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The Influence of Ultrasonic Wave Treatment on the Mechanical Properties of Nanoclay-reinforced Starch Biocomposite

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Abstract. The cassava as a starch source has a great potential as biocomposite matrix in a packaging material technology. The addition of nanoclay as biocomposite reinforcement into matrix will increase the biocomposite properties but it is difficult to homogenize a nanoclay particle into the biocomposite matrix. Accordingly, the research aimed at showing the effect of ultrasonic wave treatment on the strength of starch-based biocomposite with nanoclay as reinforcement. The research was conducted by applying a casting technique to synthesize the biocomposite. The ultrasonic wave treatment was conducted during biocomposite synthetic process with 5% (wt/wt) nanoclay with various homogenizing time and subsequently pour it into the mold. Biocomposite strength was determined by a tensile tester. The fracture morphology was observed using SEM and the functional group was analyzed using FTIR methods. The results show that the ultrasonic wave treatment affects the mechanical properties of nanoclay reinforced starch biocomposite. This result shows that the duration time of the ultrasonic wave treatment 15, 30, 45, and 60 min. increase strength and elongation of 11.79 MPa, 14.77 MPa, 20.52 MPa, 28.01 MPa, and 12.08%, 16.08%, 26.12%, 27.90%, respectively. It shows that the duration time of the ultrasonic wave treatment ≥ 45 minutes resulted in better mechanical properties of biocomposite.

Keywords: Nanoclay, biocomposite, cassava, ultrasonic wave, mechanical properties

1. Introduction

A global problem occurring in the worldwide is a plastic waste. Annually, over 300 million tons of plastics were consumed in the worldwide and about 50% of this volume was for disposable applications [1,2]. Especially in Indonesia, the disposal of plastics was 14% of total waste, about 5.4 million tons, in 2015 [3]. This waste was a potentially serious problem for the ecology and environment because of difficulty degraded and non-renewable [4].

In general, every industrial sector uses plastic because it has several advantages namely lightweight, practical, flexible, and the price is relatively cheap. The negative impact of the use of plastics is the waste caused by plastics is generally not easily destroyed by soil microbes. As a result, there is an accumulation of waste and become the cause of environmental damage [5]. Bioplastics are the solution



to decrease the plastic waste because it is able to be degraded into the environment for two months by microorganisms [6].

The starches are potential natural polymers for replacing the synthetic plastics. Due to the third largest producer of cassava in the worldwide with about 26 million tons by 2014 [7], Indonesia utilizes cassava as a potential source of starch for bioplastic raw material. Bioplastics have advantages such as wide availability, low cost, flexible, transparent, no taste, no smell, resistant to O₂, semipermeable against CO₂, and can be degraded without bringing out the toxic compound [8][9]. Usually, bioplastic has poor mechanical properties (tensile strength and elastic modulus) [10]. Many ways were conducted to improve the mechanical properties by using reinforcement/filler such as carbon nanotubes, carbon nanofibres, metal oxides, and nanoclay and dispersed it into bioplastic [11].

In the synthesis process of starch, biocomposite was performed by the addition of reinforcement such as nanoclay with no appropriate process tends to agglomerate. They tend to increase the biocomposite porosity [12] which decreasing mechanical properties [13]. The problem is how to make reinforcement such as nanoclay to be a uniform distribution and avoiding agglomeration in the biocomposite. We describe the study of the effect of ultrasonic wave treatment during the biocomposite synthesis on the strength of nanoclay reinforced starch biocomposite.

2. Methods

2.1. Materials

The raw material was the commercial cassava starch obtained from the district of Malang, East Java, Indonesia. Nanoclay was provided by the Sigma Aldrich.

2.2. Biocomposite synthesis

The synthesis methods of biocomposites were conducted in accordance with methods from [14][15]. Glycerols, 1.5% (v/v), were added in 98.5 mL distilled water and stirred for 5 min at 900 rpm then 5% (wt/wt) nanoclays were added into the solution. The solution was heated up to 80°C for 45 min while stirred under 900 rpm. The ultrasonic wave was applied to the solution with various duration treatment of 15, 30, 45, and 60 min at 300W and 20 kHz. After the homogenization process, 5.0% (wt/v) cassava starch was dissolved into the solution and heated at 80 °C for 30 min. on a hot plate. The solutions were poured in the mold then dried at 70°C for 4 h in an oven then kept in the dry box.

2.3. Strength measurement

The biocomposite strength measurement was conducted using the Fiber Tensile Tester (Techno Lab. Indonesia) with a maximum load capacity of 50 N. The sample dimension follows the ASTM D882 standard. Biocomposite film sample (60 x 5 mm²) were clamped in the grips in a range of 50 mm and pulled by a velocity of 0.025 mm s⁻¹ until the break.

2.4. Surface morphological observation

The morphology of the biocomposite surface was observed using the Scanning Electron Microscope (SEM) (FEI, Inspect-S50 type) at 10.00 kV. Prior to the SEM process, the specimens were coated with a gold layer with the thickness of 10 nm (SC7-620 Emitech sputter coater) [15].

2.5. FTIR analysis

FTIR of nanoclay reinforced biocomposite was performed using the KBr pellet technique. Samples weigh of 0.1 mg were mixed with an infrared-grade of KBr powder of 1 mg then were ground into powder and pressed into a pellet. The FTIR spectra were scanned and recorded in the Shimadzu IR Prestige-21 within the range 400 to 4000 cm⁻¹ with the scan step of 2 cm⁻¹.

3. Results and Discussion

3.1. Morphology of biocomposite

The biocomposite surface morphology observed with an optical camera and SEM are shown in Figure 1. The bioplastic made of starch show a clear transparent (Figure 1A) and after additional nanoclay, biocomposite was reduced its transparency (Figure 1B). The untreated of the ultrasonic wave, biocomposite morphology showed the agglomeration of nanoclay (Figure. 1C) after treatment process for 60 min, the biocomposite surface became rough. The nanoclays were uniformly spreading in biocomposite (Figure 1D). The biocomposite structure was changed with the presence of nanoclay through the intercalated or exfoliated mechanism in starch-layered silicate [16].

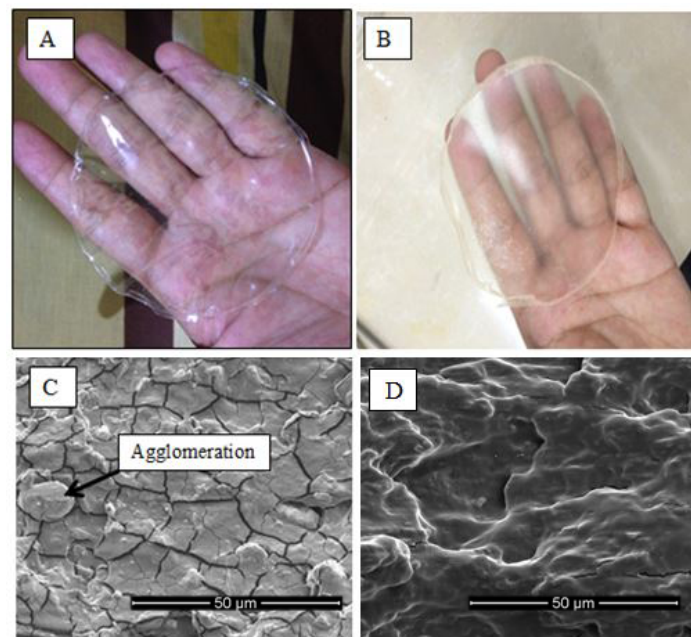


Figure 1. The surface morphology of biocomposite: (A) bioplastic starch; (B) Nanoclay-reinforced starch biocomposite; (C) Biocomposite without ultrasonic wave treatment; (D) Biocomposite with ultrasonic wave for 60 min.

3.2. Functional groups analysis

The observation using infrared spectra (IR) was performed using a Shimadzu Fourier Transmitter Infra-Red (FTIR). The nanoclay reinforced biocomposite spectra are shown in Figure 2. The result of the identification of functional group shows that there is no significant change among treatment of biocomposite with nanoclay. The shifting of several wave numbers and changes in the intensity of the absorption bands indicates the interaction between the polymer and nanoclay. The formation of $C = C$ in the region of $1400\text{--}1600\text{ cm}^{-1}$ [17] was more smooth at increasing the ultrasonic wave treatment. There was a change of curvature which was the valley at the region of $2000\text{--}2500\text{ cm}^{-1}$ indicating a vibration of the triple bond and the fingerprint region at $1500\text{--}600\text{ cm}^{-1}$ [18]. After the ultrasonic wave treatment of 60 min, this valley was disappeared. It indicates the interaction between bioplastic and nanoclay. The molecular interaction between polymers and nanoclay was able to produce two kinds of biocomposites namely intercalated and exfoliated biocomposites. The intercalated biocomposites are formed by the penetration of the polymer chains into the regions among the clay layers, forming alternating layers of polymer and clay or an ordered multilayer structure. The exfoliated biocomposites result from the extensive polymer penetration [19].

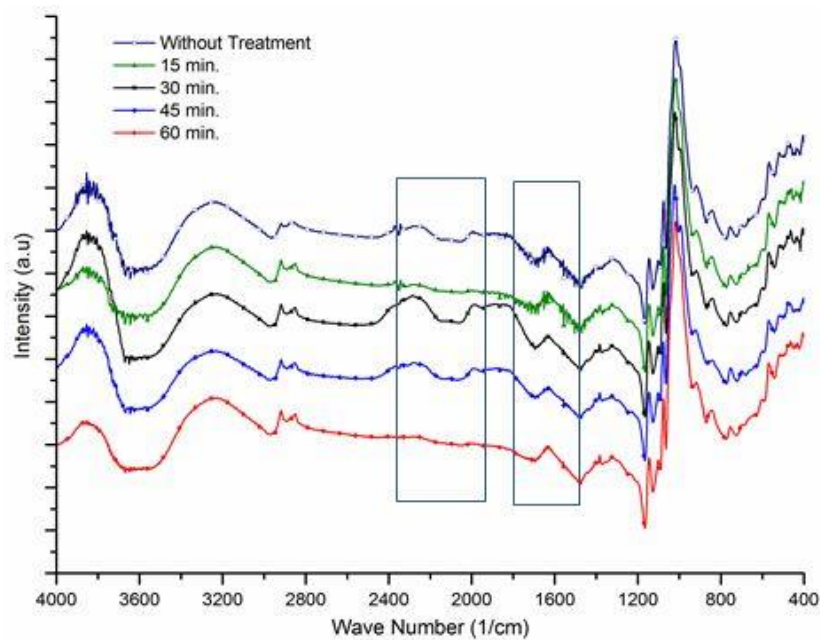


Figure 2. FTIR analysis of biocomposite

3.3. Strength of biocomposite

The mechanical properties of biocomposites are the main characteristics that have an important role in the application. The mechanical properties consisting of tensile strength and percent displayed at break and elasticity [20] are shown in Figure 3.

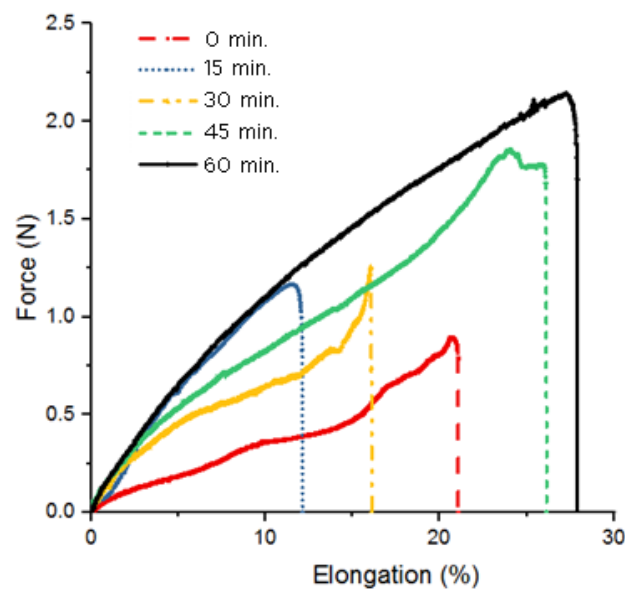
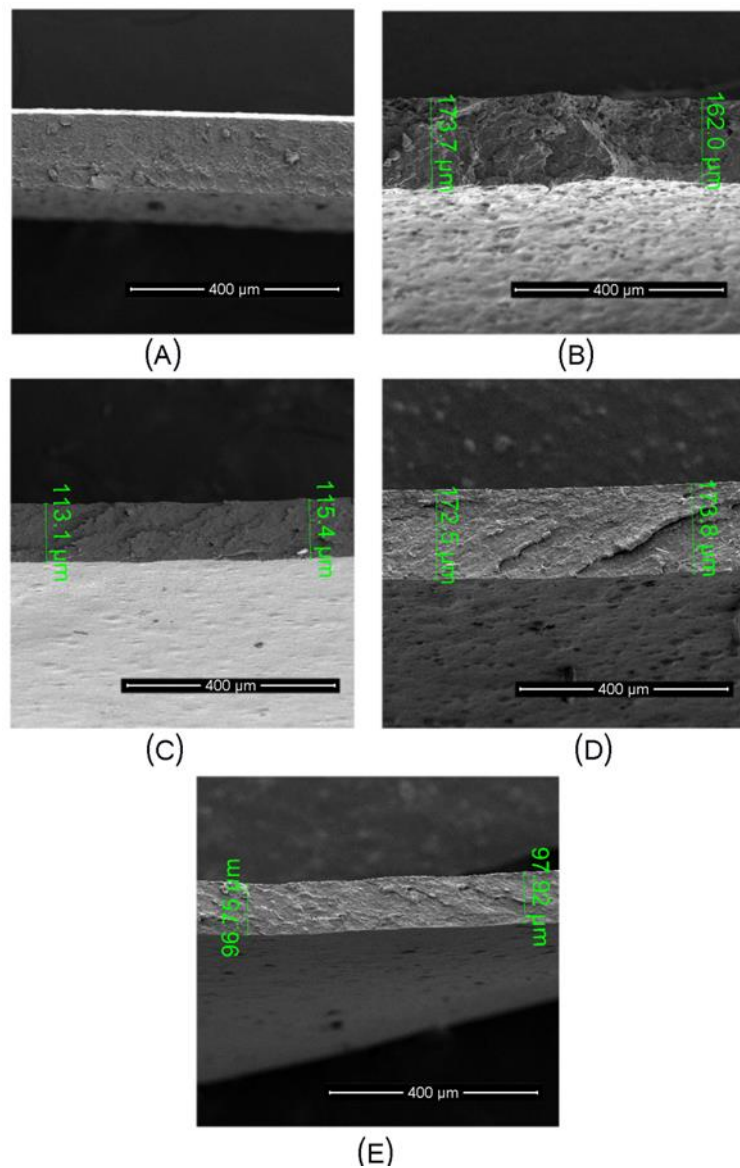


Figure 3. Force-elongation curve of biocomposite starch-nanoclay

Table 1. Results of the tensile test of biocomposite

Ultrasonic treatment (min.)	Average sample thickness (mm)	Maximum force (N)	Elongation at break (%)	Tensile strength (MPa)	Elastic modulus (MPa)
0	0.200	0.8584	21.04	8.95	42.5
15	0.198	1.1676	12.08	11.79	97.6
30	0.170	1.2553	16.08	14.77	91.8
45	0.181	1.8571	26.12	20.52	78.6
60	0.153	2.1434	27.90	28.01	100.4

**Figure 4.** Biocomposite surface fracture after the tensile test: Biocomposite without treatment (A); Treatment by Ultrasonic wave for 15 min (B); 30 min. (C); 45 min. (D); 60 min (E)

Tensile strength represents the composite material capability to receive a tension or load without any damage or broken which is expressed with a maximum tension before breaking that is the ultimate tensile strength. The tensile strength of the composite material can be influenced by several factors such as the compatibility of reinforcement into the matrix material, uniform distribution of reinforcement in the matrix, the relative ratio between the reinforcement, and the matrix materials in the composite materials. The elongation at sample break refers to the maximum change of film length while obtaining tensile strength until the film breaks, compared to the initial length.

Ultrasonic energy spreads nanoclay into biocomposite to make biocomposite has a uniform structure. Variations in a treatment time of 0 min., 15 min., 30 min., 45 min., and 60 min. results the elastic biocomposite with strain of 21.04%, 12.08%, 16.08%, 26.12%, 27.90%, respectively. Uniform distribution of the filler in the composite matrix is important for the production of bioplastics to increase its workability and flexibility. The addition of treatment duration during ultrasonic wave treatment affects the strength of nanoclay reinforced starch biocomposite. The variations of treatment duration of the ultrasonic wave of 15 min., 30 min., 45 min., and 60 min. produce biocomposites with a strength of 11.79 MPa; 14.77 MPa; 20.52 MPa; 28.01 MPa, and elastic modulus of 42.5 MPa, 97.6 MPa, 91.8 MPa, 78.6 MPa, and 100.4 MPa, respectively (Table 1). The exfoliated and partially exfoliated structure of biocomposite leads to a substantial increase the biocomposite elasticity [21]. The longest time of the ultrasonic wave treatment makes the molecule becomes homogeneous and well dispersed in the solvent or matrix [5]. The improvement of both bioplastic elongation and strength can be related to effectively interfacial adhesion between nanoclay fillers and starch matrix, through the intercalation and exfoliation mechanism of bioplastic in nanoclay, as describe in FTIR results.

The change of the mechanical properties of biocomposite was proved through the morphology of the tensile test fracture, as shown in Figure 4. Biocomposite without treatment shows several nanoclay agglomerations. It means nanoclay could not distribute uniformly at the biocomposite matrix (Figure 4A). After treated with ultrasonic wave for 15 min, the nanoclay agglomerate decreased and show the smooth surface (Figure 4B). The increasing duration of ultrasonic wave treatment, the rougher the surface of biocomposite (Figure 4C-E). It means there is a good interaction bond in the biocomposite so that the biocomposite has a crack retardation mechanism causing the increase of the tensile strength.

4. Conclusion

The duration time of the ultrasonic wave treatment during biocomposite synthesis enhanced the strength of the biocomposite. The ultrasonic wave treatment for 60 minutes resulted in high mechanical properties with an elongation percentage of 28% and tensile strength of 28.01 MPa.

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