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SUSTAINABLE BIOCOMPOSITES FROM RICE STRAW BONDED WITH POLYVINYL ALCOHOL–CITRIC ACID–BASED ADHESIVES FOR FOOD PACKAGING

S. Hutagalung[1]*, M. A. R. Lubis[2*], and T. H. R. Puspaningtyas[1]

[1]Department of Chemistry, Satya Wacana Christian University, Indonesia
[2]Research Center for Biomass and Bioproducts, National Research and Innovation Agency, Jakarta, Indonesia
****Email: sarawinda.hutagalung@uksw.edu, muha142@brin.go.id***

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ABSTRACT

Polysaccharides like cellulose, starch, and chitosan are generally co-polymerized with synthetic polymers to achieve the needed performance for packaging applications. Paddy is a rice-producing crop whose production is strived for throughout the year. Rice straw (RS) is a waste resulting from the rice milling process. RS contains lignin, cellulose, and hemicellulose. Biocomposites of RS cellulose and PVOH can be an environmentally friendly packaging alternative. In this study, the process of making biocomposites from straw and PVOH used the extrusion method and baking process. This research aims to determine the properties of RS, PVOH, and citric acid (CA) to generate biocomposite sheets and determine the characteristics of the mechanical properties of biocomposites with parameters of tensile strength and bonding strength. Fourier Transform Infra-Red (FT-IR), Fourier Transform Near Infra-Red (FT-NIR), digital microscope, and thermogravimetric analyzer (TGA) were utilized to characterize the PVOH/CA/RS. FT-IR and FT-NIR showed the presence of an ester group in PVOH/CA/RS, while TGA observed lower degradation temperatures in biocomposites, indicated by the Tonset and Tmax of 216 °C and 320 °C, respectively. Tensile strength and bonding strength of PVOH/CA/RS biocomposite showed a promising value, which is higher than the Indonesian National Standard (SNI), > 1.6 kN/m and > 100 j/m2, respectively. This study suggested that PVOH/CA/RS biocomposite can be developed as sustainable food packaging.

Keywords: biocomposite; rice straw, PVOH, citric acid; packaging

INTRODUCTION

Recent developments in biocomposites have attracted attention from a variety of industries due to the enhanced characteristics of biocomposites. Biocomposites also have advantages for the sustainable environment (Gowthama Prabu Udayakuma *et al.* 2021) and can also be altered by adding attributes concerning their functionality (Rong, Mubarak, and Tanjung 2017). Cellulose, starch, collagen, keratin, chitosan, silk, and *elastin* are examples of readily available biopolymers that were combined with synthetic polymers to create composites that had an elevated effect (Gowthama Prabu Udayakuma *et al.* 2021). Biocomposites can be found in a variety of forms, including films, membranes, moldings, coatings, particles, fibers, and foams (Kazutoshi Haraguchi 2014). Biocomposites have been created to enhance physical or mechanical properties in terms of configuration, performance, and applications such as packaging materials that can be utilized in food, pharmaceutical, and medical areas. Over the past several years, several food enterprises have emerged globally, and people's preferences have shifted toward meals that are organically derived to maintain and improve their health. Owing to the growing demand for food, the industry requires edible or biodegradable food packaging materials. Polysaccharides like cellulose, starch, and chitosan were co-polymerized with synthetic polymers to achieve the needed performance for packaging applications. The combination of natural biopolymers and synthetic polymer compounds made materials provides a strong benefit towards physical and mechanical properties (Gowthama Prabu Udayakuma *et al.* 2021), because of their ease of handling, chemistry, and reliability.

Rice (*Oryza sativa* L.) is one of the species of the genus Oryza, which belongs to *Poaceae* (*Gramineae*) family. Paddy is a plant to produce rice crop for which the production is strived for throughout the year because rice is the staple food of the Indonesian people (Kurniasih *et al.*, 2008). As the population increases, the consumption of rice from year to year increases as well (Sudadi, Sumarno, and Handi 2014). In one year, paddies are harvested 3 times to meet the food needs of the society (Putri 2015). After harvesting and during the rice production process, paddies can produce side waste in the form of rice straw, husks, and bran. Paddy waste can be a source of environmental problems if not managed properly, as it can cause water and air pollution and soil damage. Paddy straw contains fiber and important nutrients such as carbohydrates, proteins, and minerals, so it can be processed again as a recycled raw material for various products such as organic fertilizer, animal feed, building materials, fuel, compost, papermaking, and handicrafts (Sari 2023). Straw is part of the stems and stalks of paddy plants after harvesting the grains (Pratiwi *et al.*, 2016). Paddy straw contains 32-47% cellulose, 20%-30% hemicellulose, and 10%-18% lignin (Wang *et al.* 2015; Yan *et al.* 2021). As paddy straw contains lignocellulose, it can be a potential resource as filler in making a biocomposite that has functions as a reinforcing material for the biocomposite structure. Meanwhile, polyvinyl alcohol (PVA) is a matrix that functions as a binder of fiber. PVA is a synthetic polymer that has properties such as hydrophilic, biodegradability, and biocompatible. PVA can increase the flexibility and durability of a material (Mahardika *et al.* 2021), thereby improving the mechanical properties of the biocomposite. This incorporation provides biocomposites with a remarkable blend of biocompatibility, mechanical strength, and customizable properties (Al Mahmud 2023). Biocomposites of paddy straw and PVA can be an alternative to environmentally friendly packaging.

The addition of acetic acid (AA) in the development of biocomposite serves as a medium that enhances the adsorption of nonpolar molecules (Zhang *et al.* 2024). Citric acid (CA) plays a role to enhance crosslinking (Das *et al.* 2024) by facilitating covalent intermolecular di-ester interactions between its carboxyl groups and the hydroxyl groups of biopolymers. This cross-linking enhances the physical, mechanical, and thermal, of the resulting materials (Dudeja *et al.* 2023).

In previous studies, paddy straw was used as a basic material in the making of biofoam and PVA (Haiqal and Muldarisnur 2023), making bioplastics from straw and chitosan (Pratiwi, Rahayu, and Barliana 2016), making Carboxymethyl cellulose (CMC) from rice straw (Masrullita *et al.* 2021) making straw biofoam food packaging with tapioca starch bioplastic coating (Ginola, Nuraliyah, and Alwi 2024), the effect of variations in the composition of rice straw cellulose and polyethylene terephthalate (PET) plastic bottle waste on the characteristics of biodegradable plastic (Sjafarina, Syahbanu, and Nurlina 2021). Meanwhile, biocomposite sheets based on polyvinyl alcohol (PVOH) and citric acid reinforced with rice straw fibres have never been done. In this study, the extrusion and baking processes were used to make biocomposites from straw using PVOH/CA-based adhesive.

This research aims to determine the composition of fiber, PVA, and citric acid to generate biocomposite sheets and to determine the mechanical properties of biocomposites with parameters of tensile strength, water absorption, biodegradation, and FTIR analysis.

MATERIALS AND METHODS

Materials

Paddy straw, sodium hydroxide (NaOH), polyvinyl alcohol (PVA), acetic acid, magnesium stearate, citric acid, distilled water.

Instruments

Beaker glass, watch glass, baking sheet, spatula, measuring cylinder, thermometer, 80 mesh sieve, waterbath, pH meter, drying cabinet, hot plate, magnetic stirrer, grinder, analytical balance with accuracy of 0.1 mg (OHAUS PA 214), volume pipette, philius, and stirring rod were used in this research. Fourier Transform Infra-Red (FT-IR, SpectrumTwo, Perkin Elmer, USA), Fourier Transform Near Infra-Red (FT-NIR, SpectrumTwo, Perkin Elmer, USA), digital microscope (VHX 6000, Keyence, Japan), thermogravimetric analyzer (TGA 4000, Perkin Elmer, USA), and universal testing machine (UTM, Shimadzu AGS-X series 5 kN, Japan) were conducted in integrated laboratory of Bioproducts, National Research and Innovation Agency, Indonesia.

Preparation of Rice Straw Fiber

Rice straw (RS) was washed thoroughly with running water, cut into small pieces, and then powdered using a grinder. The rice straw powder was sieved using an 80-mesh sieve, after which the rice straw fibers were stored in a ziplock bag, as displayed in Figure 1.

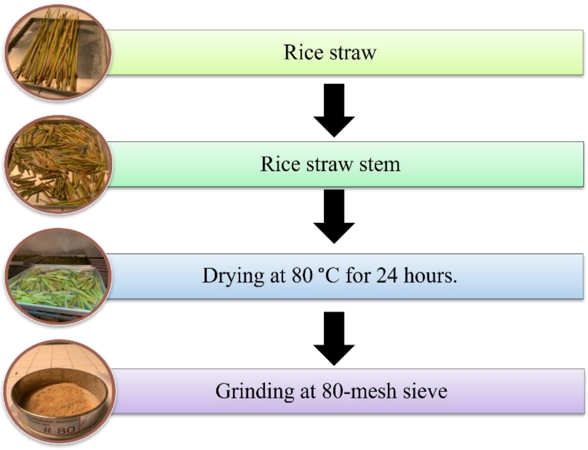

Figure 1. Preparation of rice straw fiber

Biocomposite Preparation

Polyvinyl alcohol (PVOH) was dissolved in distilled water and stirred until homogeneous. In the next step, magnesium stearate was added and then stirred until homogeneous. Afterwards, the PVOH/CA solution was added and stirred. The biocomposite mixture was poured into a baking sheet and heated in the oven at 80°C for 24 h. The pan was removed from the oven and cooled at room temperature. Figure 2 illustrates the preparation of biocomposite from rice straw (RS)

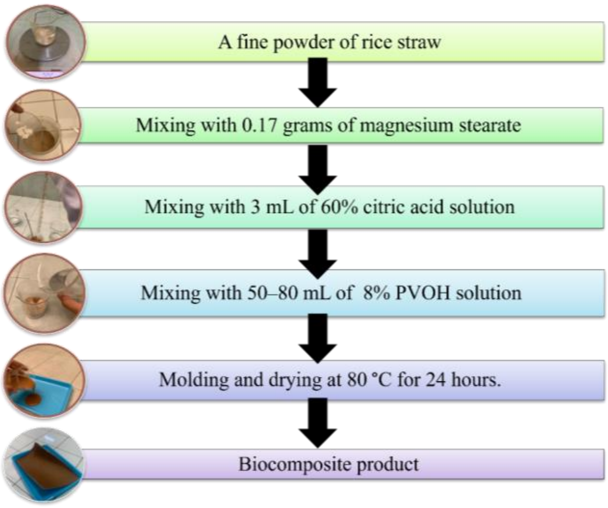

Figure 2. Preparation of biocomposite product

Functional Group Analysis

Functional group of PVOH, CA, and mixture of PVOH/CA/RS were analyzed using FT-IR spectroscopy (SpectrumTwo, Perkin Elmer, USA) and FT-NIR spectroscopy (SpectrumTwo, Perkin Elmer, USA). Before sample insertion into the sample holder, a background analysis was conducted. Subsequently, the pressed adhesive sample was placed, and its spectral signature was acquired and stored through an average of 16 scans at a resolution of 4 cm^{-1}, covering wavenumbers ranging from 4000 to 400 cm^{-1} for FT-IR and wavenumbers ranging from 8000 to 4000 cm^{-1} for FT-NIR spectroscopy. The curved generation process was executed utilizing the UATR methodology. The outcomes of the spectral analysis were visualized using the Origin software developed by OriginLab in the United States, facilitating the examination of each distinctive wavenumber.

Morphology Analysis

The morphology of biocomposites was observed using a Digital Microscope with 100-times magnification at 25±2°C. The films of PVOH, CA, and PVOH/CA/RS were prepared at 25±2°C before the observation.

Thermal Analysis

TGA (TGA 4000, Perkin Elmer, Hopkinton, MA, USA) was used to investigate the thermal stability of PVOH, CA, and PVOH/CA/RS. A conventional ceramic crucible was filled with around 20 mg of sample and heated in a nitrogen atmosphere at a 20 mL/min of flow rate. The heating temperature varies from 25 to 750°C, with a 10 °C/min of heating rate. Percentages of weight loss, derivative weight loss (DTG), and residue were calculated using Pyris software (Version 11, Pyris, Washington, MA, USA).

Mechanical Analysis

Mechanical properties of the biocomposites were evaluated based on Indonesian National Standard (SNI 8218:2015) using a universal testing machine (AG-X series, Shimadzu, Kyoto, Japan). The specimen size was 100 mm × 25 mm. The specimens were evaluated with a load cell of 5 kN at a temperature of 23°C. Bonding strength and tensile strength of biocomposites were evaluated accordingly.

RESULTS AND DISCUSSION

Figure 3 displays FT-IR spectra of PVOH and CA. In general, there are three possible degradation reactions: the hydrolysis of ether or ester bonds, the polymerization processes, and the possible cleavages of both PVOH chains, PVOH segments, or CA moieties (Iswanto and Lubis 2022; Lubis, Park, and Hong 2020; Sridach, Jonjankiat, and Wittaya 2013). The hydroxyl groups (–OH) of the PVOH at wavenumber 3350 cm^{-1} can react with carboxyl groups (–COOH) of CA at 1725 cm^{-1} (Figure 3). The first way is the proton abstraction from the hydroxyls of PVOH to give an alcoholate anion and the corresponding acid, which leads to crosslinking of PVOH. The crosslinking presents in samples cured by thermal treatment verifies the formation of the PVOH/CA ester bond (–COO–R) at 1780 cm^{-1}. The second way is the condensation reaction between the ester bond and PVOH, possibly forming bio-polyester. The third way is an alternative reaction with a similar product but a water transfer (dehydration) mechanism (Del Menezzi *et al.* 2018).

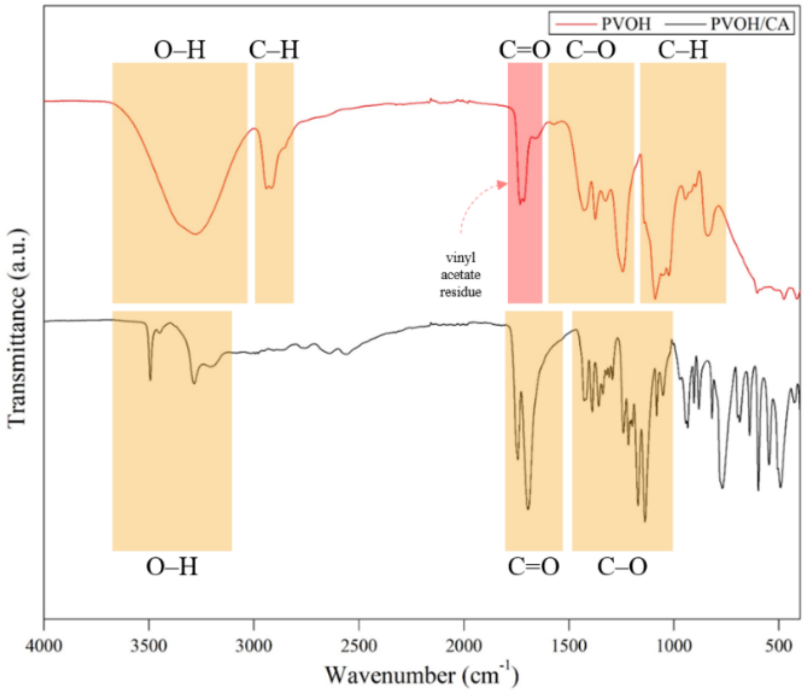

Figure 3. FT-IR spectra of PVOH and CA

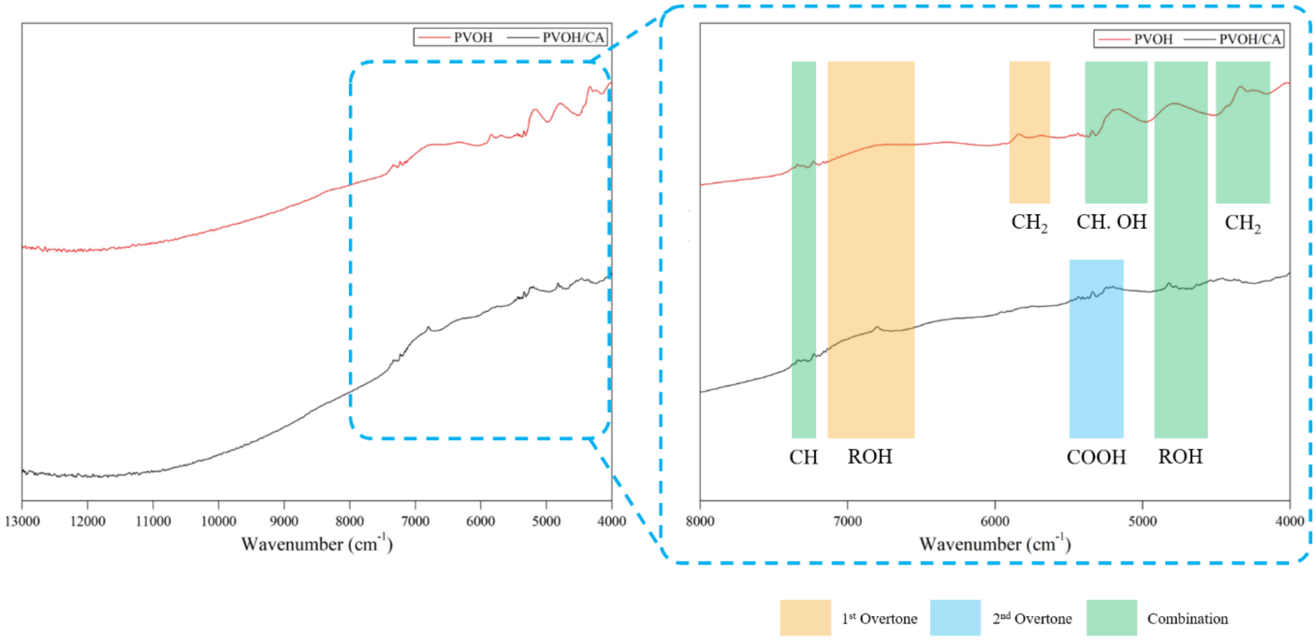

Figure 4. FT-NIR spectra of PVOH and CA

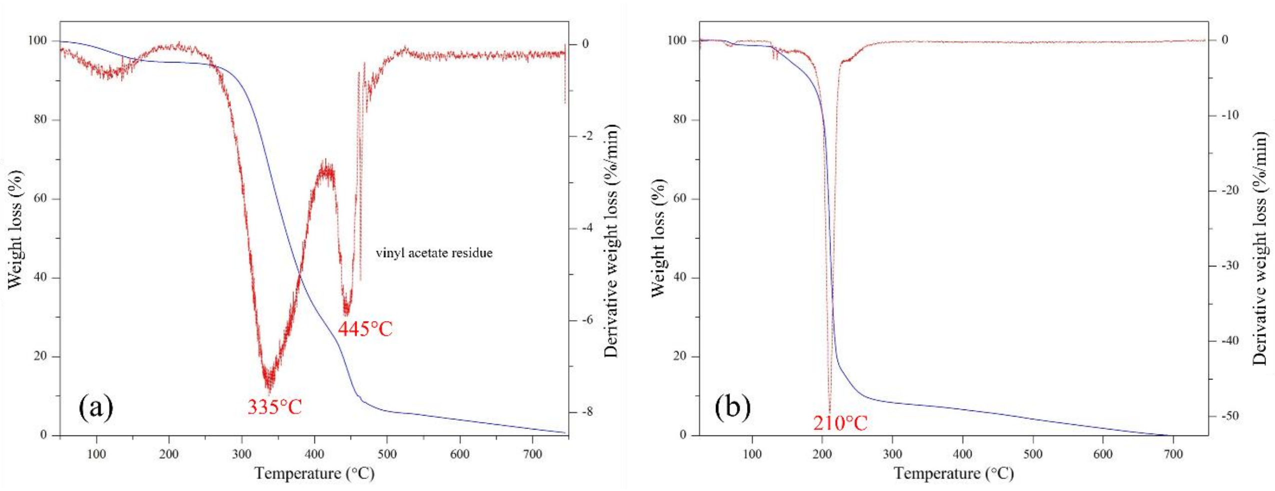

Figure 5. TGA-DTG thermogram (a) PVOH and (b) CA

Packaging materials for food applications are increasingly required to be versatile and cost-effective. In this work, the development of PVOH-based composites incorporating food-grade organic acids, such as CA, is reported. These biocomposites are expected to be capable of reducing spoilage due to the growth of pathogenic or spoilage microorganisms associated with food through active diffusion of organic acids from the biocomposites to food. Figure 4 shows the FT-NIR spectra of PVOH and CA. FT-NIR absorbance spectra were obtained from an instrument with an ATR accessory, with a scanning range from 8000 to 4000 cm^{-1}. Some additional information can be gathered from FT-NIR, such as identification of the CH and ROH at 7286 cm^{-1} and 6800 cm^{-1}. Besides that, in the region of 4600 to 5200 cm^{-1}, the band at 4716 cm^{-1} is attributable to the combination of the bent vibration of the OH. This band suggests that the possibility of branching increased due to the reaction between hydroxyl groups. However, the intensities of bands at 5090 and 5165 cm^{-1} decreased, suggesting that these bands are attributable to the cyclic bent vibration (Cho *et al.* 2016; Lohumi *et al.* 2014).

Thermogravimetric analysis (TGA) for the assessment of thermal stability of the neat PVOH and citric acid was performed on samples heated under a nitrogen atmosphere. The weight loss as a function of temperature is reported (Figure 5). TGA and DTG curves of pure PVA show a weight loss of about 98% in the temperature range of 25 to 700°C. Pure PVOH begins to decompose at approximately 125°C, and the main decomposition stage occurs over the temperature range of 225–445°C. The first mass loss stage (15%) occurred between 125 and 200°C, which is attributed to the evaporation of water molecules. The second mass loss stage, occurring between 200 and 445°C, was a major decomposition stage for PVOH. From the results, it was found that the maximum weight loss rate occurs at about 335°C, with a weight loss is about 83.5%. The maximum weight loss rate of this process is 7.8%/°C. PVOH then decomposes slowly in this stage. Most PVOH (80%) decomposed during this temperature range, which was due to the microcrystalline structure of PVOH (Ismayati *et al.* 2024; Lubis, Yadav, and Park 2021). The last stage occurred in the temperature range of 400–600°C and was due to the further thermal decomposition to char and gaseous products. There is no significant change after 600°C, indicating the stability of PVA char over this temperature range.

The weight loss of neat CA occurs within two distinct temperature ranges: from 25 °C to 210 °C, corresponds to the dissociation of carboxyl groups (-COOH) groups which is accompanied by CO and CO_2 gas emissions, and from 210 °C to 500 °C, dehydration occurred which leads to charring chars (Tsioptsias & K. Zacharis, 2024). High temperature inclusion of char burning in the TGA range from 420 °C to 650 °C and even above. The observed stages of weight loss in all samples are the same as those of neat CA, indicating that almost all the char residue is produced by CA (Sutiawan *et al.* 2023).

PVOH/CA/RS biocomposites characterization was carried out using FTIR spectroscopy (Figure 6a). The analysis was carried out at a wavenumber of 4000-400 cm^{-1}. By virtue of the unique vibrational oscillation of molecules, every chemical compound has unique infrared spectra. The peaks between 3450 and 2990 cm^{-1} were attributed to the hydroxl (O−H) and aliphatic C−H stretching vibrations of PVOH, CA, and RS. Considering that PVOH has much “free” O−H groups which can interact with RS. RS with relatively lower O−H content should have less broad band absorption when compared to pure PVOH. Two peaks at 2931 and 2852 cm^{-1} relate to the C−H stretching vibrations of RS and PVOH. The reduced band intensity at 2970 cm^{-1} for the composites may be ascribed to the reaction of the free −OH in PVOH with −C(=O)O− and −C(=O)OH of CA to form ether linkages in the composites (Frone *et al.* 2015; Nagalakshmaiah *et al.* 2018). The strong absorption at 1744 cm^{-1} is due to C=O stretching vibration mainly from CA. On one hand, RS had peak at 1730 cm^{-1} due to the carbonyl C=O bond of hemicellulose and lignin-originated carboxylic acid groups. Lower methyl C−H (peak at 1160 cm^{-1}) was ascribed to the pH induced inter/Intra H bonding in RS.

Figure 6b and c depicts the TGA and DTG curves for PVOH/CA/RS biocomposites as a function of temperature. The TGA curves of PVOH, CA, and RS are also included for comparison. The TGA curves demonstrate that the temperature remains the same even when weight loss occurs. The first derivative TGA curves (DTG) indicate the temperature at which weight loss occurs. The PVA/CA/RS composites were calcined for 1 hour in a 700 °C furnace, resulting in a black char that was stable at that temperature (Arfiathi *et al.* 2024). Based on Figure 6b and c, it was concluded that the thermal stability of the PVA/CA/RS composites decreased as rice straw was added. The peak decomposition temperature also decreased with the addition of RS. PVOH is a semi-crystalline polymer, and its degradation typically occurs at 337–480 °C with a maximum rate of mass loss (T_{max}) at 408–428 °C (Awad and Khalaf 2019). RS has an initial decomposition temperature (T_{max}) of about 302 °C. CA, with its initial weight loss temperature at 210 °C, did not significantly expand the thermal degradation temperature range of the composite. The biocomposites yielded lower degradation temperatures, indicated by the T_{onset} and T_{max} of 216 °C and 320 °C, respectively. The weight percentage loss of RS reached 99.01% and was almost complete at 700 °C.

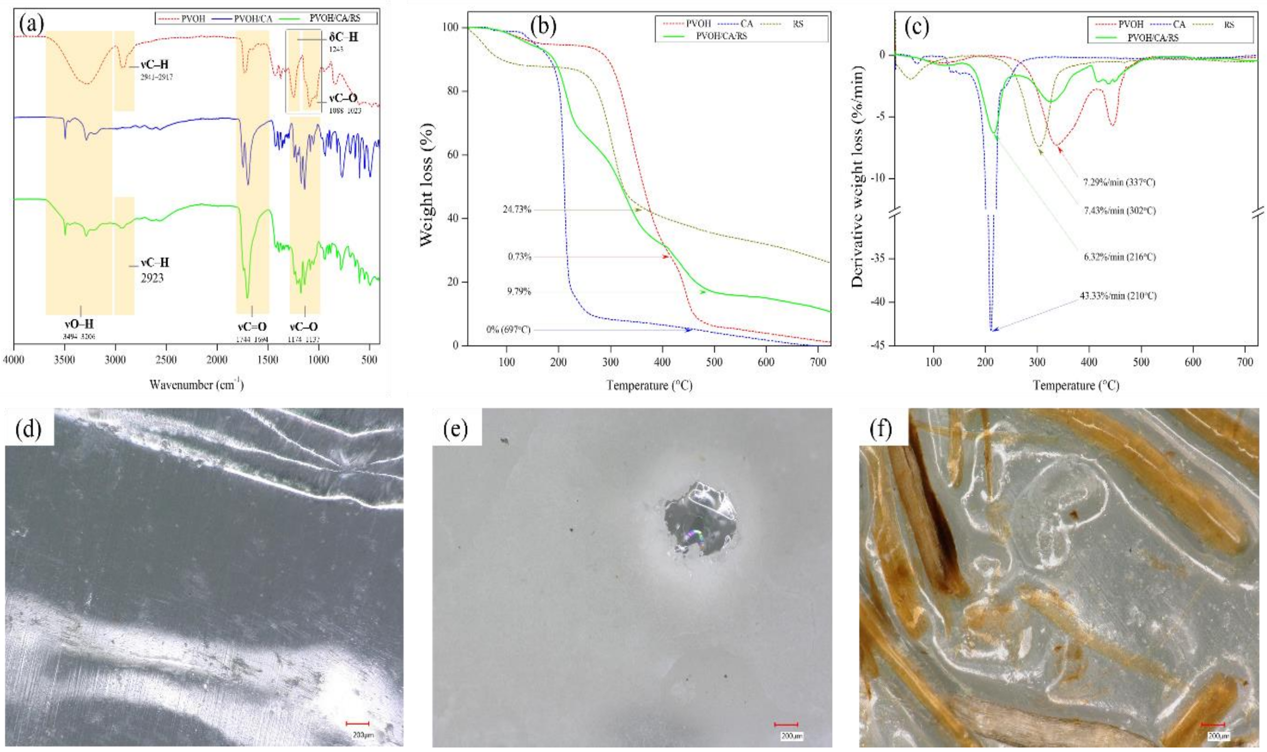

Figure 6. Characteristics of PVOH/CA/RS. (a) FTIR spectra of PVOH/CA/RS, (b) TGA of PVOH/CA/RS, (c) DTG of PVOH/CA/RS, (d) morphology of PVOH, (e) morphology of PVOH/CA, (f) morphology of PVOH/CA/RS

The digital microscope images of a PVOH, CA, and RS bio-composited film is shown in Figure 6d-f. In this research, the bio-composite film was prepared from environmentally friendly materials. The biocomposite film was formed by the interaction between hydroxyl groups of PVOH with the carboxylic group of the CA as a cross linker. The RS was a hydrophilic material, which could absorb and retain a large amount of moisture from the surrounding atmosphere. This shortcoming could limit its further applications, especially for food packaging films. In this research, the goal was to prepare and investigate PVOH/CA/RS biocomposites. To get the reinforced materials, the RS were treated by alkali (magnesium stearate) and amplified with the CA and formed cross-links in the PVOH matrix (Chen *et al.* 2017; Frone *et al.* 2015).

Tensile strength and bonding strength of PVOH/CA/RS-biocomposite were investigated using UTM (Table 1). The result showed that the average tensile strength and bonding strength values of biocomposite were higher that the Indonesian National Standard, 1.81 kN/m and 1807.21 J/m^2, respectively. This result suggested that PVOH/CA/RS biocomposite can be developed as sustainable food packaging. Further research are required to evaluate the effect of different PVOH/CA/RS combinations on mechanical properties of biocomposites.

Table 1. Mechanical properties of biocomposites of PVOH/CA/RS

Sample	Tensile strength (kN/m)	Bonding strength (J/m^2)
1	1.77	1765.04
2	1.62	1618.81
3	1.98	1975.69
4	1.87	1869.29
Average	**1.81**	**1807.21**
Std. Dev.	**0.15**	**152.22**
SNI	**> 1.6**	**> 100.0**

CONCLUSION

This research aims to determine the properties of PVOH/CA/RS biocomposite and evaluate the characteristics of the mechanical properties of biocomposites with parameters of tensile strength and bonding strength. Fourier Transform Infra-Red (FT-IR), Fourier Transform Near Infra-Red (FT-NIR), digital microscope, and thermogravimetric analyzer (TGA) were utilized to characterize the PVOH/CA/RS. FT-IR and FT-NIR showed the presence of ester group in PVOH/CA/RS, while TGA observed lower degradation temperatures in biocomposites, indicated by the Tonset and Tmax of 216 °C and 320 °C, respectively. Tensile strength and bonding strength of PVOH/CA/RS biocomposite showed a promising value which higher than the Indonesian National Standard (SNI), > 1.6 kN/m and > 100 j/m^2, respectively. This study suggested that PVOH/CA/RS biocomposite can be developed as sustainable food packaging.

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