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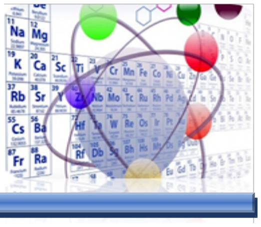

The Improvement of Mechanical Properties of PCL/Microcellulose Biocomposites from Corn Cobs by Adding PCLacac as a Compatibilizer

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ABSTRACT

Biocomposites have wide potential in eco-friendly packaging, agriculture, and biomedical applications such as scaffolds and drug delivery. This study uses poly(ε-caprolactone) (PCL) as a biocompatible, flexible matrix and microcellulose from corn cobs as a renewable, abundant, and low-cost filler. The PCL–microcellulose combination is expected to improve mechanical properties while adding value to agricultural waste. To enhance compatibility between matrix and filler, short-chain synthetic PCL (PCLacac) was added at 0%, 2.5%, 7.5%, and 10%. Serving as a compatibilizer, PCLacac promotes interaction with cellulose hydroxyl groups. The prepared biocomposites were molded according to ASTM D638 standards and characterized. Tensile results showed that the addition of 2.5% PCLacac gave the highest increase in tensile strength. FTIR analysis confirmed hydrogen bonding between PCL carbonyl and cellulose hydroxyl groups, indicated by absorption band shifts. Thus, optimal PCLacac addition (2.5%) enhances mechanical performance and transforms low-value corn cob waste into high-value biocomposites.

Keywords: biocomposite, cellulose, compatibilizer, corn cob, PCL

1. INTRODUCTION

Biocomposites are innovative materials composed of natural matrices and reinforcements, one of which comes from agricultural waste.[1] These biocomposites can be used for various applications, from eco-friendly packaging and agriculture to medical applications such as tissue scaffolds and drug delivery systems. Compared to their constituent components, biocomposites exhibit improved mechanical and thermal properties, including tensile strength, stiffness, and heat resistance, as well as biodegradability.[2]

The use of natural materials as fillers in biocomposites offers alternative renewable raw materials, such as corncobs, rice stalks, bamboo fiber, pineapple leaves, coconut fiber, empty oil palm bunches, and palm fiber.[3] Furthermore, utilizing natural materials from Indonesia can also increase the economic value of agricultural waste.

Currently, the development of biocomposites based on natural fillers has been widely carried out. This is because these natural fillers can improve the mechanical and thermal properties of biocomposites. Furthermore, various types of polymers such as polycaprolactone, polypropylene, poly(lactic acid), and epoxy have also been widely used as biocomposite matrices.[4] The presence of natural materials as fillers will create interfacial interactions through hydrogen bonding between the polymer matrix and the filler in the biocomposite. If the filler is evenly distributed in the composite, this can alter molecular mobility.[5]

However, the main obstacles in the manufacture of biocomposites are inconsistent product properties and low adhesion between hydrophilic natural fillers and the generally hydrophobic polymer matrix.[6] Furthermore, variations in the quality of raw materials limit the application of biocomposites. Therefore, innovations in processing technology, filler modification, the use of compatibilizers, and material standardization are needed so that the resulting biocomposites can improve the quality compared to fillers and matrices.

Previously, our research group has successfully synthesized short-chain PCL (PCLacac) using bis(acetylacetonato)Zr catalyst.[7,8,9] PCLacac can be used as a compatibilizer to overcome the low adhesion between filler and matrix. The presence of PCLacac is expected to improve interfacial interactions, so that the filler distribution becomes more even and has an impact on improving the mechanical properties and thermal stability of biocomposites. On the other hand, several researchers have also developed microcellulose as a natural material filler so that it can be distributed homogeneously in the biocomposite matrix, so that the risk of agglomeration can be minimized.

This research will develop a biocomposite using a PCL matrix and microcellulose filler sourced from natural corncobs. PCLacac is added to the matrix as a compatibilizer to improve interfacial compatibility and adhesion between the PCL matrix and the microcellulose filler. This is expected to produce a biocomposite with improved mechanical properties.

2. EXPERIMENTAL

2.1. Chemicals, Equipment, and Instrumentation

The equipment used in this study were *a hot plate* (Thermo Scientific Cimarec SP88857105), Fourier Transform Infra-Red (FT-IR) (Bruker Invenio-S), tensile test with Instron 2710-004 instrument, analytical balance, beaker glass, glass funnel, thermometer, Erlenmeyer, 200 mesh sieve.

The materials used in this study were commercial PCL (CAPA 6800), PCLacac[9] short chain, corn cobs obtained from corn plantations of residents in the Tanjung Morawa area, Deli Serdang. Meanwhile, the materials for cellulose isolation were as follows: acetic acid (CH_3COOH) 99% (Sigma-Aldrich), sodium chlorite ($NaClO_2$) 80% (Sigma Aldrich), sodium sulfite (Na_2SO_3) 99% (Merck), sodium hydroxide (NaOH) 97% (Merck), concentrated sulfuric acid (H_2SO_4) 95-98% (Merck), distilled water.

2.2. Research Procedure

2.2.1 Corn Cob Sample Preparation

Corn cobs obtained from local plantations are thoroughly washed to remove sand and other debris. They are then dried, cut into approximately 2 cm pieces, and blended. Afterward, they are sieved using a 200-mesh sieve to obtain a finer grain size. the more refined.

2.2.2 Isolation of Microcellulose from Corn Cobs

The initial process of microcellulose isolation involves bleaching. Ten grams of corncobs are added to a 0.7% sodium chlorite ($NaClO_2$) solution and heated for 2 hours at 70-80 °C. The precipitate is then filtered and washed with distilled water until a neutral pH is achieved. This process is repeated until the sample is white. Next, the drying process in an oven continues until white fibers are produced.

In the next stage, delignification, purification, and hydrolysis processes are carried out. White corn cob fibers are added to a 5% sodium sulfite solution and then heated for 2 hours at a temperature of 70-80 ºC. Next, the fibers are filtered and washed several times with distilled water and then dried in air. After that, the fibers are soaked in a 17.5% sodium hydroxide solution for 2 hours. This process produces alpha cellulose which is then hydrolyzed in a 64% sulfuric acid solution for 45 minutes at a temperature of 45 ºC. Then, cold water is added to stop the reaction. Next, it is filtered and dried in an oven at a temperature of 60 ºC to produce microcellulose.[10]

2.2.3 Preparation of PCL/PCLacac Biocomposite with Microcellulose Filler

The initial stage of biocomposite formation was carried out by mixing PCL/PCLacac dissolved in 25 mL of chloroform at 60 °C. The composition of PCLacac added was 0%, 2.5%, 5%, 7.5%, and 10%. Next, 0.02 grams of microcellulose was added. After that, the mixture was refluxed for 1 hour at 300 rpm. Then, the mixture of PCL/PCLacac and microcellulose filler from corn cobs was poured into a glass mold and dried at room temperature until a film was formed. The specimen was molded according to the ASTM D638 standard, then tensile testing was carried out. The biocomposite was then characterized using an FT-IR instrument [11]. The ASTM D638 dimensions are shown in Figure 1.

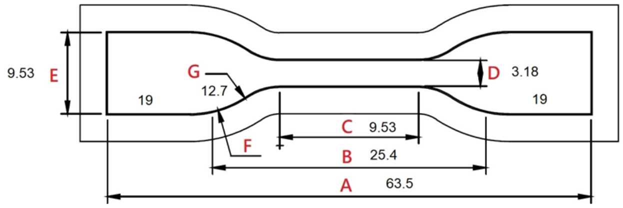

Figure 1. Tensile test specimens size based on ASTM D638

3. RESULTS AND DISCUSSION

3.1. Mechanical Properties Analysis

Mechanical property analysis was performed using a tensile test. This test aims to measure the strength of a material under tensile load, including parameters such as maximum tensile stress, breaking strain, and modulus of elasticity. The tensile test results are shown in Figures 2 and 3.

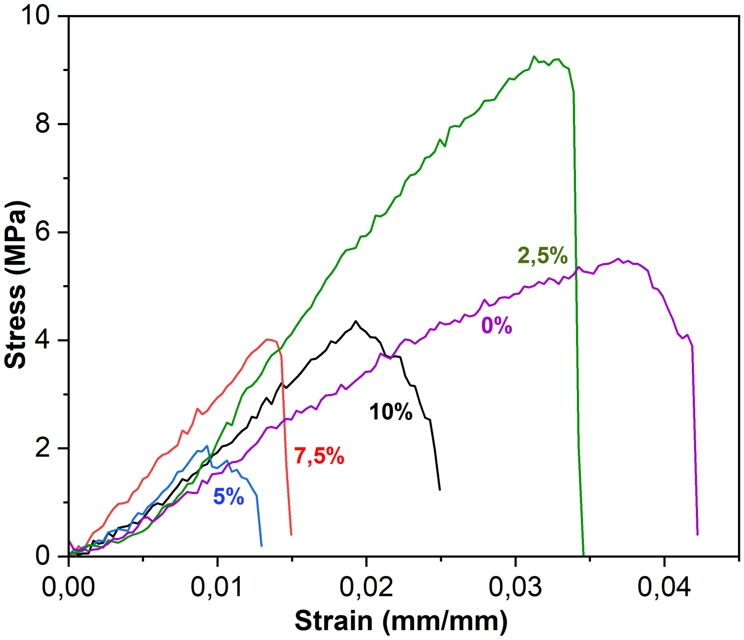

Figure 2. Tensile test results of biocomposite

Figures 2 and 3 show the results of the tensile test of the biocomposite. The resulting biocomposite consists of a mixture of commercial PCL, synthetic PCL (PCLacac), and microcellulose. In this study, variations in the addition of PCLacac to the PCL matrix were carried out with compositions of 0%, 2.5%, 5%, 7.5% and 10%. Based on the tensile test results as shown in Figures 2 and 3, it can be seen that variations in the PCLacac ratio produce varying tensile strength and elongation values as well as different levels of compatibility. The most optimum mechanical properties of the biocomposite are seen in the 2.5% PCLacac composition with a tensile strength value of 9.25 MPa, a maximum strain of 0.0345 and an elongation of 2.07%. Meanwhile, the biocomposite with a 0% PCLacac composition produces a tensile strength value of 5.51 MPa, a maximum strain of 0.0422, and an elongation of 2.53%.

Meanwhile, the addition of PCLacac to the PCL matrix of 7.5% and 10% resulted in lower tensile strength values of 4.00 MPa and 4.35 MPa. While the maximum strain was 0.0149 and 0.0249 with elongation of 0.89% and 1.49%. Furthermore, the addition of PCLacac of 5% resulted in the lowest tensile strength value among all biocomposite compositions of 2.05 MPa , the maximum strain was 0.0129 with elongation of 0.77%.

The low tensile strength and elongation values with PCLacac additions of 5%, 7.5%, and 10% are likely due to the uneven homogeneity of the resulting mixture. Consequently, the PCL particles are not well dispersed and form separate domains (aggregates), thus becoming weak points when the material is pulled. Furthermore, physical interactions at the interface are weakened, reducing the biocomposite's ability to withstand loads and deformation. Consequently, the biocomposite will be more susceptible to fracture and its elongation will be reduced.

Based on the data shown in Figure 2 and 3, it was found that the addition of PCLacac to a matrix with a low composition (2.5%) resulted in higher tensile strength. This is because PCLacac with a composition of 2.5% is more easily dispersed evenly in the biocomposite matrix. The impact is that there is no formation of aggregates (separated phases) that can weaken the tensile strength value of the biocomposite. In addition, PCLacac particles are better distributed so they are able to withstand and spread tensile energy evenly. On the other hand, physical interactions through hydrogen bonds or Van Der Wall's forces between PCLacac and the PCL matrix and microcellulose become stronger, thereby increasing tensile strength without significantly reducing elasticity.

In addition, the tensile test results also produce a modulus of elasticity (MoE) value, which is a measure of the stiffness of the biocomposite, as shown in Figure 3b. After passing the elastic limit, the biocomposite can undergo permanent (plastic) deformation, then experience maximum elongation until it finally breaks (fracture). The MoE value is usually obtained from the slope of the stress-strain graph in the elastic region (the initial part of the tensile test curve).

Observations of the MoE value showed that the addition of 2.5% PCLacac produced the highest value compared to other variations. This composition increases the intermolecular bonding within the PCL/PCLacac and cellulose filler, resulting in a stiffer structure that better withstands elastic deformation. This provides an optimal balance between flexibility and stiffness.

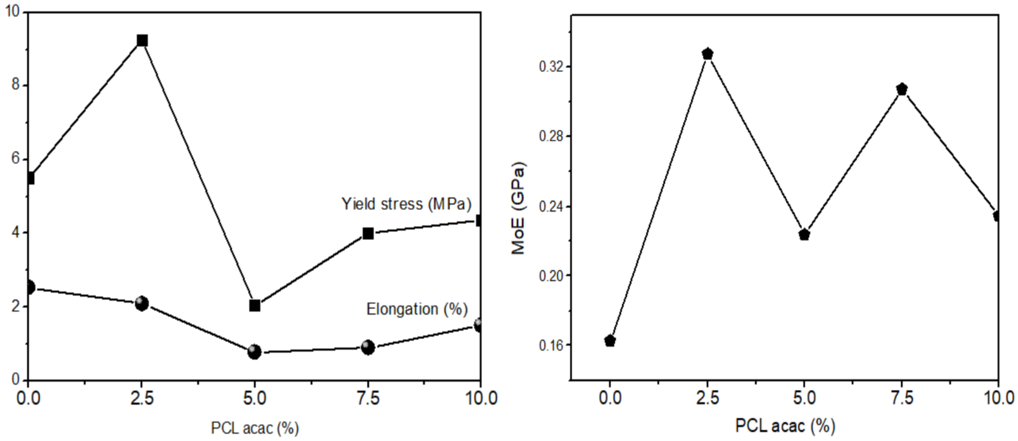

Figure 3. (a) Tensile strength and elongation of biocomposites (b) MoE of biocomposites

3.2. FTIR Analysis

FTIR analysis was performed to identify chemical functional groups and intermolecular interactions in biocomposites by detecting infrared radiation absorption by molecular bonds. Figure 4 shows the results of FTIR analysis of the PCL/PCLacac, microcellulose filler, and biocomposite.

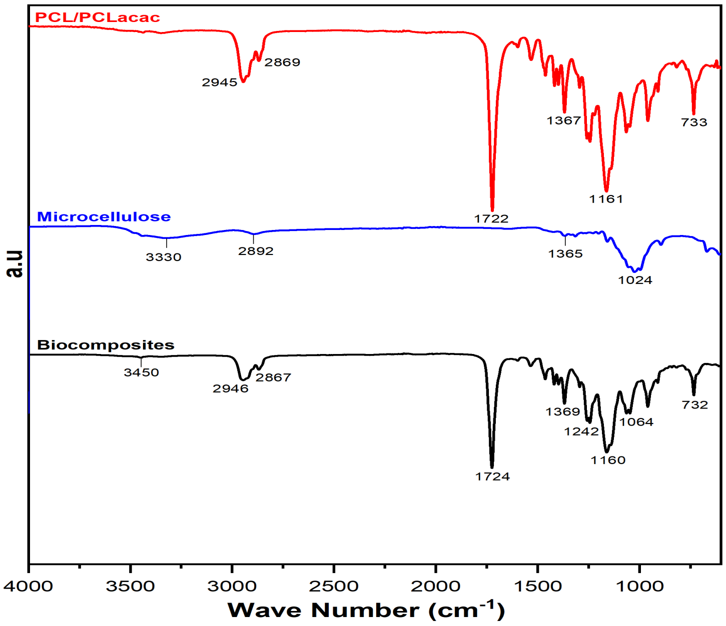

Figure 4. FTIR spectra of (a) PCL/PCLacac, (b) Microcellulose, and (c) Biocomposite

Based on the FTIR analysis of the PCL/PCLacac as shown in Figure 4a, absorption bands at 1722 cm^{-1} and 1367 cm^{-1} were identified as carbonyl groups (C=O) and C-C groups. These two groups are indicated to be ester groups of PCL. Furthermore, another absorption band at 1161 cm^{-1} was identified as the vibration of the ether group (–COC–). On the other hand, absorption bands at 2945 cm^{-1} and 2869 cm^{-1} were identified as asymmetric and symmetric stretching vibrations of the methylene group (–CH_2–). This is reinforced by the presence of an absorption band at 733 cm^{-1} originating from the methylene group (–CH_2–). The FTIR spectrum results of the PCL/PCLacac mixture show an absorption pattern that is similar to the spectrum of pure PCLacac.[9] This similarity shows that the addition of PCLacac to PCL does not significantly change the polymer framework due to the similarity of the basic structure of both, namely the polyester main chain.

Meanwhile, figure 4b shows the FTIR analysis of microcellulose. The absorption band at 3330 cm^{-1} was identified as the presence of a hydroxyl group (-OH) indicating the hydrophilic nature of microcellulose. Subsequently, the absorption band at 2892 cm^{-1} was identified as the presence of a symmetric vibration of the methylene group (–CH_2–). Another absorption band at 1024 cm^{-1} identified the presence of an ether vibration (–COC–). Finally, the absorption band 1365 cm^{-1} was identified as the presence of a C-C group.[11]

On the other hand, Figure 4c presents the FTIR analysis of the biocomposite. In the absorption band of 3450 cm^{-1}, the presence of a hydroxyl group (-OH) was identified, which is a typical characteristic of microcellulose. The presence of this polar group indicates the presence of hydrogen bonds between the PCL/PCLacac mixture and the microcellulose filler. Furthermore, the absorption band of 1724 cm^{-1} was identified as a carbonyl group (C=O). Then, the absorption bands of 1369 cm^{-1} and 1160 cm^{-1} were identified as the presence of C-C and CO groups. These groups indicate the presence of PCL ester groups as a biocomposite matrix. Another absorption band at 1064 cm^{-1} was identified as the presence of ether vibrations (–COC–). On the other hand, the absorption bands at 2946 cm^{-1} and 2867 cm^{-1} were identified as asymmetric and symmetric stretching vibrations of the methylene group (–CH_2–). This is reinforced by the presence of an absorption band at 732 cm^{-1} which comes from the methylene group (–CH_2–).

Based on the FTIR analysis results, it can be indicated that the PCL/PCLacac mixture has physically interacted with microcellulose. This is evidenced by the absence of absorption of new functional groups outside the PCL and microcellulose components. Furthermore, the shift in the intensity of carbonyl and OH groups in the FTIR spectrum of the biocomposite indicates that PCL has interacted with microcellulose. This interaction likely occurs through hydrogen bonding between the carbonyl groups of PCL and the hydroxyl groups of microcellulose.

4. CONCLUSION

Tensile test results showed that adding PCLacac significantly improved the mechanical properties of the biocomposite. The addition of 2.5% PCLacac to the matrix resulted in optimal performance, with a tensile strength of 9.25 MPa, a maximum strain of 0.0345, and an elongation of 2.07%. This improvement was attributed to better dispersion, minimal aggregation, and stronger interactions between the PCL matrix and microcellulose. FTIR analysis also confirmed the occurrence of physical interactions, indicated by the absence of new absorption bands and shifts in carbonyl and –OH intensities. These results suggest hydrogen bonding enhanced interfacial adhesion, further supporting mechanical property improvement.

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