Properties of Acrylonitrile Styrene Acrylate Copolymer Modified Eucalyptus/Polyvinyl Chloride Composites

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Eucalyptus/polyvinyl chloride (PVC) composites were prepared by extrusion molding with eucalyptus as filler, PVC as matrix, and different contents of acrylonitrile styrene acrylate copolymer (ASA). The effects of different ASA content on the mechanical properties, water absorption properties, and thermal stability of eucalyptus/PVC composites were studied. The morphology of the tensile section of the composites was observed by scanning electron microscopy (SEM). The results showed that the addition of ASA could improve the mechanical properties, heat resistance, and interfacial compatibility of eucalyptus/PVC composites. It also could reduce the water absorption of the composites. When ASA content was 10 wt%, the mechanical properties and heat resistance of eucalyptus/PVC composite were the best, and the water absorption in 24 h was the lowest.

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INTRODUCTION

Wood-plastic composite material is a category of environmentally friendly materials that has risen rapidly at home and abroad in recent years. It uses waste plastics and waste wood materials as the main raw materials and adds a certain proportion of additives, which can be fabricated through four different methods, molding, extrusion, and injection molding (Zhang *et al.* 2018; Tang 2020; Wang *et al.* 2020). It not only has the toughness and processability of plastic, but it also has the hardness and environmental protection of wood, which alleviates the environmental problems caused by waste plastics and biomass. Because of its practicability, bacteriostasis, corrosion resistance, low cost, and other advantages, it is widely used in building and gardening, furniture decoration, and automobile interior decoration.

Polyvinyl chloride (PVC) is the second most available general plastic just after polyethylene (PE). PVC has good degradation resistance, flame retardancy, small thermal conductivity, and excellent insulation ability, and it is used widely in building materials, pipes, films, and insulating materials. However, PVC is hard and brittle and subject to brittle fracture, which limits its applications (Crawford and Lesser 2000; Bing and Huang 2009). In general, adding rubber particles into the brittle matrix can effectively improve the impact properties of the blends. Butyl acrylate styrene acrylonitrile graft copolymer (ASA) is a core-shell structure modifier, in which the "core" is butyl polyacrylate (PBA) rubber phase and the "shell" is styrene acrylonitrile (SAN) copolymer (Scheirs *et al.* 2003; Zhao *et al.* 2017; Xiang and Zhang 2017;Xiang and Zhang 2018). Because of the good

compatibility between PVC and SAN, PVC can be modified with ASA to improve the impact property of the material (Guan *et al.* 2018). Ma *et al.* (2012) prepared ASA core-shell graft copolymer with butyl polyacrylate (PBA) as the core and styrene (St) and acrylonitrile (AN) as the shell. They studied the influence of ASA content on the plasticization and impact properties of PVC. They found that *m* (ASA)/*m* (PVC) = 0.06, ω(PBA) = 0. 60, ω(AN) = 0.40, and the modified PVC has the best effect. Xu *et al.* (2012) studied the mechanical properties of PVC/ASA blends and the effect of wood flour filling on the properties of the system. The results showed that with the increase of ASA content in PVC, the tensile strength of PVC blends decreased, while the impact strength increased first and then decreased. When 30 phr wood flour was added to PVC/ASA blends, the strength of the composites did not decrease significantly. Han *et al.* (2009) found that the yield strength of ASA/PVC alloy decreased linearly with the increase of ASA content. The impact property of ASA/PVC alloy was related to the core shell ratio of ASA. When it was lower than 55/45, the impact property of the alloy had a significant downward trend. Zhai and Wei (2021) prepared PVC/ASA alloy by extrusion granulation using ASA, PVC resin, and various additives, and carried out blending modification research to investigate the effect of ASA on the properties of the blend. Experiments proved that ASA can effectively modify PVC. With the increase of ASA content, the impact strength of PVC can be greatly improved. Wang *et al*. (2017) prepared PVC/α-MSAN/ASA blends by blending ASA with PVC and α-MSAN. It was found that when ASA core-shell ratio was 60/40, the blends had higher impact strength and tensile strength. The SEM image of the section of the blend showed that the blends fracture at the deep level and the flatness of the section became worse, which was matched with its high impact performance.

In recent years, there have been few studies on the effect of ASA on the properties of PVC wood-plastic composites. Sarawut *et al.* (2011) prepared coconut fiber/PVC/ASA composites. The results showed that PVC/ASA blends had excellent impact strength and thermal properties. The impact strength of PVC/ASA/coconut fiber composites was significantly higher than that of PVC/coconut fiber composites and polyolefin/coconut fiber composites. Jiang *et al.* (2019) reinforced PVC/ sorghum straw (SS) composites with microsilicon (MS) and ASA. The results showed that the addition of 6 wt% MS (particle size 2.6 μm) and 34% ASA improved the wear resistance and corrosion resistance of PVC/SS composites significantly. These findings showed that MS and ASA can improve the toughness, strength, and heat resistance of PVC matrix, thus obtaining a new type of corrosion and wear-resistant material. As a kind of high quality, fast growing, and high yielding tree species, eucalyptus has become one of the most important afforestation trees in the global plantation industry, which solves the problem of slow growth rate effectively and unsustainable development of primary forests (Wang *et al.* 2016). Zhang *et al.* (2021) studied the effects of different contents of ASA on the mechanical properties, thermal properties, and three-body wear properties of eucalyptus fiber/polyvinyl chloride (EF/PVC) composites. The results showed that the addition of ASA can improve the mechanical properties, thermal stability, and three-body wear resistance of EF/PVC/ASA composites.

In this paper, the effect of ASA content on the properties of eucalyptus/PVC composites was considered. The effect of ASA content on the mechanical properties, thermal properties, and water absorption properties of the composites were analyzed, and the interfacial compatibility of the composites was analyzed by observing their microstructure. This provides a theoretical reference for further research on the properties of modified PVC matrix composites.

EXPERIMENTAL

Materials

Eucalyptus was obtained from Jiangmen Xinhui District Shuangshui Town Mujiang Weihua fragrance factory, Guangdong, China. Before mixing, eucalyptus flour was air-dried, crushed, and sieve-screened (100-mesh); the eucalyptus fibers were soaked in deionized water at 90 ℃ for 24 h, then removed and dried at 90 ℃ for 6 h.

The SG-5 PVC, melting point 150 to 200 °C, was obtained from Dongguan Jinheng Plastic Co., Ltd., Guangdong, China. The PW-978B ASA was obtained from Dongguan Lehua Plastic Raw Material Firm, Dongguan, China. Maleic anhydride coupling agent, F508 , was obtained from Shenzhen Hai'an Plastic Chemical Co., Ltd., Guangdong, China. The ZP-510 non-toxic Ca/Zn composite stabilizer was obtained from Shanghai Zirun Chemical Auxiliaries Co., Ltd., Shanghai, China. The LP0100P PE wax was obtained from Henan Huayue Chemical Products Co., Ltd., China.

Composites Preparation

The raw materials were put in DHG-9053A electric thermostatic drying oven (Shanghai Jiecheng Experimental Instrument Co., LTD., Shanghai, China) and dried for 10 h at 90 ℃ for reserve. The following components were mixed according to the formula in Table 1: dried eucalyptus fiber, PVC, ASA, and other additives.

All materials were put into an SBH-5L three-dimensional linkage mixer (Nanjing Xinbao Electromechanical Equipment Industrial Co., Ltd., Nanjing, China) and mixed for 15 to 20 min. The mixture was sent to an RM200C conical twin-screw extruder (Harbin Harper Electrical Technology Co., Ltd., Harbin, China) for extrusion. The temperature distribution of the four processing areas was 150, 155, 160, and 165 °C, and the screw speed was 20 rpm. The plate section size was 10 mm × 7 mm, cut into samples according to the test standards.

Table 1. Ratio of Components in the Blends

Sample No	Eucalyptus fiber (g)	PVC (g)	ASA (g)	Maleic anhydride coupling agent (g)	Ca/Zn stabilizer (g)	PE wax (g)
ASA0	50	50	0	3	8	5
ASA5	50	50	5	3	8	5
ASA10	50	50	10	3	8	5
ASA15	50	50	15	3	8	5

Methods

Mechanical property testing

The tensile strength and bending strength of the sample were tested with a CMT6104 electronic universal testing machine (Meters Industrial Systems (China) Co., Ltd.), while the loading speed was set to 2 mm/min. The impact strength was tested with an XJJ-5 simply supported beam impact tester (Chengde Jinjian Testing Instrument Co., Ltd., Jinjian, China). The test methods referred to the national standards GB/T 1040.4 (2006), GB/T 9341 (2008), and GB/T 1043.1 (2008). The Rockwell hardness of the sample, using the HRR scale, was tested with XHR-150 plastic Rockwell hardness tester (Shanghai Lianer Testing Equipment Co., Ltd.), according to the national standard GB/T 3398.1 (2008). The diameter of the indenter was 12.7 mm, the external load was 60 kg, the load

duration was 5 s, and the average unloading time was 15 s. The tests were conducted at room temperature, and the results were averaged 5 times.

Thermal stability analysis

The thermogravimetric curves (TG/DTG) of composite materials were evaluated with NETZSCH STA449F3 synchronous thermal analyzer (NETZSCH Instrument Manufacturing Co., Ltd., Selb, Germany). The test environment was conducted under the cover of an inert gas Ar. The purging gas rate was 60 mL/min, the protection gas rate was 20 mL/min, the temperature condition was controlled at 30 to 800 °C, the heating rate was 20 °C per minute, the sample quality was strictly controlled at 8 to 10 mg, and the sample was loaded with Al_2O_3 crucible.

FT-IR analysis

The composites and KBr were ground evenly. They were put into a YP-2 tablet press (Suzhou Anyuan Instrument Co., Ltd., Suzhou, China), then pressed them into a translucent sheet with a diameter of 13 mm and a thickness of about 0.4 mm, as the test sample. A Nicolet iS10 Fourier transform infrared spectrometer (Thermo Fisher Technology (China) Co., Ltd., Shanghai, China) was used to scan the sample. The scanning wave number was 4000 to 400 cm^{-1}, the resolution was 4 cm^{-1}, and the scanning times were 16.

Water absorption test

The 24 h water absorption of composite materials was determined according to GB/T 1934.1 (2009). The sample was dried and weighed (m_0, g), then placed into a container containing 23.0 ℃ ± 1.0 ℃ distilled water, soaked for 24 h ± 1 h, and then the sample was removed, the water on the surface of the sample was quickly wiped off with a clean dry cloth or filter paper, and each sample weighed (m, g) again, accurate to 0.1 mg. After the sample was taken out of the water, it should be weighed within 1 min. The 24 h water absorption (A, %) was calculated using Eq. 1,

$$A\ (\%) = [(m - m_0) / m_0] \times 100 \quad (1)$$

where m_0 and m denote the mass of the sample before and after immersion, respectively.

Scanning electron microscopy

Gold was sprayed on the tensile section of the sample by ion sputtering instrument (Beijing Hetong Venture Technology Co. LTD, Beijing, China) and the micro morphology of the section was observed with Quanta FEG250 scanning electron microscope (FEI Company, Hillsboro, OR, USA).

RESULTS AND DISCUSSION

Mechanical Properties of ASA Modified Eucalyptus/PVC Composites

Figure 1 shows the influence of ASA content on the mechanical properties of eucalyptus/PVC composites. Figure 1 (a) shows that with the increase of ASA content, the tensile strength and bending strength of eucalyptus/PVC composites increased first and then decreased. When the ASA content was 10 wt%, the tensile strength of eucalyptus/PVC composite was 32.5 MPa, and the bending strength was 53.3 MPa. Compared with the

eucalyptus/PVC composite without ASA, the tensile strength was 24.4 MPa, and the bending strength was 44.6 MPa, which represents increases by 33% and 19.6%, respectively. When ASA content was 15 wt%, the tensile strength of eucalyptus/PVC composite was 29.2 MPa, and the bending strength was 50.3 MPa, which were 11.5% and 6% lower than that of the composite with ASA content of 10 wt%, respectively. It may be that when the styrene acrylonitrile copolymer (SAN) with shell structure in ASA has high rigidity, the tensile strength and bending strength of the composite can be improved by adding a small amount of ASA. With the increase of ASA content, the proportion of rubber core particles poly(butyl acrylate) (PBA) gradually increased. As the stress concentration point, the rubber phase promotes the yielding of the composite under low stress (Zhou *et al.* 2014). At the same time, the tensile strength of ASA material itself is low, which reduces the tensile strength and bending strength of the composite.

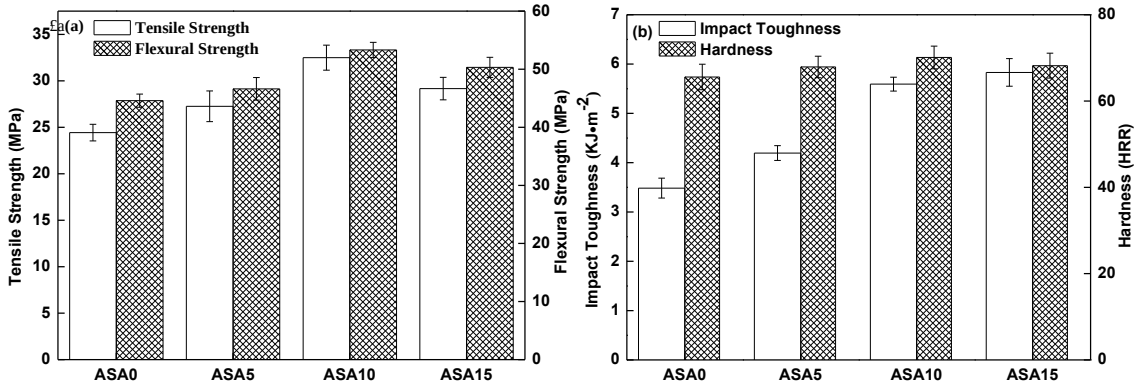


Fig. 1. Mechanical properties of ASA modified eucalyptus/PVC composite

Figure 1(b) shows that with the increase of ASA content, the impact strength of eucalyptus/PVC composites increased gradually. When ASA content was 15 wt%, the impact strength was 5.83 KJ/m^2, which is 67.5% higher than the composite without ASA. This shows that ASA plays an obvious role in toughening the composite. On the one hand, the nuclear structure of PBA in ASA can induce a large number of crazes, which can dissipate energy in the form of stress concentration, and many crazes interfere with each other and stop their expansion (Du *et al.* 2012). On the other hand, as a rigid material, the shell structured SAN in ASA will also absorb part of the energy, which will improve the impact strength of the composite. With the increase of ASA content, the surface hardness of the composite increases. This is because the acrylonitrile and styrene components in ASA materials endow ASA with a certain surface hardness and higher rigidity, which improves the surface hardness of eucalyptus/PVC composites with ASA .

FT-IR Comparison of ASA Modified Eucalyptus/PVC Composites

Figure 2 shows the FT-IR spectra of ASA modified eucalyptus/PVC composites. It can be seen that the shape of the FTIR absorbance peak of ASA modified eucalyptus/PVC composites is similar, but the intensity of the absorbance peak is different. Among them, the 2400 to 2100 cm^{-1} wavenumber segment belongs to C≡N in the molecule, the 1755 to 1670 cm^{-1} wavenumber segment belongs to the carbonyl C=O stretching vibration region, and the 1690 to 1500 cm^{-1} wavenumber segment belongs to the skeleton vibration region of the benzene ring. The above three wavenumber segments are the special vibrational bands of ASA (Ma *et al.* 2012). It can be seen from the intensity of its absorbance peak

that the content of specific functional groups in the composite with ASA content of 15 wt% is the highest. The band at 3300 to 3500 cm^{-1} belongs to the stretching vibration of O-H, while the O-H in the composite mainly comes from cellulose, hemicellulose, polysaccharide, and monosaccharide in eucalyptus wood and ASA with low esterification degree (Xu 2017). The peaks at 2928 and 2857 cm^{-1} correspond to the tensile vibration of methyl, the asymmetric tensile vibration of methylene, and the symmetric tensile vibration of methylene, respectively (Su *et al.* 2016). The peaks at 1737 and 872 cm^{-1} correspond to the stretching vibration of carbonyl C=O and ester –COOR, respectively. The figure shows that the higher the ASA content, the stronger the absorbance peak of the composite in this band, and the higher the ester content, the more hydrophobic the material. The peak at 1428 cm^{-1} corresponds to the conjugated C=C vibration in the benzene ring, and the wavenumber bands 705 cm^{-1} and 615 cm^{-1} correspond to the vibration bands of C-Cl in PVC molecules.

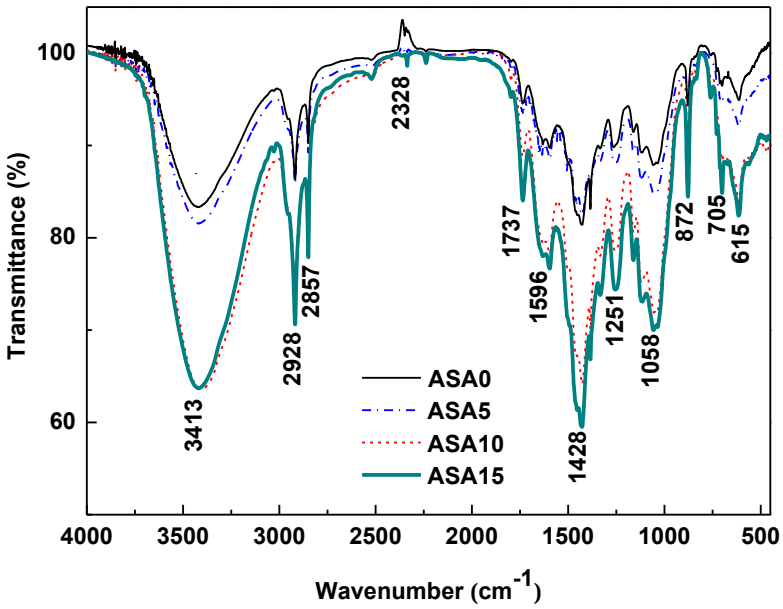


Fig. 2. FT-IR of ASA modified eucalyptus/PVC composites

Thermal Properties of ASA Modified Eucalyptus/PVC Composites

Figure 3 shows the TG and DTG curves of ASA modified eucalyptus/PVC composites, and Table 2 shows the relevant characteristic data of the TG curve. Figure 3 shows that the pyrolysis process of ASA modified eucalyptus/PVC composites with different contents was similar, and the starting temperature, ending temperature, weight loss rate, and residual mass of the pyrolysis reaction were different. Before 100 ℃, the TG curve of the composite decreased slightly and the weight loss was small, which was caused by the evaporation of the water contained in the composite. After 200 ℃, the composites exhibited two order weight loss. In the first stage, the weight loss temperature was 250 to 370 ℃, which was most of the weight loss. The weight-loss substances in this stage are mainly the pyrolysis of cellulose, hemicellulose, and lignin in eucalyptus (Chen *et al*. 2018), shell structure (SAN), and partial core structure (PBA) in ASA polymer (Cao *et al*. 2007), and HCl molecules separated from PVC plastic molecules by thermal decomposition (Liu 2016; Shao *et al*. 2019).

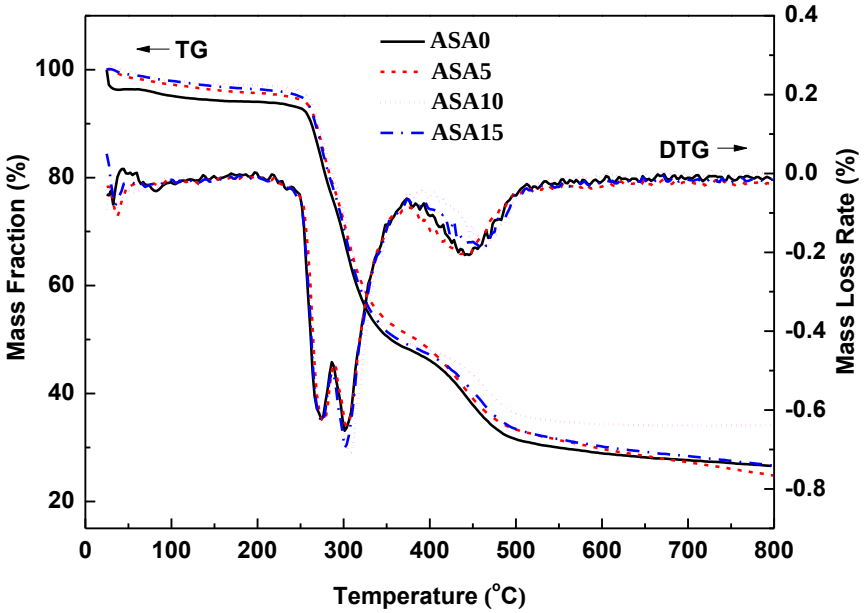


Fig. 3. TG/DTG curve of ASA modified eucalyptus/PVC composite

Table 2 shows that the first order weight loss starting temperature of composites with ASA was slightly higher than that of composites without ASA. When ASA content was 10 wt%, the initial temperature of weight loss in the first stage of eucalyptus/PVC composite was the highest at 255.3 ℃. In the second stage, the weight loss temperature was 380 to 510 ℃, and the weight loss rate was approximately 15%, which was a small part of weight loss. This stage mainly includes the continued pyrolysis of lignin and the nuclear structure (PBA) in ASA, the complete volatilization of residual HCl in PVC, and the fracture thermal decomposition of carbon chain skeleton in PVC molecular chain (Zhang *et al*. 2021). When ASA content was 10 wt%, the second stage weight loss starting temperature of eucalyptus/PVC composite was the highest at 415.8 ℃, which was considerably higher than that of the composite without ASA.

Table 2. Characteristic Data of TG Curve of ASA Modified Eucalyptus/PVC Composite

ASA Content (wt%)	T_1 (°C)	T_2 (°C)	T_3 (°C)	T_4 (°C)	Residual Mass (%)
0	250.5	339.4	383.4	480.7	26.6
5	253.5	348.9	390.9	492.5	24.9
10	255.3	365.1	415.8	508.2	34.1
15	252.5	364.1	408.1	495.6	26.7

Note: T_1 is the starting temperature of the first stage weightlessness, T_2 is the ending temperature of the first stage weightlessness, T_3 is the starting temperature of the second stage weightlessness, and T_4 is the ending temperature of the second stage weightlessness

The comparison of weight loss temperature in two stages showed that ASA can improve the heat resistance of eucalyptus/PVC composites. After 510 ℃, the quality of eucalyptus/PVC composites decreased slightly, and the remaining substances were mainly carbides generated by combustion. The residual mass ratio of ASA/PVC composites with different ASA content was different. When ASA content is 10 wt%, the residual mass of

eucalyptus/PVC composites was the highest at 34.1%. In conclusion, when ASA content was 10 wt%, eucalyptus/PVC composite had the highest residual mass.

Water Absorption Properties of ASA Modified Eucalyptus/PVC Composites

Figure 4 shows the 24 h water absorption of ASA modified eucalyptus/PVC composite. It can be seen from Fig. 4 that the addition of ASA will reduce the 24 h water absorption of eucalyptus/PVC composites. When ASA content was 10 wt%, the water absorption was the lowest, 3.43%, which was 16.9% lower than that of the composite without ASA. On the one hand, PVC has good interfacial compatibility with the shell structure styrene acrylonitrile (SAN) in ASA material, which makes the composite have a denser structure and lower water absorption (Zhang and Zhang 2019). On the other hand, it can be seen from the infrared spectrum analysis of the composite (Fig. 2) that, with the increase of ASA content, although the content of hydrophilic group -OH in the composite increases, the content of hydrophobic group ester group -COOR increases more, so the water absorption of the composite decreases.

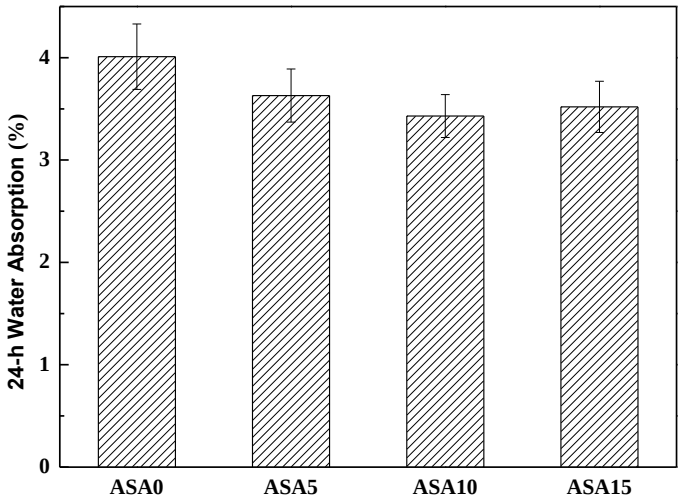
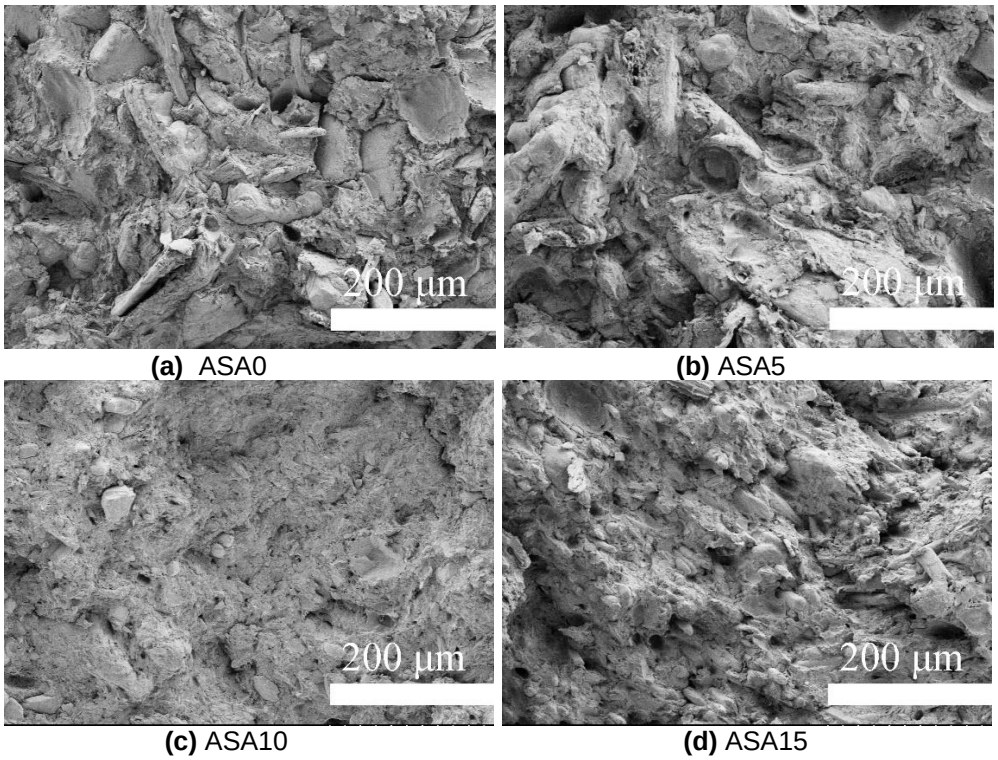


Fig. 4. 24-h Water absorption of ASA modified eucalyptus/PVC composite

Microstructure of Tensile Section of ASA Modified Eucalyptus/PVC Composites

Figure 5 shows the tensile section morphology of eucalyptus/PVC composites modified with different ASA contents. Figure 5(a) shows that the tensile section of the composite without ASA had some aggregated eucalyptus fibers exposed, and there were many voids and defects after the eucalyptus fibers were pulled out. Therefore, the composite without ASA had poor mechanical properties and high water absorption. It can be seen from Fig. 5(b through d) that there was no large amount of fiber exposed on the surface of the matrix in the tensile section of the eucalyptus/PVC composite with ASA added. The eucalyptus fiber was wrapped by the plastic matrix, and the interface compatibility was good, so the composite had good mechanical properties and low water absorption. In addition, there were some small holes in the tensile section of the composite, which were left after the ASA was etched. These holes were isolated and dispersed in the matrix, showing an "island structure". This shows that ASA and PVC did not fuse to form a uniform system, and some ASA are isolated and dispersed in the system. When ASA

content was 15 wt% (Fig. 5(d)), the tensile section became rough and there were many holes, so the water absorption was greater than ASA content at 10 wt%; and part of the wire drawing occurred, indicating that the fracture was ductile fracture, and the impact toughness of the composite material was enhanced.



(a) ASA0 **(b)** ASA5

(c) ASA10 **(d)** ASA15

Fig. 5. Tensile section micromorphology of ASA modified eucalyptus/PVC composite

CONCLUSIONS

1. The addition of acrylonitrile styrene acrylate (ASA) was found to improve the mechanical properties of eucalyptus/poly(vinyl chloride) (PVC) composites and the interfacial compatibility of the composites. When the content of ASA was 10 wt%, the mechanical properties and interface bonding of eucalyptus/PVC composites were the best, and the tensile strength, flexural strength, impact strength, and hardness of the composites were 33%, 19.58%, 60.6%, and 6.9% higher than those of the composites without ASA. The water absorption in 24 h was 16.9% lower than that of the composite without ASA.
2. The Fourier transform infrared (FTIR) spectra of eucalyptus/PVC composites showed both ASA and base material peaks, which indicates that the ASA modification enhanced the matrix cladding for the fiber.
3. The addition of ASA was found to improve the heat resistance of eucalyptus/PVC composites. When the content of ASA was 10 wt%, the heat resistance of eucalyptus/PVC composite was good, the initial decomposition temperature of the two stages was 255 ℃ and 416 ℃, and the residual mass was 34.1%.

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