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THERMAL BEHAVIOR IMPROVEMENT OF BIODEGRADABLE FIBER POLYMER COMPOSITES POLYLACTIC ACID (PLA)/COIR USING ACEH'S BENTONITE

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ABSTRACT

Research has been conducted on the manufacture of PLA Coir Bentonite composites. This study aims to examine the effect of PLA on mechanical strength with the addition of coir and bentonite fillers from North Aceh and Central Aceh. The sample formulations used were single polymer PLA/Coir and PLA/Coir with variations of filler Bentonite Aceh Utara and Aceh Tengah with 2, 4, 6 and 8% respectively. The combination of PCa samples showed the highest bacterial colony growth rate, which was more than 100 colonies/gram during the 1 week testing period. In the PBATd filler mixture sample, the maximum bacterial test value was 65 colonies/gram and the minimum value contained in the PBAUa sample was 105 colonies/gram. The best tensile strength was obtained in the PBATc sample, namely 65 MPa. PBATd samples began to degrade at 370.15°C compared to PCa samples degraded at 280.21°C. While the PBAUa sample began to degrade at a temperature of 282.11°C. The surface structure of the PCa sample is more homogeneous because there is no bentonite filler mixture, but it is brittle and crumbles easily. For the PBATd sample, the surface structure is smoother and more homogeneous compared to the PBAUa sample.

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Keywords: Polylactic Acid, Coir; Bentonite, Composites, Thermogravimetric Analysis (TGA)

1. INTRODUCTION

Research on environmentally friendly products is currently very popular, especially in the field of researchers or industrial players. Several polymer materials derived from environmentally friendly materials (biopolymers) have been produced, such as PHA Poly Hydroxy Alkanoate, Poly Hydroxy Butyrate and Poly Lactic Acid Chern et al. (2013).

Polylactic acid is one of the biopolymers belonging to polyester which has excellent biodegradable, biocompatible and recyclable abilities and the ability to process thermoplastic properties. Hin et al. (2012), Bino et al. (2015) PLA polymers have received a lot of attention and show great potential in bioplastic and biomedical applications among synthetic polymers. However, this biopolymer also has disadvantages, one of which is its low melting point, so that its heat resistance is not very good. Therefore, to overcome these weaknesses, PLA's properties can be improved by adding nano-sized fillers to form nanocomposites. Douglas et al. (2015) .

Based on several well-known references, one of the other fillers that are widely researched which is believed to be superior is bentonite. Bentonite is getting the most attention based on its ability to spread between layers widelyand its ability to expand. Bentonite is a mineral consisting of hydrated alumino-silicate crystals containing alkaline or alkaline earth cations in a three-dimensional framework. These metal ions can be replaced by other cations without damaging the structure of the bentonite and can reversibly absorb water. Bentonite can expand, have ion-exchange properties, large surface area and easily absorbs water allowing its use as an adsorbent Hasnan et al. (2015), Najafi et al. (2012).

In addition, the surface acidity of bentonite associated with Brønsted acid and Lewis's acid allows the use of bentonite as a catalyst. In addition, to add high mechanical properties and biodegradable characteristics, Coir composites were added. This composite has several major advantages over conventional composite materials, such as environmental friendliness, light weight, low volumetric cost. Among natural plant fibers, coir is widely used in several applications. Teuku et al. (2019), Pradeep and Edwin (2015) The annual production of coconuts worldwide is about more than 40 million tons, which is about 50 billion coconuts. However, coconut coir fiber has a low cellulose content of about 42% and hemicellulose 0.3%, with a high lignin content of 45% compared to other natural fibers. This fiber is one of the few natural fibers that are resistant to damage by salt water and is therefore relatively water-resistant. Purwaningsih et al. (2012), Shumigin et al. (2011), Suryani et al. (2016), Syamani et al. (2014), Suryani et al. (2018)

Among natural plant fibers, in this research, we will try to modify montmorillonite which is the result of purification from North Aceh Bentonite and Central Aceh Bentonite, which the mixing process will then carry out with PLA and Coir, where the resulting product will be compared with commercial products. The sample formulations used were PLA/Coir polymer with filler variations of Bentonite Aceh Utara 2, 4, 6 and 8% and PLA/Coir polymer, which were given 2, 4, 6 and 8% Aceh Tengah bentonite, respectively. The polymer characteristics with the addition of bentonite produce a more flexible and stronger polymer than the PLA/Coir alloy. The type of bentonite and its shape affect biodegradability, mechanical properties, thermal properties, and the morphological structure of the composite showing the increasing presence of molecules based on bacterial test results tensile test, TGA test (thermal) and SEM test. The addition of bentonite filler to PLA and Coir polymers can affect the material's mechanical properties. Tawakkal et al. (2012)

2. MATERIALS AND METHODS

2.1. MATERIALS

The main material used in this research is Poly Lactic Acid (Nature Work Co). The bentonite used comes from two different areas: bentonite from the Nisam area, North Aceh, and Bentonite from the Kab. Bener Meriah, Takengon, Central Aceh. Additional materials used in this study were Cetyl Trimethyl Ammonium Bromide/CTAB (Sigma Aldrich), Coconut Coir (Coconut Coir) and $(NaPO_3)_6$ (Merck).

2.2. METHODS

2.2.1. PREPARATION COIR

The chemical treatment carried out is by alkalizing treatment on the fiber. The chemical treatment is done by soaking the fiber using NaOH (sodium hydroxide) and $KmnO^4$ (potassium permanganate). Because NaOH and $KmnO^{4-}$ are alkaline types, they are therefore used to remove the oil content (lignin layer) in fibers and reduce impurities that cause sera not to bond completely with the matrix/resin when the composite is printed, making the fiber last longer. longer against the attack of bacteria/microorganisms compared to fibers without immersion of NaOH and KmnO^{4-}. Soaking the fibers with NaOH solution itself is done by adding 5% NaOH and 5% KmnO^{4-}.

2.2.2. SAMPLE PREPARATION

Four containers were prepared for samples of the PLA/Coir mixture and bentonite. Mixed PLA/Coir and Bentonite Aceh Utara (total weight = 20 g) with the following ratio: 19.6 g: 0.4 g (2% wt); 19.2 g: 0.8 g (4% wt); 18.8 g: 1.2 g (6% wt); 18.6 g: 1.4 g (8%). The mixture is melted in melt blending at a temperature of 140^{o}C. Then it will harden by itself to form PLA/Coir-Bentonite nanocomposite. PLA/Coir-Bentonite nanocomposite is then cut into granules and put into specimen molds according to ASTM 638 D Type IV Standard, then compacted by hot press with a temperature of 180^{o}C. PLA/Coir-Bentonite nanocomposites that have been molded are then dried in a vacuum oven at 60oC for 24 hours or dried in ambient air. Zulkifli et al. (2018), Rihayat et al. (2018)

2.3. CHARACTERIZATION TECHNIQUE

2.3.1. BACTERIA TEST

Microbial testing was carried out using a method that is often used, namely compost soil media. All samples were placed in a container filled with soil/directly in nature and then left until the degradation level obtained reached approximately two months until completely degraded and the changes in the sample were only seen to change for one month. Jaafar et al. (2019)

2.3.2. PULL TEST

Engineering tensile tests are mostly carried out to complement the basic design in-formation for the strength of a material and as supporting data for material specifications. In the tensile test, the test object is given an axial tensile force load which increases continuously, at the same time observing the elongation experienced by the test object. Zulkifli et al. (2018), Rihayat et al. (2018)

2.3.3. THERMOGRAVIMETRIC ANALYSIS (TGA)

TGA is the most common, simple, and fast technique for kinetics analysis for de-volatilization processes. In principle, this method measures the reduction in the mass of the material when heated from room temperature to a high temperature which is usually around 900^{o}C. Rihayat et al. (2017), Azhar et al. (2021), Qamara et al. (2021) , Nurdiana et al. (2013).

2.3.4. SCANNING ELECTRON MICROSCOPY (SEM)

Scanning Electron Microscope (SEM) is an electron microscope that is used to view the image surface of a material; besides that, it can also provide information regarding the chemical composition of a material, both conductive and non-conductive materials. This microscope uses electromagnetic and electro static to control the incoming light and the appearance of the resulting image. Bhasney et al. (2020)

3. RESULTS AND DISCUSSIONS

3.1. BACTERIA TEST

Bacterial testing was carried out using the ALT (Total Plate Number) method and biodegradation testing using the composting technique. The process of biodegradability can occur by hydrolysis (chemical degradation), bacteria/fungi, enzymes (enzymatic degradation), wind and abrasion (mechanical degradation), light (photodegradation). Fahmi and Hermansyah (2011), Ginting (2016), Ajeng and Dianita (2017) The time interval of the checks carried out in the sample testing lasted for 0; 3; 6; 9; and 12 days by observing the development of the stained plate that has been placed Potato Dextrose Agar (PDA) as a growing medium for bacteria. The development of bacteria in 9 types of samples is shown in Table 1.

Table 1 Bacteria Test Sample

No	Sample	Day (%)			
		3	4	9	12
1	PCa	74	93	119	130
2	PBAUa	68	81	93	105
3	PBAUb	62	70	84	92
4	PBAUc	54	66	79	87
5	PBAUd	41	50	63	70
6	PBATa	59	74	84	96
7	PBATb	53	69	72	84
8	PBATc	46	51	61	71
9	PBATd	39	47	55	65

From Table 1, Microbial development during the testing process that occurred in PCa samples showed the highest colony growth rate, which was more than 100 colonies/gram during the 1 week testing period. This is because the composite is directly contaminated with air which contains various types of microbes that can affect the material both physically and chemically. The results showed that the PBAUTd sample had the lowest colony growth rate, 65 colonies/gram. It was followed by PBATa, PBATc and PBATd with colony growth rates of 96, 84 and 71%. Then the PBAUa, PBAUb, PBAUc and PBAUd samples obtained colony growth rates of 105, 92, 87 and 70%.

In samples without filler, the average structure change tends to be porous and darker because more filler is added. While the PLA/Coir combination polymer without bentonite filler mixture has a structure that tends to crack and has a color that is not so dark. This proves the collaboration of polymer alloys and filler alloys, which provide resistance to microbial degradation. Astika and Dwijana (2018)

3.2. TENSILE STRENGTH TEST RESULTS

Tensile strength PLA/Coir, PLA/Coir/Bentonite AU, PLA/Coir/Bentonite AT. The tests conducted show the variations of the polymer and its fillers, in research on the addition of bentonite fillers with a concentration of 2% 4% 6% to 8%. But the results obtained contradict the observations.

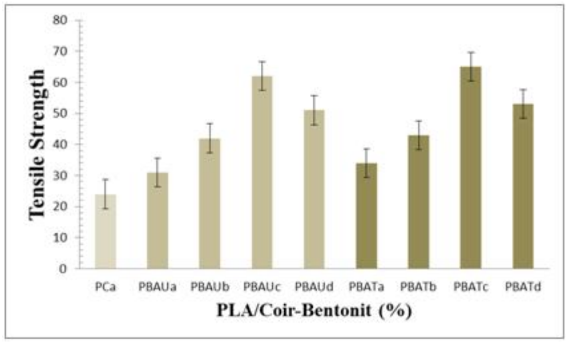


Figure 1 Graph of Tensile Test Results for Sample Formulations

Based on the test results, it can be seen that the addition of a polymer filler shows an increase in the properties of the composite. The new material produced shows an increase in the quality of its strength properties which are better than the pure PLA/Coir polymer without mixing. The PLA/Coir-Bentonitenanocomposite samples in each variation in the type and amount of bentonite filler used showed differences. The value of the tensile strength test is directly proportional to the number of fillers used. The more amount of bentonite mixed into the PLA matrix, the greater the value of the resulting tensile test, but if the filler is added with overload, it will experience a decrease in the tensile strength value. The results of the tensile test of the PCa sample, the tensile strength value of 24 Mpa is brittle, then with the bentonite filler from Central Aceh region for each variation of 2, 4, 6 and 8%, the tensile strength and stress at break values were higher at 34, 43, 65 and 53 Mpa. Compared to mixing with bentonite filler from North Aceh for each variation of 2,4, 6 and 8% of the North Aceh bentonite, the tensile strength values were obtained at 31, 42, 62 and 51 Mpa. The maximum value in the PBAUTd sample is 65 MPa and the minimum is the PBAUa sample with a value of 31 MPa.

3.3. THERMOGRAVIMETRIC ANALYSIS

Mass loss in the thermal test (TGA) occurs due to the decomposition process, namely the breaking of chemical bonds in the composite material. Fahmi and Hermansyah (2011), Ginting (2016). Figure 2 is a graph of the TGA test results for nine samples with the highest values in the previous test, namely PLA/Coir Coir without bentonite filler, PLA/Coir Coir/Bentonite AU variations and PLA/Coir Coir/Bentonite AT variations.TGA results showed an im-provement in the thermal stability of the biocomposites as nanoclay content was in-creased due to thermal barrier effects of nanoclays. Piekarska et al. (2017), Kurzina et al. (2020)

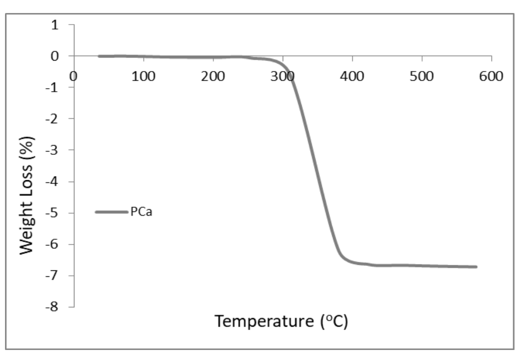


Figure 2 TGA Graph of Each Sample PCa (pure PLA) to Temperature

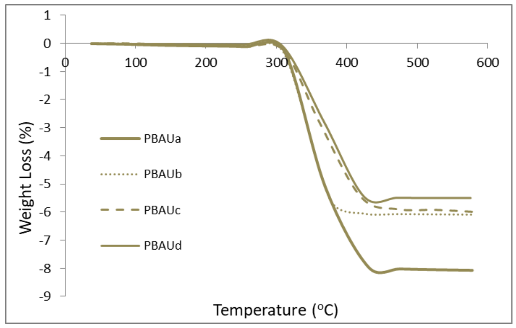


Figure 3 TGA Graph of Each Sample PBAU to Temperature

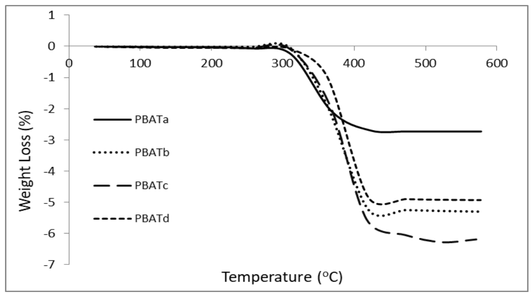


Figure 4 TGA Graph of Each Sample PBAT to Temperature

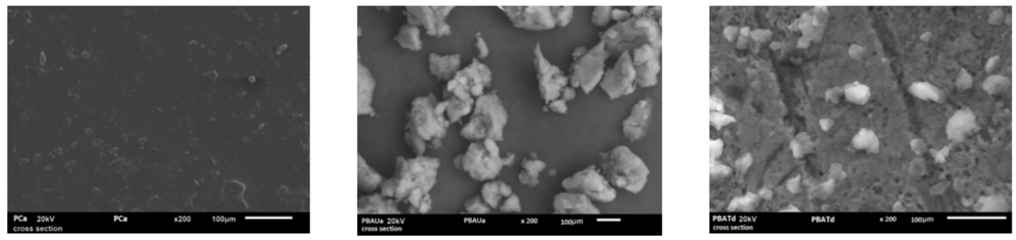
it can be seen that the graph shows that all samples undergo a single decomposition. The degradation temperature range is in the temperature range of 280-370°C. At a temperature of 105-200°C, the composite loses mass, which is the evaporation of the moisture content in the composite. Linyun et al. (2020)

PLA/Coir composite mixture with the addition of bentonite has a better thermal stability ability than without bentonite. PCa samples were degraded at a temperature of 280.21°C. While the PBAUa, PBAUb, PBAUc and PBAUd samples began to degrade at temperatures of 282.11°C, 289.76°C, 305.54°C and 315.04°C, then samples of PBATa, PBATb, PBATc and PBATd began to degrade at 320°C each. 119°C, 349.99°C, 357.76°C and 370.51°C the results of this study indicate that the addition of this AT bentonite filler mixture has succeeded in increasing the thermal and mechanical stability of AU bentonite which is characterized by an increase in the degradation temperature. The increase in temperature in the composite is caused by the loss of polymer and filler bonds, making it difficult to break and the decomposition of the material becomes slower. Rodchanasuripron et al. (2020), Joowon et al. (2020)

3.4. SCANNING ELECTRON MICROSCOPE ANALYSIS OF BENTONITE

Scanning Electron Microscope is a type of electron microscope that depicts the surface of a sample through a scanning process using a high-energy beam of electrons in a raster scan pattern. Yingfeng et al. (2020) Electrons interact with atoms that will make the sample generate signals and provide information about the sample's surface topography, composition and other properties such as electrical conductivity. SEM analysis was carried out to determine the morphology of the single polymer PLA/Coir, PLA/Coir with the addition of AU bentonite and AT bentonite. In principle, if there is a change, for example, a fracture, indentation and a difference in the material structure, the energy tends to change. Tsung-Han et al.

(2020) The changed energy can be emitted, reflected, absorbed, and converted into a wave function of electrons that can be captured and read on a SEM photo. SEM analysis results show differences in the morphology of the single polymer PLA/Coir, PLA/Coir with the addition of fillers of AU bentonite and AT bentonite which have been modified using cationic surfactants. SEM analysis results can be seen in Figure 5.



(a) (b) (c)

Figure 5 SEM analysis results (a) PCa (b) PBAUa (c) PBATd

SEM images of PLA/Coir single polymer, North Aceh bentonite and Central Aceh bentonite were taken at a magnification of 1,000 X. Based on the results of surface morphological characterization seen from the SEM results, the surface structure of PLA/Coir single polymer, North Aceh bentonite and Central Aceh bentonite has a surface that is In contrast, the surface structure of the PCa sample is more homogeneous because there is no bentonite filler mixture, for the PBATd sample, the surface structure is smoother and more homogeneous compared to the PBAUa sample. The large particle surface size indicates a good combination quality with the addition of filler to the PLA/Coir polymer. SEM images of PLA/Coir single polymer, North Aceh bentonite and Central Aceh bentonite were taken at a magnification of 1,000 X. Based on the results of surface morphological characterization seen from the SEM results, the surface structure of PLA/Coir single polymer, North Aceh bentonite and Central Aceh bentonite has a surface that is In contrast, the surface structure of the PCa sample is more homogeneous because there is no bentonite filler mixture, for the PBATd sample, the surface structure is smoother and more homogeneous compared to the PBAUa sample. The large particle surface size indicates a good combination quality with the addition of filler to the PLA/Coir polymer. Observing the internal morphology of the material using a Scanning Electron Microscope (SEM) in the fault area indicates the presence of voids and fiber breaks in the internal part of the microcomposite material. Some fibers experience pull-out when tensile and bending tests are carried out. Sung et al. (2017)

3.5. BENDING TEST ANALYSIS

The bending test or flexure test is applied to materials that receive bending loads, such as metal springs, ceramic floors, stone, plastic wood, and concrete girders. In general, the bending testing process has two test methods, namely: three points bending and four-point bending. Nurdiana et al. (2013), Bhasney et al. (2020) To determine the flexural strength of a material can be done by testing the flexural of the composite material. The flexural test refers to the ASTM D790 standard with static test conditions. Flexural strength or bending strength is the largest flexural stress that can be accepted due to external loading without undergoing major deformation or failure. The amount of flexural strength depends on the type of material and loading. As a result of the bending test, the upper part of the test object experiences stress, while the lower part will experience tensile stress.

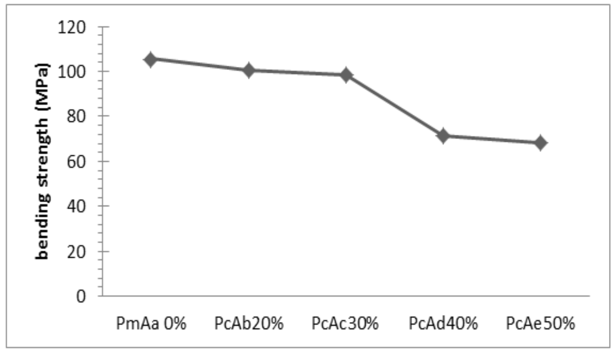


Figure 6 Pure PLA bending test results and fiber volume fraction

The highest bending strength of the composite material as a result of this study was owned by a 0% unreinforced material of 105.61 Mpa and a composite material with a 20% mass fraction of coconut fiber, which was 100.76 Mpa. The addition of the mass fraction of coco fiber resulted in a decrease in the value of the bending strength of the synthesized composite material.

4. CONCLUSIONS AND RECOMMENDATIONS

The results showed that the PBATd sample produced the best bacterial test value, 65 colonies/gram in 12 days. In the tensile strength test of PLA/Coir-Bentonite, nanocomposites were able to produce a better tensile strength value compared to pure PLA/Coir without mixing with bentonite filler. The value of the PBATc sample tensile strength is 65 MPa. The tensile strength value increases with the addition of the filler concentration, but the tensile strength value will decrease if an excess concentration or overload of filler is added. PLA/Coir composite mixtures with the addition of bentonite have better thermal stability capabilities. PBATd samples were degraded at 370.51^{o}C compared to PCa samples degraded at 280.21^{o}C. While the PBAUa, PBAUb samples, PBAUc and PBAUd began to degrade at temperatures of 282.11^{o}C, 289.76^{o}C, 305.54^{o}C and 315.04^{o}C, respectively. For the single polymer surface structure of PLA/Coir, Aceh Utara bentonite and Aceh Tengah bentonite have different surfaces, the surface of the PCa sample is more homogeneous and looks very scattered compared to PBAU and PBAT, the surface structure can still have fine lumps but the PBAT sample is visible the structure is more homogeneous and smoother than the PBAU sample. The type of bentonite that is best used as a filler in this study is bentonite from the Central Aceh region because of its higher montmorillonite content and greater swelling power compared to bentonite from the North Aceh region. Aceh Utara bentonite and Aceh Tengah bentonite have different surfaces. The surface of the PCa sample is more homogeneous and looks very scattered compared to PBAU and PBAT, the surface structure can still contain fine lumps but the PBAT sample looks more homogeneous and smoother than the PBAU sample. The type of bentonite that is best used as a filler in this study is bentonite from the Central Aceh region because of its higher montmorillonite content and greater swelling power compared to bentonite from the North Aceh region.

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