



Influence of lemon skin powder on the mechanical and thermal properties of polyvinyl alcohol (PVA)/cassava starch biocomposites prepared by solution casting method



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Abstract

More environmentally friendly alternative materials are needed to replace synthetic plastics to prevent environmental pollution. One biocomposite, a mixture of polyvinyl alcohol (PVA) and cassava starch, is an appropriate solution; however, its mechanical properties are low. Lemon skin, which is often considered waste, contains promising cellulose and phenolic extract content, making it a potential superior and functional filler for biocomposites, particularly in active packaging applications. This study presents an investigation into the use of lemon skin powder (LSP) as a filler for PVA- and cassava starch-based biocomposites. The biocomposites were prepared using the solvent casting method by varying the concentration of lemon skin powder filler, namely 0%, 1%, 3%, and 5%, with the final product being a film. Analysis was carried out on the mechanical properties, morphology (Scanning Electron Microscopy (SEM)), and thermal properties of the film. The results show that the mechanical properties of the film increased when LSP was added, with the highest tensile strength of 13.82 MPa for biocomposite films containing 5% LSP compared to the tensile strength of pure PVA and PVA/cassava starch blends. In addition, the thermal properties of biocomposites also increased at this content, as evidenced by an increase in the initial decomposition temperature. Specifically, the initial decomposition temperature of pure PVA, which was 204°C, increased to 214°C in biocomposites with 5% LSP. This improvement in thermal properties and tensile strength can be attributed to the strong interfacial bonding between the filler and the matrix, which contributes to the overall compact structure of the biocomposite. Morphological observations confirm the increased interface interaction and uniformity of filler distribution in the composite. These results confirm that lemon skin fibers have positive potential as reinforcements for PVA and cassava starch-based biocomposites, particularly in food packaging applications.

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INTRODUCTION

Environmental pollution issues caused by the use of synthetic plastics need to be urgently addressed [1]. One of the problems with synthetic plastics is their resistance to decomposition in

nature [2]. Therefore, environmentally friendly and sustainable alternative materials are needed. Biocomposites are a good candidate because, in addition to being based on environmentally friendly natural materials, the combination of two

or more materials can maintain their unique characteristics [3][4]. Biocomposites have a wide range of applications, from packaging and construction materials to supercapacitors [5]. There are several types used as biocomposite matrices, such as PVA (Polyvinyl Alcohol), Chitosan, and others [6]. Polyvinyl Alcohol has unique properties in its ability to form films, biodegradability, high resistance to oils and solvents, and gas barrier properties [7]. However, PVA has disadvantages such as low mechanical strength and a relatively high purchase price [8].

To overcome these weaknesses, combining PVA with natural starch is a solution to minimize production costs. Previous research has shown that adding jicama starch to the polyvinyl alcohol matrix results in a decrease in mechanical properties and an increase in water absorption [9]. This is due to the weak interface bond between polyvinyl alcohol and hydrophilic starch. Similar findings also reported a decrease in tensile strength with the addition of cassava starch. The best mixing composition of 80% polyvinyl alcohol and 20% cassava starch produced the highest tensile strength, but it was still lower than that of pure polyvinyl alcohol [10]. To overcome this, modifications such as the addition of cellulose filler from natural fibers are needed [11].

Several previous studies discussed PVA-based biocomposites and starch matrices with natural fiber fillers. One study has reported on the use of palm fiber with and without treatment in PVA (polyvinyl alcohol) and wheat starch biocomposites [12]. The results showed an increase in tensile strength and elastic modulus, with values of 12 and 245 MPa, respectively, with the addition of 9% palm fiber. These results were the highest values compared to the addition of fibers with other contents, although they were still lower than the control film. This improvement in mechanical properties was supported by strong hydrogen bonds at the interface, which provided a strengthening effect on tensile strength. Similar results were found with the addition of bacterial cellulose to PVA and cassava starch biocomposites [13]. The addition of 10 g of reinforcement increased the tensile strength by 215%. In addition, the thermal resistance of the biocomposite increased after the addition of 10 g. The decomposition at the addition of 10 g was 0.0109 mg/°C and without addition was 0.0120 mg/°C. The phenomenon of increased thermal resistance was also found in other studies. PVA and cassava starch biocomposites with date leaves as filler [14]. The addition of date leaves as filler increased the decomposition temperature from 240 °C to 290 °C. This proves that the

addition of natural fibers to the polyvinyl alcohol and cassava starch matrix can affect the tensile strength and thermal resistance of biocomposites.

Lemon skin is a waste product from the food processing industry that is not utilized effectively [14]. Lemon skin contains high levels of bioactive compounds such as phenolics and flavonoids, making it a potential functional ingredient with antibacterial, antioxidant, and UV protection properties [15]. It has even been reported that gram-negative bacteria (*Klebsiella pneumoniae*) are highly sensitive to lemon skin extract [16]. Lemon skin has been widely applied in various fields, such as carbon-based electrodes [17], nickel and cadmium removal biosorbents [18], and tissue engineering scaffolds [19]. Natural fibers such as lemon skin must undergo an alkalization treatment in order to be used as reinforcements in biocomposites [20]. In addition to its active properties, this waste contains 21.2% cellulose, 1.6% hemicellulose, 0.4% lignin, and 31% pectin [21], which has a positive role as a composite filler.

Different from other natural fibers, the cellulose and bioactive compound content in lemon skin offers a superior and active filler in biocomposites, especially those based on PVA-cassava flour. Therefore, the novelty of this study is the experimental approach to PVA and cassava starch biocomposites with lemon skin fiber filler. The lemon skin content added was based on previous studies on other natural fibers, such as 1%, 3%, and 5% volume fraction. The purpose of this study was to demonstrate the mechanical and thermal properties of PVA and cassava starch added with lemon skin powder (LSP) as a biocomposite filler. The results of this biocomposite research were expected to represent a biocomposite discovery that can become a new alternative material by utilizing unused lemon skin as a biocomposite filler, especially for food packaging applications.

MATERIALS AND METHODS

Materials

Polyvinyl Alcohol BP-26 (density: 1.19 g/cm³) was obtained from Chang Chun Petrochemical LTD. Cassava starch (density: 0.99 g/cm³) was obtained from a local shop in Jember, Indonesia. Lemon skin waste was obtained from a herbal and juice shop in Jember, Indonesia. Sodium hydroxide (NaOH) for alkali treatment was obtained from a local chemical store, CV Aneka Kimia, Jember, Indonesia.

Lemon skin powder Preparation

Preparation of the filler includes an alkali treatment scheme as shown in Figure 1. The

process begins by drying the lemon skin powder in an oven at 90°C at 2-hour intervals until its weight is constant. A 5% NaOH solution is prepared, and the lemon skin powder is added to the NaOH solution for 6 hours, then neutralized with distilled water to a pH of 7. The lemon skins were blended using a blender or mixer. The lemon skins were filtered using an 80-mesh sieve at the beginning of the process and a 100-mesh sieve at the end of the process. The resulting powder was that which could pass through the 80-mesh sieve and that which could not pass through the 100-mesh sieve, ensuring a particle size of approximately 148-177 µm. This fiber size was chosen to maximize its function as an ideal filler in composites, in accordance with previous studies. This fiber size offers advantages such as producing fewer agglomerates and bubbles, having color, mechanical properties, transparency, and light transmittance similar to conventional plastics when applied in composites [22].

Biocomposites Production

The process of making biocomposite films is illustrated in Figure 1. Starting from dissolving PVA using distilled water and then stirring at 90°C for 1 hour using a stirrer at 500 rpm until a gel is formed. Adding starch and then stirring using a stirrer at 60°C for 40 minutes at 400 rpm. Adding lemon skin powder filler according to each variation (0%, 1%, 3%, and 5%). Pouring the hydrogel solution into a glass mould measuring 190 mm x 120 mm x 3 mm and drying in an oven at 40°C for 24 hours. After drying, the specimens were cut according to the ASTM D882-18 standard and then tested. Table 1 shows the biocomposite composition.

Tensile Test

The tensile test was conducted using the ASTM D882 standard. The testing process was carried out using a Universal Testing Machine HT2402 with a capacity of 5 kN. The crosshead speed used was 10 mm/min. All sample variations were tested individually with 3 replicates.

Table 1. Biocomposite Composition

Sample Code	PVA (%)	CS (%)	LSP (%)
PVA	100	0	0
PVA/CS	80	20	0
PVA/CS/1LSP	80	19	1
PVA/CS/3LSP	80	17	3
PVA/CS/5LSP	80	15	5

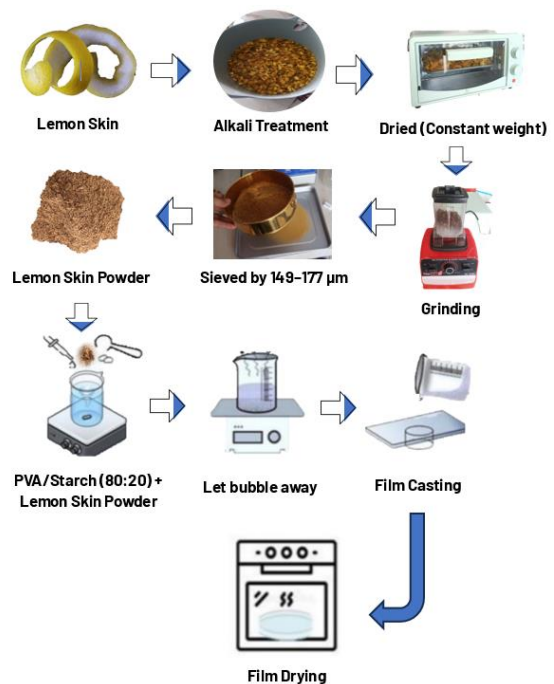


Figure 1. Biocomposites Production Scheme

Scanning Electron Microscopy (SEM)

Morphological testing of this biocomposite specimen using SEM (Scanning Electron Microscope) on a Hitachi SU3800 machine. This test was carried out to determine the morphology of the fracture of the specimen. Each variation was observed for fracture morphology. The magnification used was 800x with a voltage of 3kV.

Thermogravimetric Analysis (TGA)

In this study, Thermogravimetric Analysis testing was carried out to determine thermal resistance. Biocomposite specimens were tested at 30-500 °C. The heating speed was 10°C/minute

RESULTS AND DISCUSSION

Biocomposites Film

The results of the production of polyvinyl alcohol and cassava starch biocomposites with the addition of lemon skin filler at levels of 1%, 3%, and 5% are shown in Figure 2. The thickness of the biocomposites obtained ranged from 0.19 mm to 0.29 mm. The biocomposite specimens produced were approximately 13 cm long and 1 cm wide. It can be seen that, the plastic becomes slightly more yellowish with the increase in lemon skin powder filler.

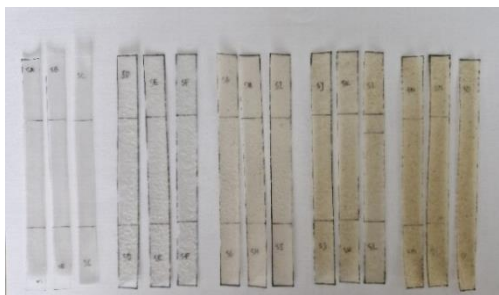


Figure 2. Polyvinyl Alcohol/Cassava Starch filled with Lemon Skin Powder Biocomposites

Tensile Properties

The addition of powder was expected to increase the tensile strength of this biocomposite research. As shown in Figure 3, the tensile strength of PVA decreases when starch is added. This phenomenon occurred due to poor interfacial bonding between PVA and starch [9]. The large number of freed OH bonds causes poor compatibility between PVA and starch [13]. This was confirmed by similar research on PVA and cassava starch biocomposites, where the addition of cassava starch to PVA decreased the tensile strength due to poor compatibility between PVA and starch and also reflects weak intermolecular hydrogen bonds between the two polymers [10]. In addition, another study reported a similar thing on the tensile strength of PVA films, which decreased by 327% when starch was added because starch has high molecular mobility [23]. This condition is supported by the SEM results in Figure 6(b), which show many cracks and gaps that indicate poor bonding between PVA and cassava starch, resulting in cracks.

The tensile strength value of the biocomposite increased as the percentage of lemon skin powder filler increased. The tensile strength of the addition of 1% lemon skin powder increased by 269%, from 3.07 MPa to 11.34 MPa. This phenomenon occurs due to good adhesion at the interface of the matrix and fiber [12]. The trend continued to increase with the addition of 3% and 5% lemon skin powder. The addition of 3% lemon skin powder produced a tensile strength of 12.74 MPa and increased again at the addition of 5% to 13.82 MPa. This result shows that the addition of 5% has the highest tensile strength. The addition of natural fiber fillers into PVA and starch causes the polymer chains to become less mobile due to the increased number of hydrogen bonds resulting from a higher percentage of fillers so that the fibers are dispersed in the matrix [13][24]. Therefore, the addition of fibers improves the tensile properties of biocomposite films [25][26].

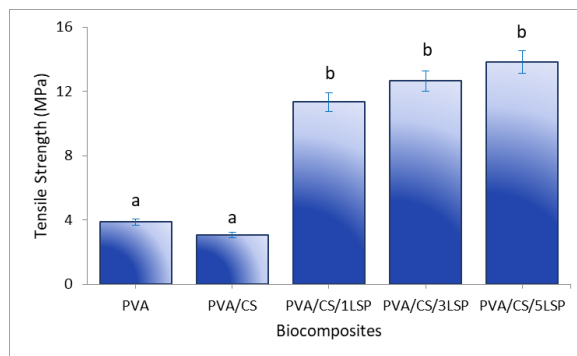


Figure 3. Tensile Strength of Biocomposites ($p < 0.05$)

This increase in tensile strength is confirmed by the observation of good adhesion between the fibers and the matrix in the morphological structure observed using SEM in Figure 6(e).

Figure 4 shows that the highest elongation value is found in the PVA variation, which has an elongation value of 790.67%. This phenomenon is in accordance with previous research, which shows the elongation value of pure PVA is 172% compared to after adding starch [8]. A similar study also reported that pure PVA has the highest elongation value of 140% compared to PVA added with starch and fiber [27]. However, it was found that the elongation value decreased when starch and fiber were added. This result is in accordance with research related to the addition of corn starch to PVA, which states that the decrease in elongation at break is caused by the decreased hydrogen bond density due to the presence of starch content [28]. This condition is supported by research using a PVA matrix that has a dramatically decreased elongation curve, this phenomenon is due to the lack of strong hydrogen bonds that lead to the limitation of polymer chain movement and reduction of elongation behaviour [29]. According to SEM observations, the aggregation of starch and lemon skin powder and the resulting porosity were the main factors causing the decrease in elongation value. However, interestingly, the addition of 5% lemon skin resulted in a better structure and a reduction in porosity and agglomeration, indicating the onset of good compatibility between PVA, starch, and LSP, which supports an increase in elongation value again. The elongation value with the addition of 5% LSP was identified as 432.33%. With homogeneous distribution, the bonds between alkaline starch, lignin, and PVA in the biocomposite became more effective, resulting in a stronger structure, which increased the mechanical strength and elongation at break [30].

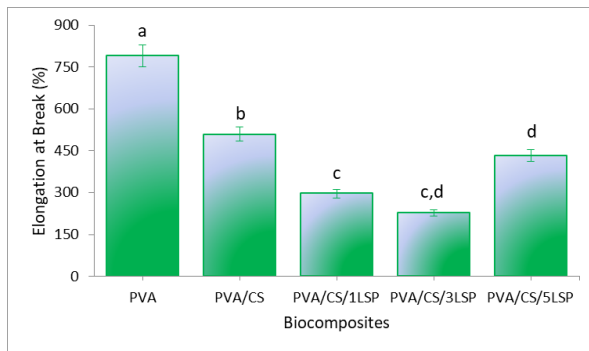


Figure 4. Elongation at Break Strain of Biocomposites ($p < 0.05$)

Additionally, chemical interactions, physical properties of the fibers, and appropriate composition can also influence the increase in elongation value [31]. These results are confirmed by the 5% fiber content having the highest tensile strength.

The elasticity modulus of biocomposites from Polyvinyl Alcohol and Cassava starch filled with lemon skin powder is shown in Figure 5. The addition of lemon skin into the matrix of PVA and cassava starch increases the modulus of elasticity. High modulus fibers improve the mechanical properties of composites, enhancing their structural bonding and stiffness [32]. This is corroborated by another research, which highlights that the role of fibers in the PVA matrix can increase the ductility and strain hardening of composite materials, which contributes positively to their mechanical properties [33]. The combination of natural fibers with PVA not only increases the elastic modulus but also improves the overall mechanical properties of the composite, making it suitable for various applications. This result is supported by a previous study that mixed PVA and starch with coffee grounds filler, which experienced an increase in elastic modulus [34]. This occurs because natural fibers can reduce the mobility of the polymer chains in the matrix, making the material more rigid.

The mechanical properties obtained from this study are competitive with conventional plastics used for food packaging, such as high-density polyethylene (HDPE) and low-density polyethylene (LDPE). The tensile strength of 13.82 MPa is comparable to that of conventional LDPE plastics, which range from 10 to 30 MPa. In addition, the maximum elongation of the PVA/CS/5LSP biocomposite film of 432.33% is comparable to the elongation of LDPE (100%-700%) and even better than HDPE, which has an elongation between 100%-300% [35].

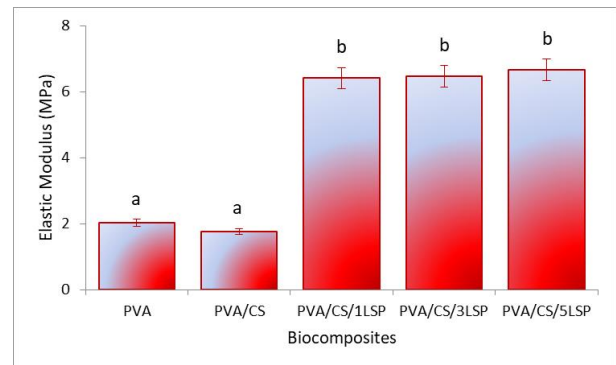


Figure 5. Elasticity Modulus of Biocomposites ($p < 0.05$)

These findings indicate that PVA-based biocomposites filled with 5% LSP have high potential as an alternative to conventional plastics, particularly in food packaging, when viewed from the perspective of a mechanical property.

Fracture Surface Morphology

Figure 6 shows the observation of the fracture morphology of the biocomposite. As can be seen from Figure 6(a), the surface of pure PVA is very smooth. This happens because there is no filler, so the PVA is completely dissolved with water and produces a smooth surface [36]. This is confirmed by the previous research, where the pure PVA film has a smooth structure [34]. In Figure 6(b), there is a mixture of PVA and cassava starch, which shows that there are cracks and gaps indicated by yellow arrows. In addition, there is an agglomeration of starch. This phenomenon is in accordance with the previous research, where poor compatibility between PVA and starch causes this phenomenon to occur [14]. In addition, agglomeration of starch was found in the observation of fault morphology. Agglomeration is the occurrence of a buildup of particles that causes the force distributed between the matrix and the fiber to be imperfect, which will cause a decrease in the tensile strength value [37].

Figure 6(c) shows PVA and cassava starch with the addition of 1% lemon skin powder filler. In this variation, it produces the lowest tensile strength among samples with lemon skin powder fillers. The results of morphological observation using SEM show that the surface of the sample has porosity or an empty space in the biocomposite sample. This is because the fibers are not uniform and not evenly distributed, and there is an inhomogeneous bond between the matrix and fiber, which can reduce the tensile strength [38]. In addition, agglomeration of fibers was found, which resulted in incompletely distributed forces in tensile testing.

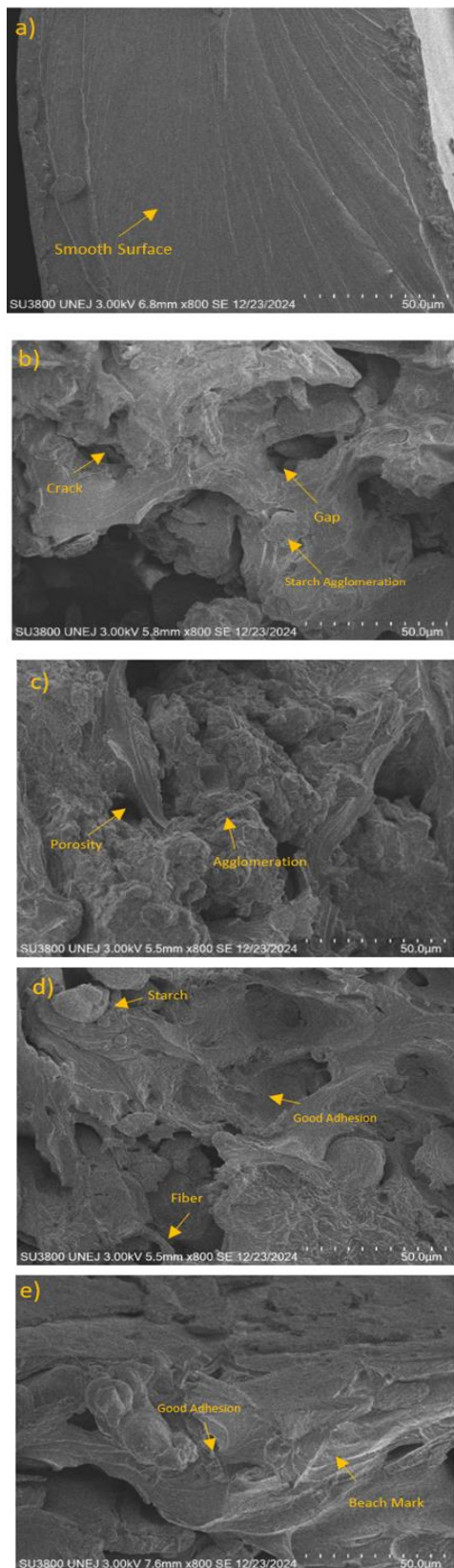


Figure 6. Fracture Surface Morphology of Biocomposites with variation of: a) PVA, b) PVA/CS, c) PVA/CS/1LSP, d) PVA/CS/3LSP, e) PVA/CS/5LSP

Imperfect mixing can cause agglomeration events, poor adhesion and no further treatment when the sample is molded into a biocomposite mold [39]. Additional treatment, such as ultrasonication, is required to maximize the mixing between PVA, starch, and LSP.

Another phenomenon is found in Figure 6(d), which shows PVA and cassava starch with 3% lemon skin powder filler. In this variation, an even structure began to form. This is due to good bonding that results in a compact structure. A compact structure can occur due to good fiber dispersion without fiber accumulation, causing an increase in the tensile strength of the sample [40]. This result is confirmed by the increasing tensile strength of the biocomposites.

Figure 6(e) shows the results of morphological observations using SEM on PVA biocomposites and cassava starch with 5% lemon skin powder filler. In this variation, the tensile strength exhibited the highest tensile strength. In this variation, the tensile strength increased. There is a wave line phenomenon, where the wave line occurs because the crack propagation path passes through the weak part due to the addition of dispersed starch gel [13]. The presence of wave lines and good bonding results in increased tensile strength [34]. The presence of wave lines indicates an even distribution of starch and fiber and the tensile stress passes through the weakest bond increasing tensile strength. The SEM results in this study are supported by previous research [13][34].

Thermal Stability Properties

Figure 7 shows the weight loss of PVA and cassava starch biocomposites with the addition of lemon skin powder. It can be seen that at temperatures ranging from 10 °C to 100 °C, which is the initial temperature, the biocomposite experiences weight loss of up to 10% due to the loss of water content [41]. Significant weight loss of up to 45% occurs at temperatures ranging from 197 °C to 435 °C, which is the initial decomposition temperature [27]. The addition of 5% lemon skin powder to the PVA and cassava starch matrix resulted in an initial decomposition temperature of 214 °C. The bonds between PVA, cassava starch, and lemon skin powder became more thermally stable than pure PVA, which began to decompose at 204 °C. This phenomenon occurs because the added fibers strengthen the intermolecular bonds with the PVA and cassava starch molecular chains that complement the intermolecular hydrogen bonds [42].

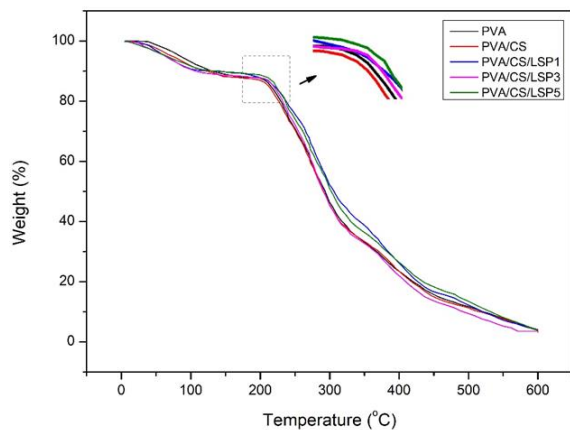


Figure 7. Thermal Properties

This is supported by the SEM results in Figure 6(e), which show good interfacial bonds. In addition, the tensile strength of adding 5% lemon skin powder to the PVA and cassava starch matrix was the highest result. These results are similar to related research on the use of orange skin as a PVA filler, which was reported to increase thermal stability with high content. However, the difference is that orange skin fiber is not further processed with chemical treatment as a filler [43]. In the context of starch and PVA, treating the fiber with chemical treatment is important to support more responsive fibers when bonding with the matrix. Similar studies have shown that the addition of chemically treated natural date palm leaf fibers in PVA and starch matrices can result in better thermal resistance even at higher concentrations [27]. In addition, the presence of bioactive compounds in lemon skin also affects the thermal resistance of biocomposite films. This is explained in a study related to the addition of pomegranate skin powder to PVA-based biocomposites. The results show that the presence of bioactive compounds in fillers such as alkanoids and flavonoids, which are rich in (-OH) groups, also contributes to resisting the thermal degradation of biocomposites [44]. Therefore, lemon peel, which is rich in similar bioactive compounds, is effective in improving the thermal properties of starch and PVA-based biocomposites.

CONCLUSIONS

PVA and cassava starch biocomposites with LSP filler have been successfully produced using the casting method. The addition of LSP filler can improve the mechanical and thermal properties of biocomposites. The highest tensile strength was found in the variation with 5% lemon skin powder filler, with a result of 13.82 MPa, an elongation value of 432.33%, and an elastic

modulus of 6.68 MPa. The thermal properties of the 5% LSP addition also increased from 204 °C to 214 °C when compared to pure PVA. These results were reinforced by fracture morphology observations using SEM, which showed that the fracture surface of the sample with good characteristics was found in the addition of 5% lemon skin powder filler due to its compact structure, good adhesion, and the presence of beach marks. These results indicate that LSP is a potentially positive candidate as a filler for PVA and starch-based biocomposites, especially in biodegradable packaging film applications. The addition of higher LSP content and its potential as an active packaging film needs to be further studied in subsequent research. This is necessary to determine the maximum LSP content that can be achieved in PVA and starch-based biocomposites and their effectiveness in protecting products such as food from biological or chemical damage.

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