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RESEARCH ARTICLE
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Mechanical Properties of Biocomposite Films Based Polyvinyl Alcohol/Potato Starch Filled by Coffee Ground Waste

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ABSTRACT

Plastic is a ubiquitous material used to meet various human needs. However, its use negatively impacts the environment due to its non-biodegradability, contribution to pollution, and potential health threats. Biocomposite materials offer a promising solution to these problems. This study aimed to develop biocomposite films using a polyvinyl alcohol / potato starch matrix reinforced with coffee grounds waste as a filler. This experimental study involved three tests: tensile testing, fracture morphological observation, and biodegradation in soil. The addition of CGW to the PVA/PS matrix increased tensile stress, reaching a maximum of 9.42MPa at a 2% filler loading. This result corresponded with the tensile modulus, which also peaked at 2% CGW (0.28MPa). Fracture morphological analysis via scanning electron microscopy confirmed these findings, revealing wave-like patterns and strong interfacial bonding between the matrix and filler at this concentration. The lowest tensile stress (6.31MPa) was observed at a 3% filler loading. Strain values remained relatively consistent between pure PVA and the biocomposites. Biodegradation testing revealed degradation rates of 32, 34, 36, 39, and 37% for PVA, PVA/PS, and CGW loadings of 1, 2, and 3%, respectively, after 15 days of soil burial. The biocomposite films, particularly at the optimal CGW loading, exhibit competitive tensile stress and biodegradation rates compared to synthetic plastics, suggesting their potential suitability for food packaging applications.

Keywords: Tensile stress, Biocomposite, Polyvinyl alcohol, Potato starch, Coffee ground waste

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INTRODUCTION

While plastics are ubiquitous in modern society, fulfilling a range of consumer demands from packaging to single-use items like shopping bags, their persistence in the environment presents significant challenges. The non-biodegradable nature of conventional plastics contributes to environmental pollution, exacerbates flooding due to drainage blockage, and poses potential risks to human health (Acquavia et al., 2021; Kibria et al., 2023). Therefore, one of the efforts to overcome this problem is to switch to biocomposite materials. Biocomposite have several advantages in replacing synthetic plastics such as environmentally friendly, low prices, abundant availability, and can be used as compost (Sadasivuni et al., 2020; Al Amin et al., 2023). However, it has a disadvantage such as lower mechanical and thermal properties than synthetic plastics (Neves et al., 2020). To overcome this problem, other components are needed to improve the properties of biocomposite such as natural fillers.

Biocomposite is defined as a multi-phase material, where the biopolymer is mixed with two or more fibers or particles from nature as a reinforcement which results increasing the mechanical properties (Zwawi, 2021; Ilyas & Sapuan, 2020). Several matrices of biocomposite are usually used such as polylactic acid (PLA), chitosan,

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polyvinyl alcohol (PVA), starch, and others. From these matrices, PVA is one of the biocomposite matrix that has several advantages namely soluble in water, resistance to chemical, and good film forming (Zulkiflee and Fauzi, 2021). Due to its properties, PVA has received attention for its ability to decompose in nature in a relatively short period of time (Begum et al., 2019). However, it has some drawbacks such as the high price. Therefore, mixing with starch is a solution to reduce the production cost of the biocomposite (Asrofi et al., 2019).

Study on PVA matrix with a mixture of starch has been investigated by previous researchers, for example the mixture of PVA and starch. In this study, the addition of starch into PVA reduce mechanical properties. This phenomenon due to the bad interface PVA and starch. Furthermore, starch has hydrophilic properties (Asrofi et al., 2019). To overcome this problem, the addition of natural fillers is a solution to improve the properties without reducing the biodegradation value. This is evidenced by a study conducted by previous researchers which showed that fillers from nature were able to improve the mechanical properties of the PVA/Starch matrix. This is due to the good hydrogen bonding between the fillers in the matrix (Sreekumar et al., 2019).

Referring to the previous research, this study uses a PVA and potato starch as with coffee grounds waste. The use of potato starch as a mixture in the PVA matrix in this study was motivated by the previous study that potato starch can dissolve large amounts of amylose resulting easily soluble in water in a short time (Han et al., 2019).

In the current study, natural fillers such as coffee grounds waste have been prepared to improve the properties of the biocomposite. The selection of coffee ground waste as a filler in biocomposites because coffee is one of the most abundant garden products in Jember Regency, Indonesia. According to previous study, the coffee grounds waste is the main residue after coffee has been brewed. Millions of tons of coffee grounds residue consist of several elements, namely water, cellulose, lignin, hemicellulose, fat, ash, protein, and aliphatic acid. If the coffee grounds waste is not controlled properly, it can cause a polluted environment because the decomposition process of coffee grounds requires large amounts of oxygen (Lessa et al., 2018). Therefore, the use of coffee grounds as a biocomposite filler aims to reduce coffee grounds waste and is expected to be able to obtain added value from coffee grounds waste.

This study examines the effect of the addition coffee grounds waste to the PVA and potato starch matrix. This study presents the mechanical properties of the PVA and potato starch as matrix without and with the addition of coffee grounds waste as filler. These biocomposites were tested using tensile test (mechanical properties), scanning electron microscopy (fracture morphology), and soil burial tests (biodegradation). The results of this study are expected to find new types of biocomposites to substitute synthetic plastic packaging.

MATERIALS & METHODS

Materials

Polyvinyl Alcohol (PVA) with a viscosity of 49.2 cps and a degree of alcoholysis of 87.58 mole% was obtained from Chang Chun Petrochemical Co., Ltd. Potato starch (PS) was obtained from plantation products in Jember Regency. Coffee grounds waste (CGW) was obtained from coffee processing waste at local coffee shops in Jember Regency. Other chemicals were obtained from a chemical shop in Jember Regency, Indonesia.

Preparation of Coffee Grounds Waste (CGW)

CGW was dried using drying oven for 24 hours at 105℃. The dried CGW was treated with alkalization with a 1% NaOH (0.25 M) solution for 24 hours at room temperature. Then, it was neutralized with distilled water to pH 7. After the alkalization process, the CGW was dried in an oven for 24 hours at 105℃. Finally, it was sieved the coffee grounds using an 80-mesh sieve.

Synthesis of PVA/Potato Starch/CGW Biocomposite

PVA was mixed with distilled water by weight ratio of 1:10. Then, 6% of PS and glycerol in a ratio 1:5 was mixed in a beaker glass. CGW was added as much 1% (CGW1), 2% (CGW2), 3% (CGW3) with a ratio of CGW to starch of 1:100. Besides that, PVA and distilled water were mixed using hot plate magnetic stirrer at 90℃ at 500rpm for 60min until gelatin. The starch, glycerol, and CGW were added and stirred into PVA solution at 70℃, 400 rpm for 40 minutes to produce biocomposite gelatin. The biocomposite gelatin was then poured into a rectangular glass mold. It was dried using drying oven at 40℃ for 24 hours. Finally, the biocomposite film was cut according to ASTM D-882 standard as reported by previous study (Asrofi et al., 2020).

Tensile Test

All biocomposite specimens were prepared for tensile test. The tensile test was carried out using the Universal Testing Machine HT-2402 tensile machine. Three samples of each variation were tested and mean values were reported. The test was conducted at room temperature.

Scanning Electron Microscopy (SEM)

SEM was carried out after the sample undergoes a tensile testing process. The SEM test was carried out using a machine with the Hitachi 3400 N series. This test was carried out on the fracture surface of the biocomposite sample. The observation was done at voltage and magnification for 1 kV and 2000x, respectively.

Soil Burial Test

The biodegradation test was carried out using compost soil containing 25% Nitrogen, 7% Phosphorus, 9% potassium, 3.7% Iron, 55.3% other nutrients, and pH 6. Biocomposite specimens were cut to a size of 20×20mm and then buried in the soil on depth of 5cm from the top of the soil. The biodegradation test was carried out with variations in burial for 0, 5, 10, and 15 days.

RESULTS & DISCUSSION

Tensile Stress

The tensile stress curves of PVA/PS/CGW biocomposite containing different ratio of CGW as fillers into PVA/PS matrix are shown in Fig. 1. It shows that the addition of starch to the PVA matrix reduce the tensile stress of the biocomposite from 8.29MPa to 7.08MPa. This is supported by previous research which showed that the PVA tensile stress value of 31.21MPa decreased to 9.54MPa when starch was added to the PVA matrix (Mittal et al., 2020). This is because starch has amorphous properties. The decrease in the value of tensile stress is also supported by previous study which shows that the addition of starch into the PVA matrix reduce the tensile stress caused by a decrease in the density of hydrogen bonds in the polymer matrix (Musa & Hameed, 2020). When CGW are added to the biocomposite, it increases the tensile stress value. The result of the highest tensile stress lies in the variation of the CGW by 2% for 9.42MPa. This is supported by previous report which conducted research on PVA/Potato Starch biocomposite with nanocrystalline cellulose (CNC) filler with a variation of 3-20% (Noshirvani et al., 2018). It is known that the value of the tensile stress increases with the addition of the mass fraction of the filler. It can be indicated that the increase in the tensile stress value is caused by hydrogen bonds between the filler and the polymer matrix which causes the polymer chain to have limited mobility thereby increasing the stiffness of the composite (Noshirvani et al., 2018; Nurazzi et al., 2021).

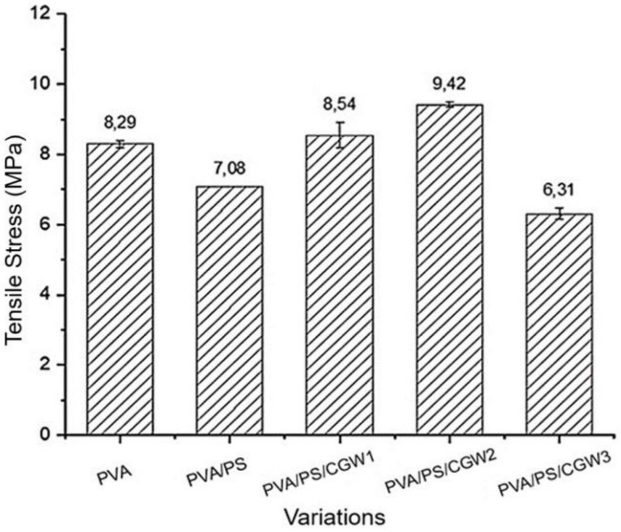

Fig. 1: Tensile stress of all biocomposite tested

The increase in the tensile stress value in the 2% CGW variation was strengthened by the results of morphological observations with SEM which showed a compact structure and coastline to produce good interfacial bonds between the matrix and filler which could increase the tensile stress of biocomposite samples (Asrofi et al., 2018). With the addition of 3% CGW, it showed a decrease in the tensile stress value to 6.31MPa. These results are similar with previous report regarding biocomposites with PVA matrix and kenaf fiber filler (Arumugam et al., 2020). This study resulted in a decrease in tensile stress along with the addition of fiber into the matrix. This occurs due to poor adhesion between the matrix and the fiber, causing the tensile stress to decrease with the addition of the fiber mass fraction (Bharath et al., 2024). The decrease in the tensile stress value is also evidenced by the results of morphological observations with SEM which show cracks and poor interfacial bonds (bad adhesion) which reduce the tensile stress value of biocomposite samples (Syafri et al., 2019).

Fig. 2 shows that the strain at break has an insignificant difference along with the addition of CGW as filler in the matrix. The highest value is found in the 2% filler variation with a value of 33.335% and the lowest value is in the 1% filler variation with a value of 32.072%. However, in the 2% filler variation, there was a very small increase in the strain value, which was 1.263%. From the Fig. 2, it can be concluded that along with the addition of the mass fraction of the filler, the CGW can increase the strain at break value.

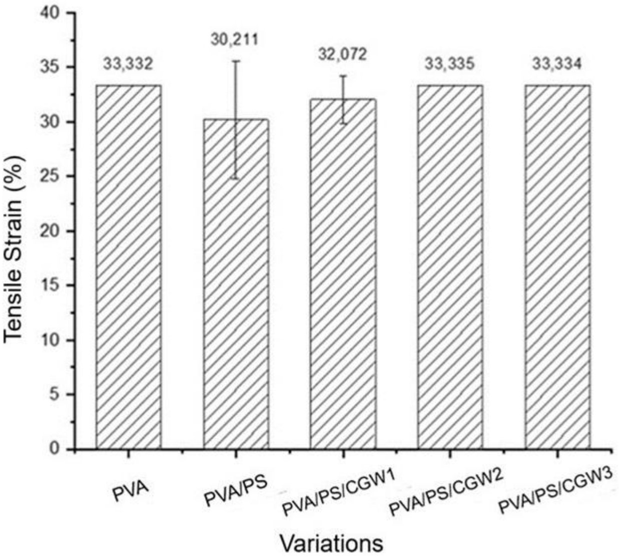

Fig. 2: Tensile strain of all biocomposite tested

This result is also evidenced by research conducted by previous study which conducted research on mixing PVA/starch as a matrix and natural fiber as a filler with a variation of 10-40% (Mallick et al., 2020). The research results show that the higher the mass fraction of rice husk fiber in the biocomposite produces a high strain value. The highest yield was shown in the variation of 40% rice husk fiber with a value of 300.02%. This happens because of the good compatibility between the fiber and the matrix. The presence of hydroxyl groups in natural fibers makes it compatible with the PVA/starch matrix. In addition, the increase in strain value was caused by an increase in the crystallinity of cellulose as the variety of natural fibers increased (Mallick et al., 2020). This result is also evidenced by research conducted by previous researcher who conducted research on mixing PVA with starch as a matrix and nanocrystalline cellulose as filler with a variation of 3-20%. The result of strain with the highest value is found in the fiber variation of 7% with a value of 71.1% (Noshirvani et al., 2018).

Fig. 3 shows that the value of the tensile modulus has a good upward trend along with the addition of the mass fraction of CGW to PVA/PS matrix. The tensile modulus on pure PVA has a value of 0.25MPa. When starch was added to the PVA matrix, the value of the tensile modulus decreased to 0.23MPa. However, the addition of CGW in this study proved to increase the value of the tensile modulus. The increase in the tensile modulus value occurs when the addition of CGW filler is 1% with a value of 0.27MPa and the addition of CGW is 2% with a value of 0.28MPa.

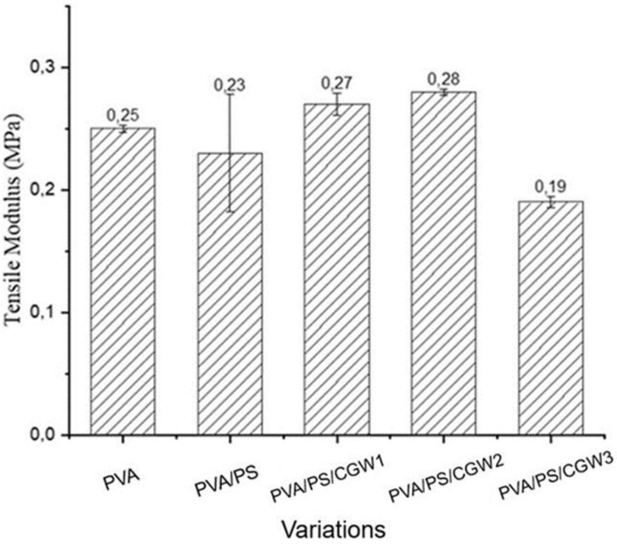

Fig. 3: Tensile modulus of all biocomposite tested

The increase in the value of the tensile modulus is supported by previous research which examined the effect of adding coir fibers to the PLA matrix on mechanical properties (Sun et al., 2017). This study showed that the addition of coir fibers to the PLA matrix resulted in an increase in tensile modulus. This occurs because the addition of fibers to the matrix causes the material to become stiffer due to the reduced mobility of the polymer chains with the addition of fibers (Sun et al., 2017).

Morphological Analysis

The biocomposite samples used in the SEM testing in this study were variations of pure PVA, PVA/PS/CGW2, and PVA/PS/CGW3 as shown in Fig. 4. This variation was chosen because the result of the highest tensile stress with a mixture of PVA/PS/CGW2 lies in the variation of coffee grounds by 2%. Meanwhile, the variation of 3% coffee grounds was chosen because it experienced a decrease in the value of tensile stress after the addition of fiber.

Fig. 4 (a-c) shows the results of the SEM test with a magnification of 2000x to determine the fracture surface of the biocomposite sample. Fig. 4a is the result of SEM testing on pure PVA biocomposite samples. The results showed that the pure PVA sample had a smooth surface which indicated that there was no filler component in the matrix. This result is evidenced by previous research which obtained pure PVA biocomposite samples having a smooth surface structure which indicates that the homogeneity of the PVA solution makes a good sample (Kansiz et al. 2024). Similar results were also shown in previous studies related to the phenomenon of smooth PVA structures without fillers (Mahardika et al., 2021).

Fig. 4b is the result of the fracture of the biocomposite sample with variations of PVA/PS/CGW2 which shows that there are wave lines and compact structures that are evenly distributed on the fracture surface of the biocomposite sample. The wave lines and compact structure are an indication that the filler is evenly distributed in the matrix so that there is no filler agglomeration which results in an increased tensile stress value. The compact structure and good fiber dispersion indicate a good interfacial bond between the matrix and filler so that the sample has an increase in tensile stress (Asrofi et al., 2018; Mahardika et al., 2021).

Fig. 4c is the result of SEM observations on the PVA/PS/CGW3 variation which shows that there are long cracks. Cracks are an indication of poor interfacial bonding between the matrix and filler. Poor interfacial bonding indicates that the filler is not homogeneous with the matrix due to the process of stirring the biocomposite solution, resulting in a decrease in the tensile stress value (Syafri et al., 2019).

Soil Burial Degradation Properties

Fig. 5 shows the rate of biodegradation of biocomposite samples with the effect of variations in the mass fraction of CGW by 1, 2, and 3%. The Fig. 5 displays the rate of weight loss increases with the addition of variations in the mass fraction of the CGW filler. The results showed that at 5-day burial, the samples showed weight loss values of 17, 22, 26, 26, and 28%, respectively. In pure PVA samples, the value of the biodegradation rate increased up to day 15. This happened because according to previous researcher, PVA has hydrophilic properties so that it can absorb water in the soil which can cause the sample to experience increased weight loss (Marinas et al., 2024).

On day 10, each biocomposite sample experienced an increased weight loss compared to day 5 with values of 24, 28, 30, 31, and 36%, respectively. The PVA/Starch biocomposite film sample showed that the addition of starch into the PVA matrix could increase the rate of degradation than the pure PVA sample. This happens because PVA has high hydrolyzability properties, causing resistance to soil burial degradation (Silva et al., 2023; Asrofi et al., 2023). Therefore, the addition of starch can increase the rate of degradation growth of microorganisms faster so that it helps the degradation process (Mallick et al., 2019). These results are also supported by previous research conducted which shows that along with the addition of the starch mass fraction into the PVA matrix, the degradation rate increases (Majeed et al., 2023; Asrofi et al., 2023).

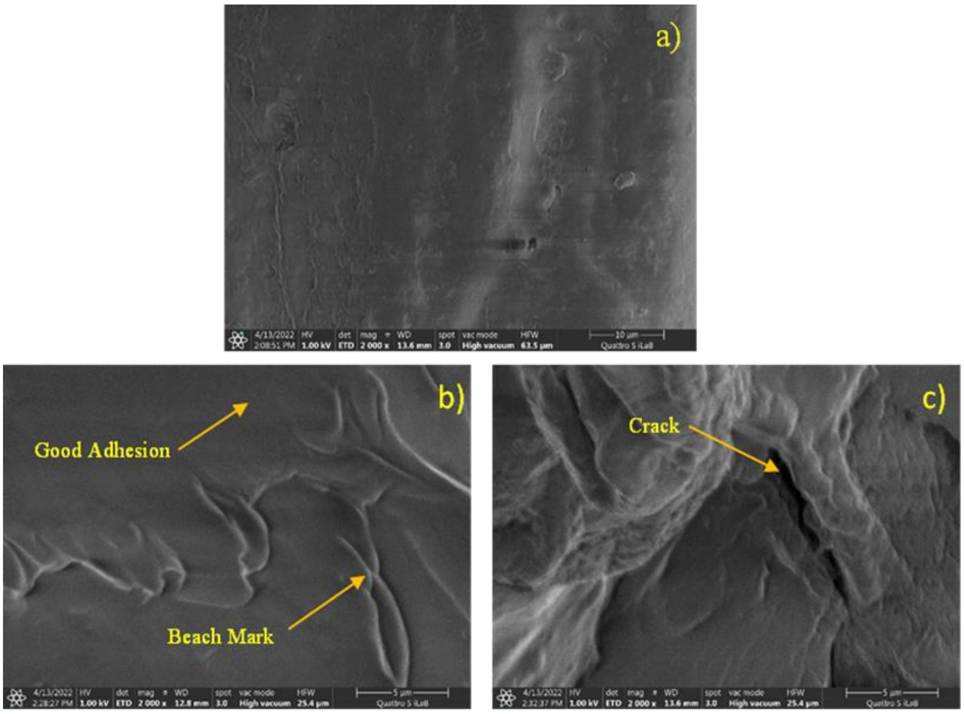

Fig. 4: Fracture morphology by Scanning Electron Microscopy (SEM): (a) PVA, (b) PVA/PS/CGW2, (c) PVA/PS/CGW3

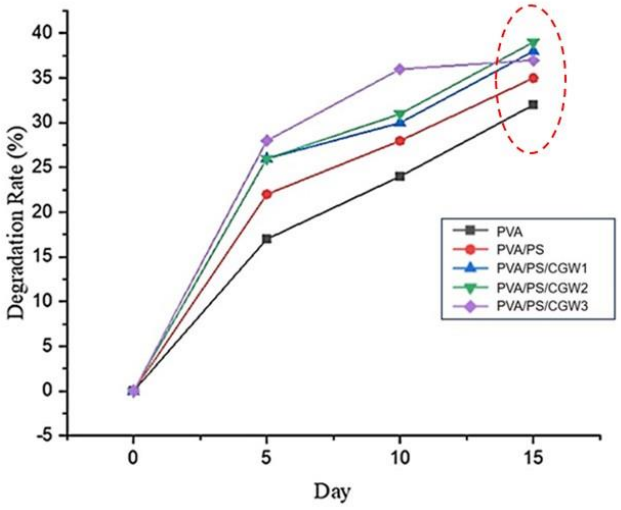

Fig. 5: Degradation rate of all biocomposite tested

On day 15, each biocomposite sample experienced an increase in the rate of degradation compared to day 10 with values of 32, 35, 38, 39, and 37%, respectively. The increase in the rate of degradation was caused by the addition of the mass fraction of CGW into the PVA/PS matrix. The results of this study are supported by previous report which shows that as the mass fraction of the fiber in the matrix, the degradation rate increases (Asrofi et al., 2023). This happens because the organic compounds in the fiber make the biocomposite film vulnerable to attack by microorganisms. This is also supported by previous study which states that the natural fiber used can affect the degradation process because the fiber that has been treated with alkalization has undergone a chemical process so that it can increase the rate of degradation (Sun et al., 2021).

Conclusion

In this study it can be concluded that the addition of the mass fraction of CGW filler increase the tensile stress of the biocomposite. The highest maximum value is found in the variation of the addition of CGW by 2% with a tensile stress value of 9.42MPa, strain of 33.335%, and tensile modulus with a value of 0.28MPa. This is due to the good interfacial bond between the matrix and filler which can increase the tensile stress of the biocomposite sample. Observation of morphology test used three samples, namely PVA, PVA/PS/CGW2, and PVA/PS/CGW3. The sample that has the best bond is found in the PVA/PS/CGW2 variation which indicates that there is a wave line which can indicate a good interfacial bond between the matrix and the filler, while the PVA/PS/CGW3 variation shows a crack which indicates that the bond is poor interface between matrix and filler. The addition of variations in the mass fraction of CGW filler can increase the rate of biodegradation in nature. The highest value of biodegradation rate was found in the variation of PVA/PS/CGW3 with an average weight reduction of 25% in a span of 15 days.

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Author Contributions

Mochamad Asrofi served as the research coordinator, providing overall guidance, research conception, and data analysis. Hilmy Dzaki Arisandi and Revvan Rifada Pradiza assisted in conducting the experiments and collecting data. Salahuddin Junus, Melbi Mahardika, and Putri Amanda contributed to the analysis of mechanical properties. R. A. Ilyas, M. R. M. Asyraf, and S. M. Sapuan provided the SEM and biodegradation data, and assisted with manuscript proofreading.

Conflict of Interest

The authors state no conflict of interest.

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