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The Effect of Alkalization Process on The Mechanical Properties of Straw Biocomposites as Egg Tray Materials

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Abstract. *Rice plants constitute the majority of crops grown by farmers in Indonesia. Straw is a harvest from rice plants which is still underutilized. The majority of farmers burn this straw waste to destroy it. This creates serious problems and can pollute the environment. One alternative for utilizing waste from straw is to make a biocomposite product in the form of egg tray molds from rice straw. One material that can be used as an alternative is cassava starch because it can decompose naturally. In this study, biocomposite material was made from cassava starch reinforced with rice straw which had been alkalized with varying concentrations of NaOH (7, 8, and 9%), boiling time (30, 45, and 60 minutes), and boiling temperature (70-80, 80 -90, and 90-100°C) and uses the casting method in the manufacturing process. The tests carried out are tensile testing and biodegradable testing. Tensile testing was carried out to determine tensile strength, strain and tensile modulus using ASTM D638 Type IV. Biodegradable testing was carried out to determine the level of natural degradation of biocomposites using ASTM D6868. The highest tensile test results were shown with the A3 specimen with variations in the alkalization process of 9% NaOH, 45 minutes, and 70-80°C. This specimen produces a tensile stress of 9.23 MPa, a strain of 14.82%, and an elastic modulus of 62.28 MPa. The results of the degradation test showed that specimen t1 with variations in alkalization of 8% NaOH concentration, 30 minutes, and 80-90°C with a degradation rate of 3.7%/day. These findings demonstrate that alkalization significantly improves the mechanical integrity and biodegradability of rice straw biocomposites, highlighting their potential as eco-friendly alternative materials for bio-based packaging applications, such as molded pulp or egg tray packaging.*
Keywords: *Cassava Strach; Rice Straw; Biocomposites; Mechanical Properties; Alkalization.*

Type of the Paper: Regular Article.

1. Introduction

Indonesia is an agrarian country with abundant natural resource potential so that is the agrarian sector, especially agriculture, becomes dominant [1]. One of the greatest natural resources in Indonesia is a food resource. The size of the agricultural sector makes rice production will leave behind large amounts of waste in the form of straw too.

According to Parshad et al. [2], rice straw is agricultural waste. with the largest amount in the world. Usually, Indonesian people utilize straw waste as animal feed. However, the utilization of straw waste is still not optimal because animal feed from rotting straw, people tend to burn it and this This causes environmental pollution and is a contributor to greenhouse gases. glass.

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Therefore, it is necessary to carry out development in the form of technological progress and support policies to increase productivity and sustainability [3].

According to Koekoeh et al. [4], rice straw has the potential to be used as a reinforcement in the manufacture of biocomposites. In order to reduce straw waste, researchers utilize the waste optimally by turning it into a material for Egg tray mold biocomposite. The following is an example of an egg tray biocomposite.



Fig. 1. Egg tray from biocomposites [5]

Fig. 1 shows an example of an egg tray biocomposite [5]. The manufacture of this biocomposite aims to create a material with better mechanical properties than egg trays generally (sold in the market). Rice straw contains 37.71% cellulose; 21.99% hemicellulose; and 16.62% lignin. This relatively high cellulose content can be utilized in various ways, including as a bioplastic material [6,7]. In order to maximize the results of straw biocomposites, we must First process the rice straw through an alkalization process.

The alkalization process is a chemical process that is often carried out with the aim of removes lignin structure and other impurities in fiber. Alkalization is one of the important parameters in the composite manufacturing process [8] because it is expected can affect the physical structure so that it can have greater mechanical strength good [9].

During the alkalization process, the use of NaOH can release the fiber from the hydroxyl groups. (OH-) so that the group binds with Na+ and forms ONa+. The ONa+ bond can degrades the lignin contained in the fiber so that when it is rinsed, the lignin will also lifted. OH- ions react with the H groups in lignin and form H2O. With the alkalization process can increase the cellulose content in rice straw by up to 90% [10].

Biocomposites consist of a matrix and a reinforcement. Researchers chose cassava starch as the matrix because cassava starch has good binding properties so that when we mix it with fiber, it can be bound well to produce a better biocomposite. Cassava starch has the advantages of good tenacity, viscosity and permeability, and suitable for use as a natural polymer that can be degraded because of its good degradation ability [11,12].

Specifically, this work explores the novelty of chemical surface modification via alkalization, systematically examining the combined effects of NaOH concentration, immersion time, and treatment temperature on the tensile performance and biodegradability of the

biocomposites. Shang et al. [13] Stated that alkalization treatment with heating parameters alkali base NaOH, holding time 1 hour, and heating temperature 90°C showed the same results. more effective by increasing the flexural and tensile strength values by 22.3%. Although practical packaging items like egg trays require resistance to compressive and impact forces during transport, tensile strength and elastic modulus provide foundational insights into the structural integrity, flexibility, and fiber-matrix interfacial bonding of the composite. Evaluating these tensile and biodegradation properties serves as a essential preliminary step to establish the baseline material capability before proceeding to product-level testing such as compression or drop testing.

2. Materials and methods

1.1. Materials

Rice straw was obtained from agricultural waste in the Lumajang area of East Java, cassava starch (99% purity) was purchased from an Organic store, Natrium Hydroxide (NaOH) powder (99% purity) was produced by Asahimas Chemicals Co, Ltd., and liquid glycerol was purchased from a Pratama Abadi Chemicals store.

1.2. Preparation of Rice Straw Fiber

Rice straw is washed thoroughly with running water and dried in the oven until constant weight, then cut into small pieces measuring ~1 cm. The alkalization process is carried out according to the procedure with variations listed in Table 1. The ratio between rice straw and NaOH solution is 1:10. Alkalized rice straw fibers are cleaned with distilled water until the pH is neutral, then dried in an oven until constant weight. The dried rice straw fibers were ground with an ice blender at 10.000 rpm for 10 minutes. The fine fiber is stored in a plastic clip.

Table 1. Research data sample

Sample	Biocomposite Sample	NaOH	Time	Temperature
AK	A1	7%	45 minute	70-80°C
	A2	8%	45 minute	70-80°C
	A3	9%	45 minute	70-80°C
t	t1	8%	30 minute	80-90°C
	t2	8%	45 minute	80-90°C
	t3	8%	60 minute	80-90°C
T	T1	8%	45 minute	70-80°C
	T2	8%	45 minute	80-90°C
	T3	8%	45 minute	90-100°C

For specimen samples t2 and T2, the results are the same because the variation parameters used are the same. However, both specimens were tested individually (t2 and T2).

1.3. Biocomposites Preparation

The initial stage is weighing ingredients such as cassava starch, rice straw fiber, glycerol, and distilled water according to the mass fraction calculation used. The glycerol used is 10% of the mass of cassava starch and the distilled water used is 3:1 of the total biocomposite material. These ingredients were put into a glass beaker and stirred for 5 minutes at 200 rpm using a mixer. The

hot plate is turned on at a temperature of 60°C, then the ingredients are heated and stirred again at 200 rpm for 30 minutes until they gelatinize. The finished dough is then poured into a glass mold, then dried in the oven at a temperature of 50°C until the weight is constant.

The finished specimens were cut and shaped in accordance with ASTM D638 type IV for tensile testing and ASTM D6868 for biodegradable test. The specimens tested contained nine types of samples. The number of specimens made for one variation was three specimens, so the total number of specimens for tensile and biodegradation testing was 27 specimens.

1.4. Tensile Test

Tensile testing was carried out to determine the ultimate tensile strength, tensile modulus, and elongation at break values of the rice straw fiber/cassava starch biocomposite. This test uses a Universal Testing Machine model HT 2402 with a crosshead speed of 1 mm/minute carried out at room temperature. The tested specimens were formed according to ASTM D638 type IV as shown in Fig. 2.

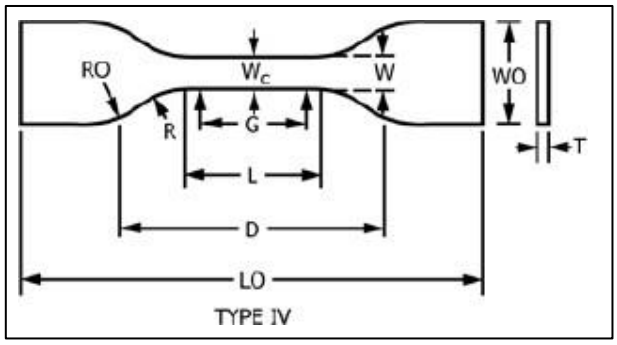


Fig. 2. ASTM D638 tipe IV [14]

1.5. Biodegradable Test

The biodegradation test of biocomposite was carried out in soil (pH 6) (soil burial test). The biodegradation samples were cut in a size 2 cm x 2 cm. There are 3 samples for each variation when soil burial test. The samples were buried 5 cm deep from the soil surface at open environment. The length of the time what was carried out in this test was burial for 15 days with weighing specimens every 3 days.

1.6. Macro Observation

Macro observations of the fracture surface of the specimen after the tensile test and biodegradable test were carried out using a Digital Microscope to determine whether or not there were cavities in the biocomposite and the shape of the fracture indicating fiber pull-out or fiber breakage. Macro observations were carried out using 70 x magnification.

3. Results and Discussion

3.1 Tensile Testing

Tensile testing in this research was carried out to determine the properties of the rice straw fiber/cassava starch biocomposite such as ultimate tensile strength, tensile modulus, and elongation at break. Graphs of the tensile test results, such as the ultimate tensile strength, tensile modulus, and elongation at break values, are shown in Figs. 3, 5, and 7 respectively.

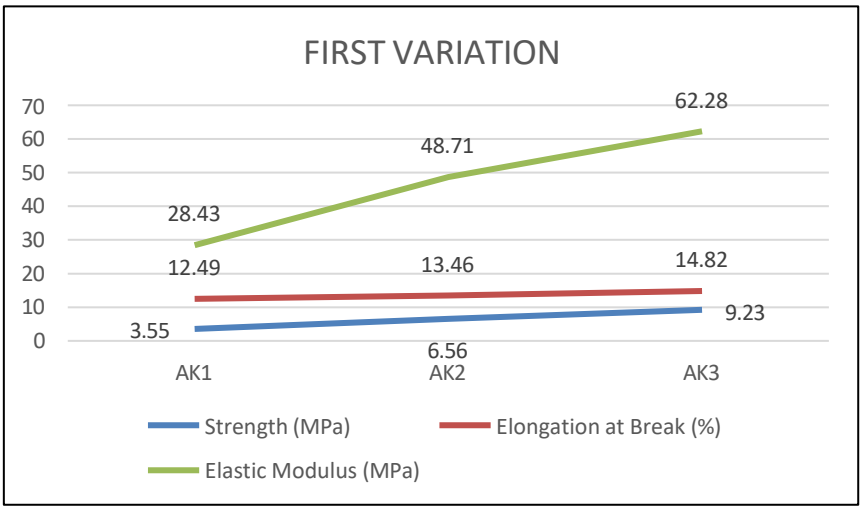


Fig. 3. Results of the First Variation Tensile Test

Fig. 3 shows a graph of the tensile test values which increase with increasing concentration of NaOH given. The stress value, strain, and the highest modulus of elasticity was found in the third treatment (A3) at 9.23 MPa. The matrix bonding and better reinforcement creates increasing strain values as it increases the concentration of NaOH given. A3 shows the highest strain value at 14.82%. The value of the modulus of elasticity in the variation The first also experienced in each treatment. The highest modulus of elasticity value was found in the third treatment was 62.28 MPa. This modulus value determines the level of stiffness of a material. The greater the value of the elastic modulus, the greater the stiffness of the material. The A3 specimen has the highest tensile test value, the following is a picture of the microstructure of the A3 specimen shown in Fig. 4.

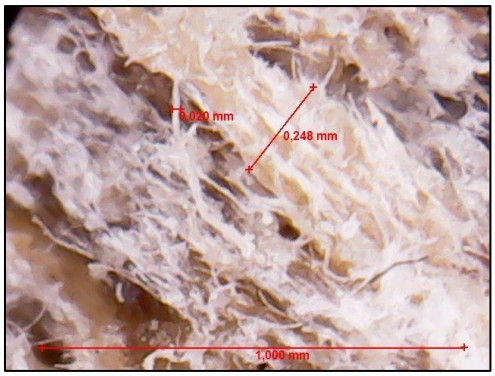


Fig. 4. Microstructure of Specimen A3

This shows that the greater the concentration given, the greater the content. lignin and other impurities contained in the fiber become degraded, thus increasing the interaction between the fiber and the polymer matrix [15].

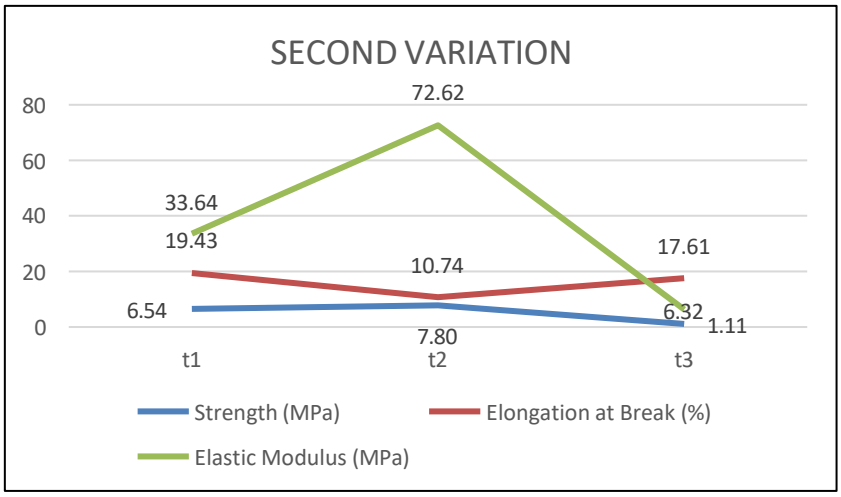


Fig. 5. Results of the Second Tensile Test Variation

Fig. 5 shows a graph of the values from the tensile test which fluctuate as the boiling time given increases. The tensile stress in this second variation increased during the first treatment second and there was a decrease between the second and third treatments. Other factors contributing to the difference in tensile strength values are several defects such as cracks, debonding, and cavities in the matrix [16]. The highest tensile stress value was at The second variation is found in specimen t2 at 7.80 MPa. This occurs because at the time The alkalization process results in the degradation of lignin and other impurities so that the bonds between the matrix and the fiber becomes good and increases the tensile strength value (Fig. 6a).

The strain value has a graph that fluctuates with increasing time given. In the first and second treatments the strain graph experienced decline. This is inversely proportional to the tensile stress value in the first treatment and secondly, the bond between the matrix and fibers actually increases the tensile strength. However, increasing the tensile strength value makes elastic deformation not evenly distributed as evidenced by the decreasing strain value (8.69%). Apart from that, another factor that causes the tensile modulus value to decrease is the presence of defects such as porosity that occur in the biocomposite during fabrication [17]. The second and third treatments experienced an increase in strain values (6.87%).

The elastic modulus value increases and decreases with time. increasing the boiling time given. In the first and second treatments the modulus value elasticity increased (38.98 MPa). This shows that the more Increasing the boiling time makes the material stiffer. However In the second and third treatments, the modulus value decreased (66.3 MPa). Alkalization leads to over-delignification and partial degradation of the cellulose microfibrils, damaging the structural integrity of the rice straw fibers and reducing their stiffness and increase modulus elasticity (Fig.

6b).

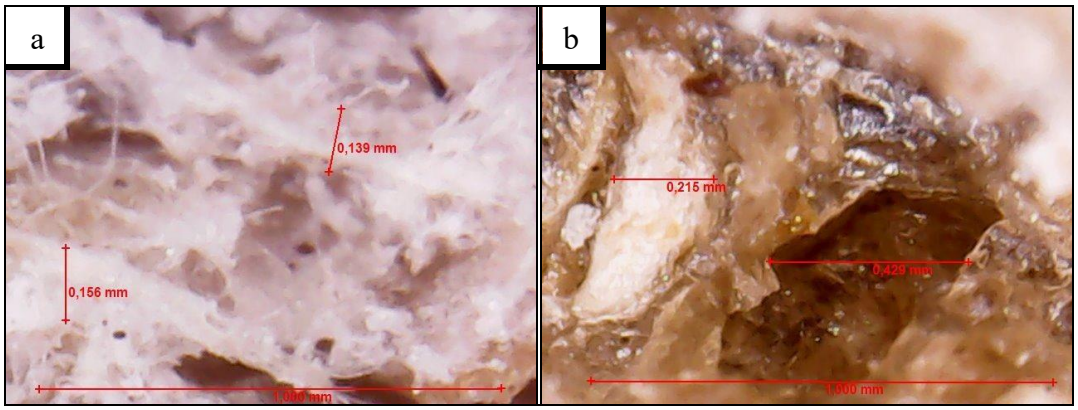


Fig. 6. Microstructure of Specimen t2 (a) and Specimen t3 (b)

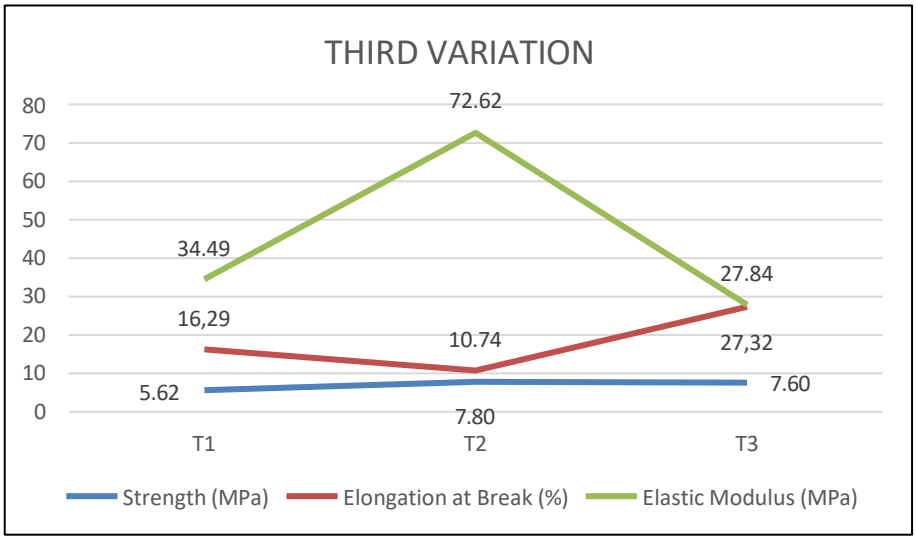


Fig. 7. Results of the Third Tensile Test Variation

Fig. 7 shows a graph of the values from the tensile test which fluctuate as the given temperature increases. The smallest tensile stress value is found at first treatment (5.62 MPa). This can occur due to a lack of uniformity in fiber distribution so that the specimen experiences agglomeration and this creates a value tensile stress becomes small [18] (Fig 8a).

The strain value has a graph that fluctuates with increasing temperature. In the first and secondtreatments, the strain graph decreased. This is inversely proportional to the tensile stress value in the first and second treatments. The bond between the matrix and fiber does increase the tensile stress value. Figure 8b illustrates that increasing the tensile strength value creates unevenly distributed elastic deformation as evidenced by the decrease in the strain value (5.55%). The second and third treatments experienced an increase in the strain value (16.58%).

The modulus of elasticity value fluctuated with increasing temperature. In the first and second treatments, the modulus of elasticity value increased (38.11 MPa). This indicates that the longer the boiling time, the more cavities formed, and the low interfacial interaction between the rice straw fibers and the cassava starch matrix, so that the biocomposite became brittle and the

elongation at break value obtained was lower [19]. However, in the second and third treatments, the modulus value decreased (44.78 MPa).

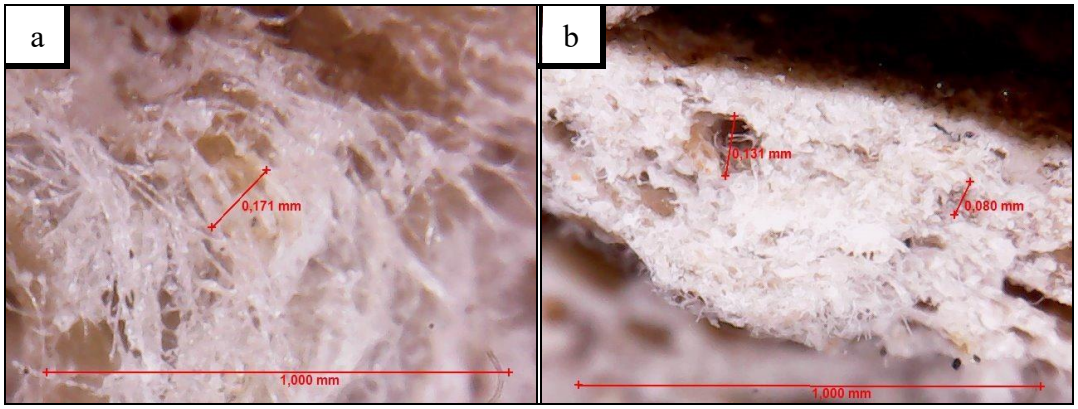


Fig. 8. Microstructure of Specimen T1 (a) and Specimen T3 (b)

3.2 Biodegradable Testing

Biodegradability tests are carried out to measure the extent to which a specimen can be degraded. naturally in certain environments. To The degradation rate can be seen in Fig. 9.

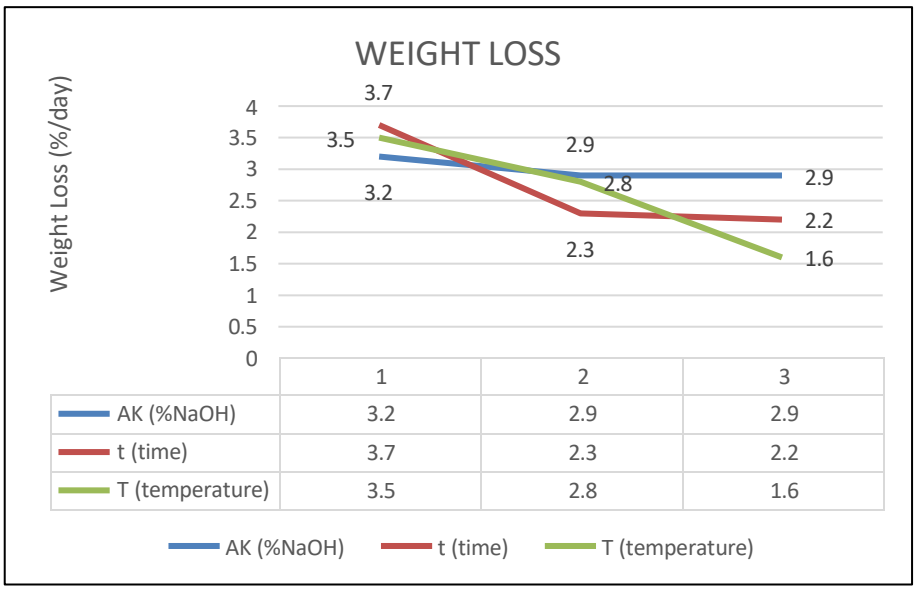


Fig. 9. Degradation Rate Graph

The image above explains the relationship between mass loss (Y axis) and the three treatments (X axis). The biodegradation behaviour of the fabricated composites was assessed by conducting a weight loss study [20]. Based on Fig. 9, it shows that the greatest degradation rate is found in the second variation. with the first treatment (t1) which was 3.7%/day. Fig 10a, shows that in the specimen contains scratches and holes. This indicates that microorganisms in the specimen the land can work and develop well.

The smallest degradation rate value was found in the third variation with the third treatment. degradation in the specimen was 1.6%/day. Fig. 10b illustrates that the treatment This

causes the fiber distribution to be uneven, so the starch tends to bind with glycerol. The addition of Glycerol also affects the rate of degradation. Because glycerol acts as a plasticizer, microorganisms contained in the soil have difficulty penetrating the existing specimens [21]. And this This causes the rate of degradation to decrease.

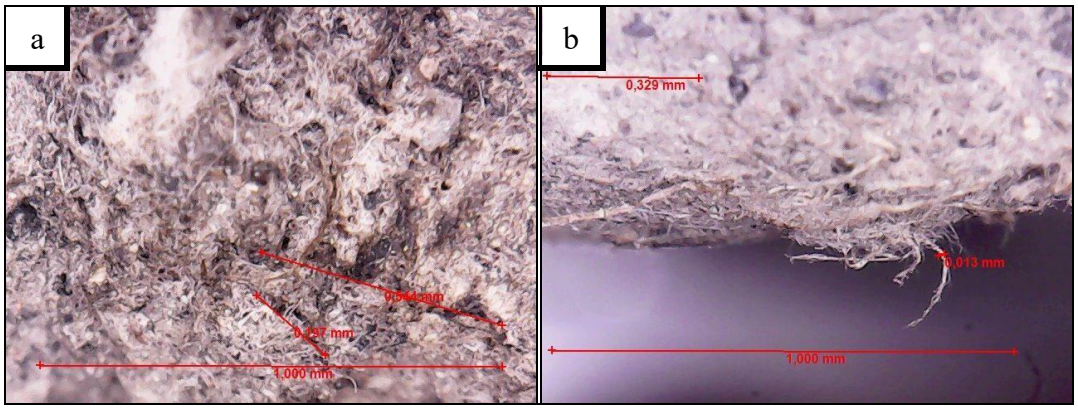


Fig. 10. Microstructure of Biodegradation Test of Specimen t1 (a) and Specimen T3 (b)

4. Conclusions

Based on the research results of the effect of the alkalization process on the mechanical properties of biocomposites straw as a molding material for egg trays, shows that the alkalization treatment process carried out on straw fiber can increase the tensile strength value of rice straw biocomposites cassava starch matrix.

Tensile testing was carried out to determine the ultimate tensile strength, tensile modulus, and elongation at break values of the rice straw fiber/cassava starch biocomposite. The highest tensile strength value is found in the first variation with the third treatment (A3) is by providing a NaOH concentration of 9%, time Boiling for 45 minutes and a boiling temperature of 70-80°C. The tensile strength value is 9.23 MPa.

In the biodegradation test result using soil as a burial medium. The length of the time what was carried out in this test was burial for 15 days with weighing specimens every 3 days. It was seen that the tested specimens experienced degradation and could proven by the damage to the specimen which is marked by the presence of scratches or even damage test specimens after burial. The highest degradation rate values were found in specimens with variation of boiling time with the first treatment (t1). The degradation rate value is 3.7%/day.

Metallographic testing was conducted through observation to determine the microstructure of each specimen. Each specimen was observed to determine the condition between the matrix and the reinforcement. Furthermore, defects in the specimens were visible through microscopic observation, thus supporting the results of this study.

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