added with the aim of reducing deficiencies and improving the biocomposite character. Modification of PLA by blending with other polymers can improve the mechanical properties in the form of tensile strength , elongation at break and degradation of PLA[1].

Another polymer that has the potential to improve the characteristics of PLA is Polycaprolactone (PCL). PCL is an aliphatic polyester which is biocompatible and has good permeability. In addition, PCL also has high crystallinity, low degradation rate and low melting point so that it has poor mechanical properties. However, with a relatively low melting point, it can be processed easily using conventional methods. PLA and PCL have the same properties, which are hydrophobic and can be well degraded. However, these two polymers also have different physical properties, namely PLA is transparent, very flexible and strong. Meanwhile, PCL is non-transparent, very brittle and stiff so it breaks easily. If these two compounds are combined, the resulting polymer can correct the deficiencies of the properties possessed by the original polymer[2].

The development of environmentally friendly plastic technology is currently experiencing very rapid progress as well as research on the effect of adding filler . Based on the research that has been done before, in general, the development of the mechanical, morphological and biodegradable properties of bioplastics replaces conventional plastics by varying the composition of the raw materials and fillers used. However, the resulting mechanical, morphological and biodegradable properties are inconsistent with differences in the type and composition of the raw materials and fillers used, so these three variables are the main concern of researchers to make bioplastics that are more representative[3].

Polylactic Acid (PLA) is a type of biopolymer that can be used as a substitute for conventional plastics, due to its biodegradability so it is classified as

a biodegradable polymer . Poly Lactic Acid was first produced in 1932 by Wallace Carothers who produced low molecular weight PLA and heated lactic acid under vacuum. Poly Lactic Acid with the chemical formula (CH3 CHOHCOOH) is a plastic polymer that is biodegradable, thermoplastic and is an aliphatic polyester produced from natural raw materials. PLA can be formed through the esterification process of lactic acid obtained by fermentation by bacteria using starch or simple sugar as a substrate. Lactic acid is the simplest hydroxy acid compound consisting of an asymmetric carbon atom. This acid can be produced from the fermentation of carbohydrates by bacteria in the form of L-lactic acid or D-lactic acid[4].

PLA has the ability to biologically degrade in the soil because it is obtained from renewable sources. PLA can be obtained from lactic acid which comes from sugar, starch, cellulose and glycerin from biodiesel residue. PLA is a polymer that has several uses, including for packaging, film-making and the medical industry (drug coating material, bone implantation and for surgical threads). In addition to the advantages possessed by PLA, this polymer also has disadvantages, namely PLA has hydrophobic properties which causes the rate of degradation through hydrolysis of the final ester bond to take quite a long time, which is an obstacle in biomedical applications and food packaging. However, this weakness can be reduced by modifying the polymer through a blending process with other biodegradable polymers . So that it can improve the mechanical properties in the form of tensile strength and elongation (elongation at break) as well as the rate of degradation. Polymer blending is an effective, simple and versatile method for developing new materials with certain properties without synthesizing new polymers[5].

Polycaprolactone (PCL) was discovered in 1973 which is an aliphatic semicrystalline polyester. Polycaprolactone with the chemical formula $C_6H_{10}O_2$ is an aliphatic polyester that is biodegradable, biocompatible and has good permeability. PCL is an ideal type of polymer because it is non-toxic, can be adsorbed after implantation and has good mechanical properties. This polyester is resistant to water, oil, solvents and chlorine, has low viscosity and is easy to shape. In addition, PCL also has several drawbacks, namely being hydrophobic, the biodegradation process is a little slow and has a sensitivity to microbial activity. PCL has a melting point of about 60ºC and a glass transition temperature (Tg) of -60 ºC. With these viscosity and melting points, PCL can be combined with other polymers and can also be processed easily. The nature of PCL which is permeable to drugs with low molecular weight (<400 Da) and non-toxic, makes PCL widely used in biomedical fields such as surgical threads and as a matrix for controlling drug delivery systems[6].

Chitosan is the result of deacylation of chitin found in the shells of shrimp, crabs, lobsters and insects with sodium hydroxide. Chitosan has a molecular formula glucuronamide ($C_6H_{11}NO_6$) with a molecular weight of 2.5 x 10 -5 Dalton. Due to the nature of chitosan which is environmentally friendly and non-toxic, it is widely used to synthesize biodegradable materials . The use of chitosan as a filler in the bioplastic synthesis process is due to the fact that chitosan can form clear, strong and flexible thin films. Chitosan is an additive in the manufacture of biodegradable plastics which functions to improve the transparency of the resulting plastic films. In addition, chitosan has properties as antimicrobial, non-toxic, biodegradable and easy to combine with other materials.

PLA and PCL in this study were produced from corn starch. Bioplastics made from starch have drawbacks, namely the low tensile strength of the resulting bioplastics and the high rate of water absorption. Therefore, starch-based bioplastics need to be mixed with chitosan (a biopolymer) which can reduce the level of water absorption because it is hydrophobic. Because corn starch is stiff and brittle, chitosan is used as a reinforcement for biodegradable plastics[7].

Chitosan concentration that is too high will decrease the tensile strength value because chitosan has a linear polymer chain structure. Where the linear chain structure tends to form a crystalline phase because it is able to arrange polymer molecules in an orderly manner. The crystalline phase provides strength, stiffness and hardness so that it can cause plastic films to break more easily[8].
The addition of chitosan aims to improve the physical and mechanical properties and protect the plastic film from microorganisms that can damage the plastic film[9].

In this study, variations in the ratio of PLA/PCL were used 2/8, 3/7, 5/5, 7/3 and 8/2 grams, while variations in the composition of chitosan were 0.2; 0.3; 0.4; 0.5 and 0.6 grams. Where the addition of chitosan can produce good physical properties and degradation processes. The use of PLA/PCL/chitosan can improve the mechanical properties of the resulting plastic films. These results can be seen from the degradation test, tensile strength test, functional group test and morphological test.

EXPERIMENTAL

2.1 Materials

The materials used in this study were Polylactic Acid (PLA), Polycaprolactone (PCL) and chitosan.

2.2 Methodology

2.2.1 Procedure for Making Eco-Friendly Plastic

Mix the PLA, PCL and chitosan polymers in specimen molds of ASTM Standard D-638 which are coated with aluminum foil using a hot press at a melting temperature of 200ºC for 1 hour. The ratio of PLA/PCL polymer mixed with various variations of chitosan where each total mixture meets the weight of 10 grams for each sample. The biocomposite that has been formed is allowed to stand at room temperature until it solidifies completely for further testing of tensile strength, SEM, FTIR and biodegradation.

2.3 Characterization Techniques

2.3.1 Biodegradation Analysis

Purpose ful degradation to estimate bioplastic decomposition time in the environment. Testing for bioplastics is carried out by planting samples in the soil for a certain time. Biodegradability testing is carried out by calculating the percentage of bioplastic weight loss and the rate of biodegradability.

2.3.2 Tensile Strength Analysis

Analysis is a method used to test the strength of a material which is one of the mechanical characteristics of a polymer material using a Universal Tensile Machine (UTM) tensile tester.

2.3.3 Functional Group Analysis (FTIR)

Functional group testing was carried out using a Shimadzu IR Prestige – 21 Fourier Transform Infrared (FTIR) instrument. FTIR is an instrument that uses the principle of spectroscopy. The purpose of FTIR analysis on plastic film samples is to see the wavelength and characteristic peaks in the sample.

2.3.4 Morphological Analysis (SEM)

Analysis was performed using *Scanning Electron Microscopy* (SEM). SEM is a type of electron microscope that produces an image of a sample by scanning a surface using an electron beam . This SEM analysis aims to determine the surface morphological structure of the material, crystallography and type of specimen.

RESULTS AND DISCUSSION

3.1 Biodegradation Analysis

Biodegradability is the main goal of making bio-polymer-based eco-friendly plastics . The biodegradation test aims to determine whether a material can be properly degraded in the environment so that it can be classified as an environmentally friendly polymer. Biodegradation processes can occur by

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hydrolysis (chemical degradation), bacteria or fungi, enzymatic degradation, wind and abrasion (mechanical degradation) and light (photodegradation). This process can also be done anaerobically and aerobically. In this study, the biodegradation process was carried out under aerobic conditions with the help of bacteria and fungi present in the soil for 7 days.

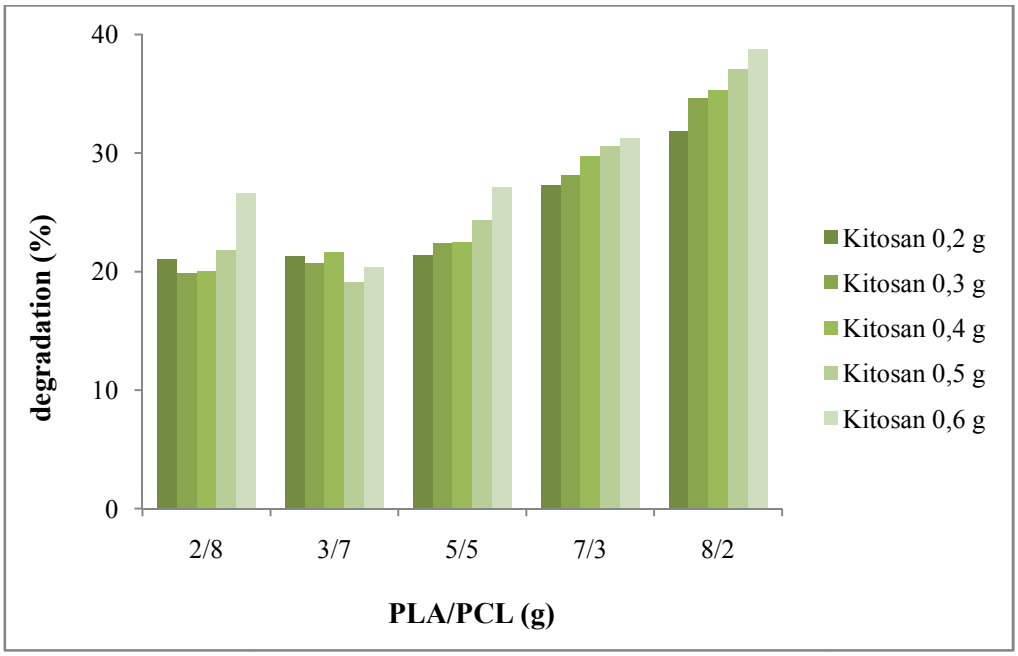

Figure 3.1 Graph of Relationship between PLA/PCL/Chitosan Composition to Degradation Percent

From graph 3.1 it can be seen that the highest score obtained was the percentage of degradation with the composition of PLA/PCL/Chitosan 8/2/0.6 gram, which was 38.8%. The results obtained show the loss of mass after the plastic film is embedded in the soil for 1 week. Increasing the composition of chitosan can increase the amount of mass lost due to the degradation process, so as to increase the percentage of degradation of the plastic film. In addition, the more composition PLA, the better the biodegradation process that occurs, this shows that PLA it has good degradability. While the use PCL of more composition compared to PLAit will give an irregular percentage of degradation along with the addition of chitosan. PLA and PCL is a biopolymer that has the ability to biologically degrade in the soil because it is obtained from a renewable source, namely it is produced from starch. So the blending process PLAand PCLwith the best composition can increase the percentage of plastic film degradation.

3.2 Tensile Strength Analysis

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Tensile strength analysis aims to determine the effect of variations of Polylactic Acid (PLA), Polycaprolactone (PCL) and chitosan on the tensile strength value and percent elongation (elongation) of the resulting plastic films. Tensile strength analysis was taken from the 3 (three) best samples of the biodegradation test results, namely with a composition of 8/2 gram of PLA/PCL and 0.2 of chitosan; 0.4 and 0.6 grams.

3.2.1 Effect of PLA/PCL/Chitosan Ratio on Tensile Strength of Plastics

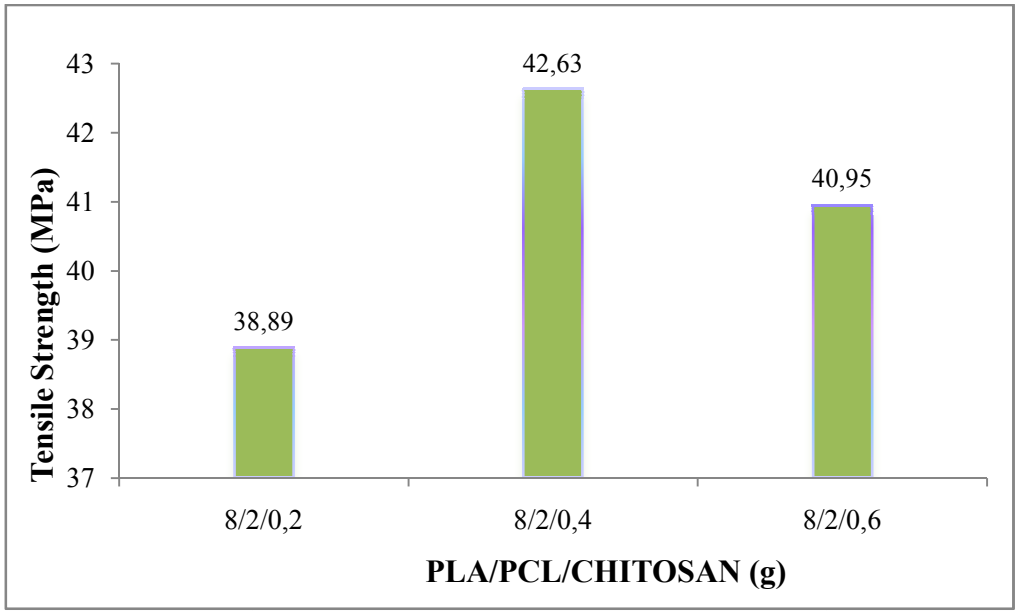

Figure 3.2 Graph of Relationship between PLA/PCL/Chitosan Composition on Tensile Strength

Graph 3.2 shows that the tensile strength of the plastic film produced has increased with the addition of chitosan from a concentration of 2% to 4%. In general, chitosan functions as a reinforcing material which is mixed into plastic film samples. However, along with the addition of chitosan at a concentration of 6%, the tensile strength of the plastic film decreased. It can be seen that the highest tensile strength is in the 0.4 gram (4% w/w) chitosan formulation. This shows that the addition of chitosan does not continuously increase the tensile strength of plastic films. The decrease in the tensile strength is due to the saturated conditions in the bioplastic matrix, so that the filler added with a large concentration cannot be distributed and mixed with the matrix.

In addition, the composition of PLA and PCL also affects the tensile strength value. In this study, the tensile strength test was carried out, namely the composition of PLA/PCL (8/2) gram. According to research conducted [10] the optimal composition for a PLA/PCL mixture is around 8/2 (w/w). The PLA/PCL

mixture with these compositions can maintain the high rigidity of the PLA matrix and sufficient PCL concentration to achieve high toughness. Based on research conducted [11] that the optimal composition for the PLA/PCL/chitosan mixture is 8/2/0.2 grams, which is 38.1 MPa. The results showed that the value of tensile strength and percent elongation decreased with the addition of chitosan.

3.2.2 Effect of PLA/PCL/Chitosan Ratio on Percent Plastic Elongation

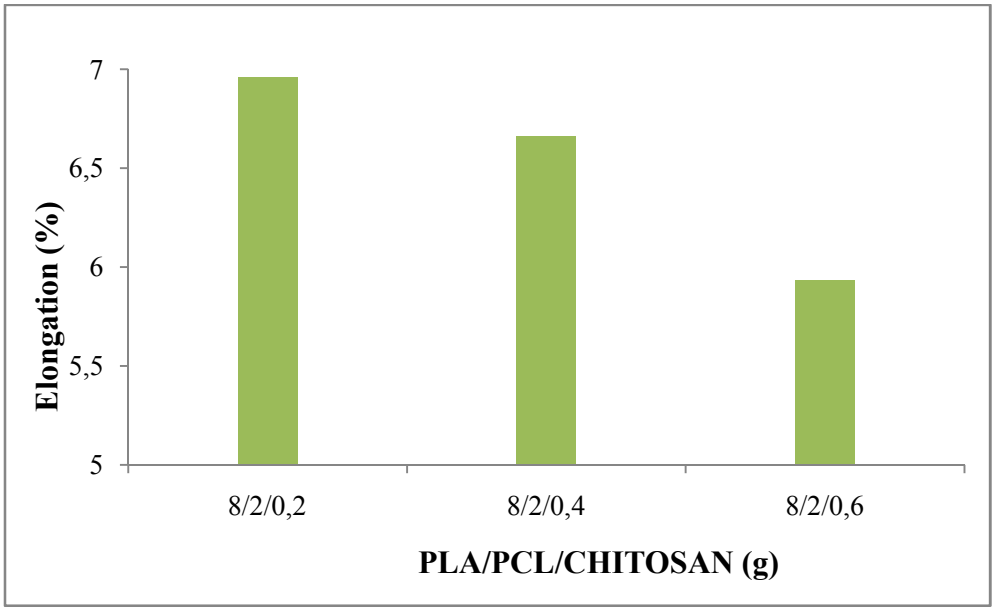

Figure 3.3 Graph of the Relationship of PLA/PCL/Chitosan Composition to Elongation

The percent elongation test is a test that is carried out simultaneously with the tensile strength test, in which the results of this test will get the percentage of plasticity properties and the maximum change in length when stretching occurs until the plastic film sample is broken. With this elongation test, it can be seen the rate of addition to the length of the material. Based on the graph above, it can be seen that the increasing concentration of chitosan addition causes the percentage of elongation to decrease, this is directly proportional to the tensile strength value, meaning that the resulting plastic film is more easily broken. This decrease in elasticity is due to the decreasing intermolecular bond distance as it passes the saturation point, which will reduce the intermolecular forces between the chains. In this test, the highest percentage of elongation was obtained with a composition of 0.2 gram (2% w/w) chitosan resulting in an elongation of 6.96%.

3.3 Functional Group Analysis of Compounds with FT-IR

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Functional group analysis was carried out to identify the functional groups present in plastic film samples using the Fourier Transform Infrared (FT-IR) tool. This analysis is based on the characteristic peak wavelengths of a sample. The wavelengths of these peaks indicate the presence of certain functional groups present in the sample because each functional group has specific characteristic peaks for certain functional groups. The spectrum of FT-IR analysis results can be seen in the image below.

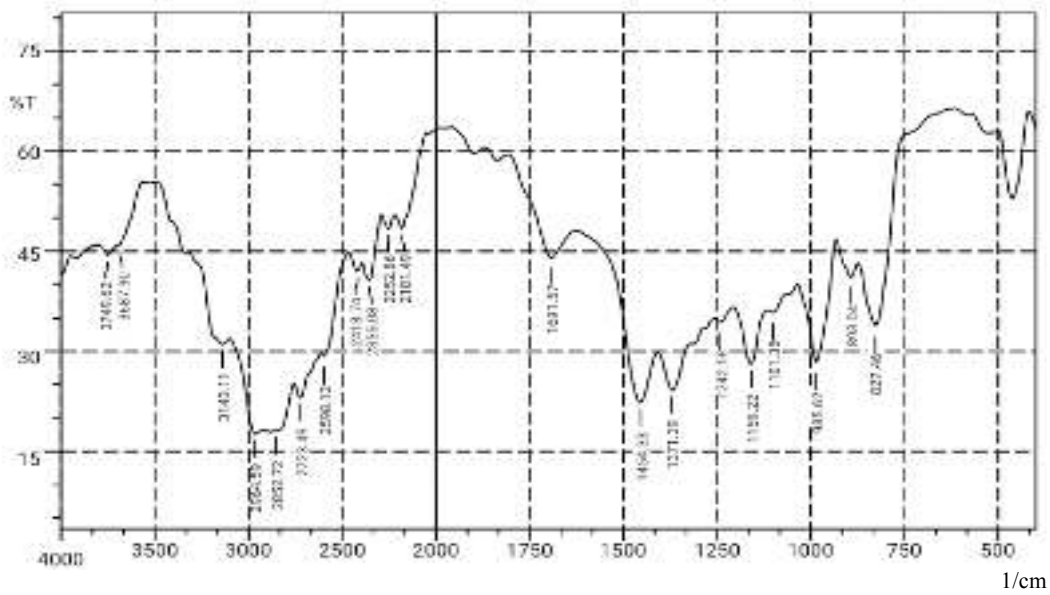

Figure 3.4 FT-IR Spectrum on PLA/PCL/Chitosan Samples (8/2/0.4) gram

Table 3.1 Functional Group Analysis Results in PLA/PCL/Chitosan Samples (8/2/0.4) gram

Frequency	Absorption (cm^{-1})	Functional groups
3300 - 3500	3140.11	N-H Stretching
3000 - 2850	2964.59	C-H Stretching
2500 - 2000	2355.08	C = O Stretching
1500 - 1250	1454.33	C – O Stretching

From figure 3 . 4 shows the results of the blending sample Polylactic Acid (PLA)/ Polycaprolactone (PCL)/chitosan 8/2/0.4 gram which is the best result from the research that has been done that there is absorption at 3140.11 cm -1 , which is evidenced by the presence of N - H groups, in accordance with In the literature, the absorption width that appears in the 3300 – 3500 area is an absorption of the N – H group. Meanwhile, in the 3000 – 2850 area there is an

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absorption of 2964.59 cm -1 where there is a C – H group, in the area 2500 – 2000 there is an absorption at 2355.08 cm - 1 where there is a C = O group and in the area 1500 – 1250 there is absorption at 1454.33 cm -1 where there is a C – O group. These results indicate the functional groups that make up cellulose.

The results of the identification of functional groups in table 4.4 show that of all the functional groups that appear the same as the basic ingredients used, namely Polylactic Acid (PLA), Polycaprolactone (PCL) and chitosan, they do not show the formation of new functional groups. So it can be concluded that the process of making eco-friendly plastic is just a mixing process without any reaction on the constituent materials. This causes the resulting plastic biocomposite to still have the properties of its constituent components.

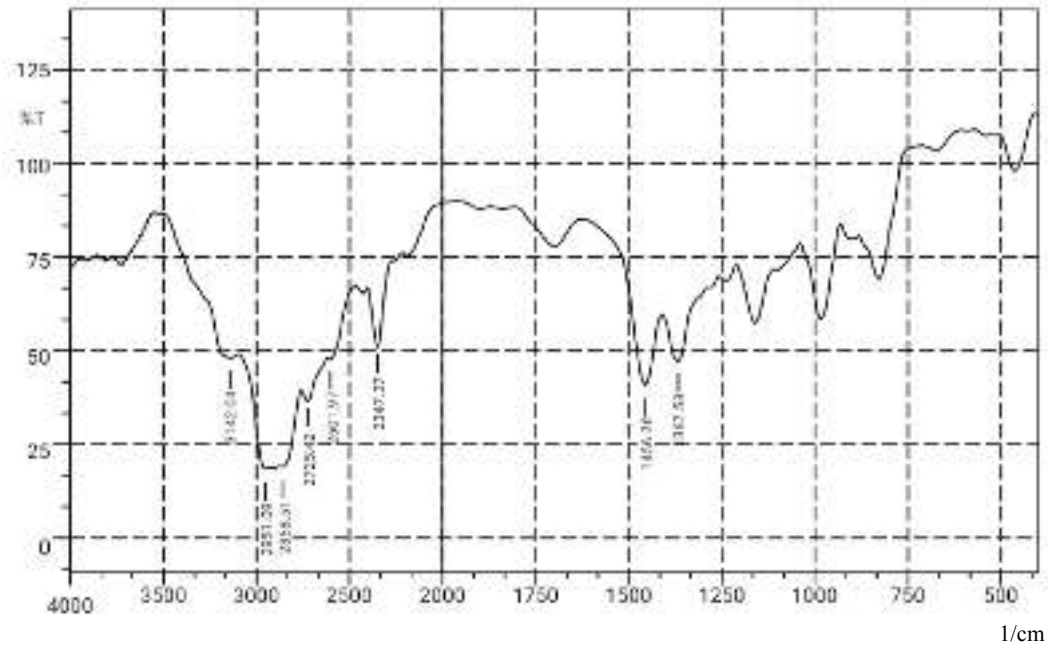

Figure 3 . 5 FT-IR Spectrum on PLA/PCL/Chitosan Samples (8/2/0.6) gram

Table 3.2 Functional Group Analysis Results in PLA/PCL/Chitosan Samples (8/2/0.6) gram

Frequency	Absorption (cm^{-1})	Functional groups
3300 - 3500	3142.04	N-H Stretching
3000 - 2850	2951.09	C-H Stretching
2500 – 2000	2347,37	C = O Stretching
1500 - 1250	1456,26	C – O Stretching

From figure 3.5 shows the results of the blending sample Polylactic Acid (PLA)/ Polycaprolactone (PCL)/chitosan 8/2/0.6 gram which has been carried out

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that there is absorption at 3142.04cm -1 , which is evidenced by the presence of N – H groups, in accordance with the literature that the absorption width appears at the 3300 – 3500 area is an absorption of the N – H group. While the 3000 – 2850 area has an absorption of 2951.09cm -1 where there is a C – H group, the 2500 – 2000 area has absorption at 2347.37cm -1 where there is a C = O group and the 1500 – 1250 there is absorption at 1456.26cm -1 where there is a C – O group. These results indicate the functional groups that make up cellulose. In Suryani's research, et al 2022 in the manufacture of environmentally friendly bioplastics from PLA/PCL with the addition of catechins and chitosan, similar results were obtained, namely that there were N – H, C –H, C = O and C – O groups formed in bioplastics.

The results of the identification of functional groups in table 4.5 also show that of all the functional groups that appear the same as the basic ingredients used, namely Polylactic Acid (PLA), Polycaprolactone (PCL) and chitosan, they do not show the formation of new functional groups. So it can be concluded that the process of making eco-friendly plastic is just a mixing process without any reaction on the constituent materials. This causes the resulting plastic biocomposite to still have the properties of its constituent components.

3.4 Morphological Analysis with SEM

Morphological testing is an additional test in this study which aims to support the best sample results from the biodegradation test. This test aims to look at the morphological structure of the PLA/PCL/chitosan blending biocomposite using a microscope that relies on electron beams to describe the surface shape of the material being analyzed. The following is an image of the results of the analysis using the Scanning Electron Microscopy (SEM) tool.

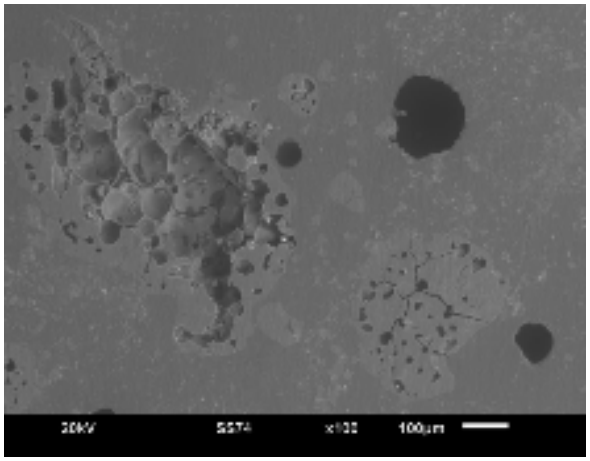

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Figure 3.6 Morphological Test Results for PLA/PCL/Chitosan Samples (8/2/0.4) gram

Figure 3.6 shows the morphological structure of the eco-friendly plastic film at 100x magnification. These results show that the surface structure of the sample still has a few white dots because the chitosan has not dissolved evenly. The image also shows the presence of bubbles scattered on the surface of the plastic film. This shows that the blending process between Polylactic Acid (PLA) and Polycaprolactone (PCL) was not perfect due to poor heating. The results obtained indicate that the sample has a smooth surface. However, there is still insoluble chitosan because the mixing process is not homogeneous. This is because the process of heating and stirring between the chitosan and the matrix is still not optimal. If there is a perfect heating and stirring process, it will be easy to combine the chitosan particles with the matrix, thereby strengthening the resulting plastic film.

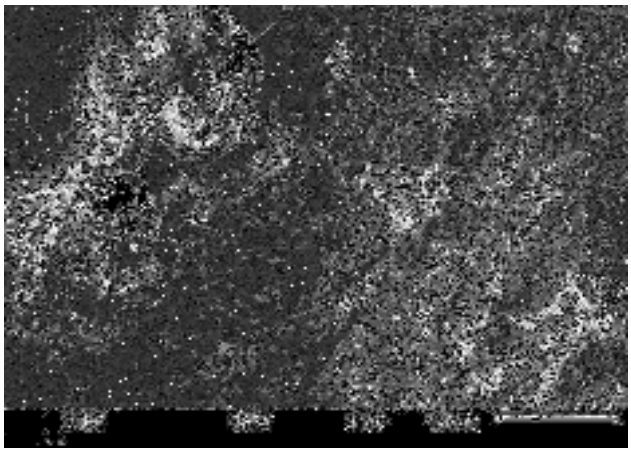

Figure 3.7 Morphological Test Results for PLA/PCL/Chitosan Samples (8/2/0.6) gram

Figure 3.7 shows the morphological structure of an eco-friendly plastic film at 100x magnification. These results indicate that the surface structure of the sample has white dots. This indicates that the chitosan particles undergo agglomeration in groups, causing the distribution of chitosan in the plastic film layer to not be spread evenly due to the absence of proper treatment such as heating and stirring between the chitosan and the matrix which causes uneven distribution of chitosan. If there is proper treatment such as good stirring during the mixing process at the gelatinization temperature it will easily combine the chitosan particles, thereby strengthening the plastic film. The image also shows the presence of bubbles scattered on the surface of the plastic film. This shows that the blending process between Polylactic Acid (PLA) and Polycaprolactone

(PCL) was imperfect due to poor heating. These results indicate that the sample has a less smooth surface, forms clumping aggregates and cracked plastic film surfaces due to poor bond formation between matrix and filler . In the study of Pamilia Coniwanti, 2014, it showed that the surface of the plastic film was not smooth because there was poor treatment in the stirring and heating processes

CONCLUSION

Based on the results obtained in this study, the best percentage of degradation was found in the composition of PLA/PCL/chitosan 8/2/0.6 gram, which was 38.8% . In the analysis of tensile strength values , the best (optimum) results were obtained for the composition of PLA/PCL/chitosan 8/2/0.4 gram, which was 42.63 MPa. Meanwhile, the best elongation percentage was with a composition of 8/2/0.2 gram PLA/PCL/chitosan, which was 6.96%. The results of the identification of functional groups show that it does not indicate the formation of new functional groups, it is only a mixing process without any reaction on the constituent materials. Based on the results of morphological analysis showed that the sample has a smooth surface. However, there is still insoluble chitosan because the mixing process is not homogeneous. This is due to the less than optimal stirring and heating. It can be concluded that the addition of chitosan will increase the tensile strength of the plastic film. However, if the addition is greater, the tensile strength value will also decrease and the percentage of elongation will also decrease. The larger composition of Polylactic Acid (PLA) can increase the percentage of plastic film biodegradation and the value of tensile strength.

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