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STUDY ON PREPARATION AND PROPERTIES OF ANTI-ULTRAVIOLET AGING WOOD-PLASTIC COMPOSITES

ZHITAO LEI[1,2], JIE LIU[1,2], ZHIYUAN ZHANG[1,2], XUESONG ZHAO[1], QI LI[1,2]

[1]INNER MONGOLIA AGRICULTURAL UNIVERSITY
[2]INNER MONGOLIA KEY LABORATORY OF SANDY SHRUBS FIBROSIS AND ENERGY DEVELOPMENT AND UTILIZATION
CHINA

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ABSTRACT

The degradable wood-plastic composites (WPC) were prepared by compression molding in this study. Polylactic acid (PLA), poly (butylene adipate-co-terephthalate) (PBAT) and salix powder were used as the main raw materials and nano-titanium dioxide (nano-TiO_2) was used as anti-ultraviolet filler. The results show that when the addition amount of nano-TIO_2 was 2%, the static bending strength and elastic modulus of WPC reach 41.88 MPa and 3730 MPa, respectively, which can meet the commercial application of WPC in building formwork. At this time, the composite material has a better effect of absorbing and reflecting ultraviolet light. The static bending strength, elastic modulus, tensile strength and impact strength of WPC were reduced by 68.3%, 61.5%, 51.9% and 57.4%, respectively. The mass loss rate and water absorption were 6.1% and 22.6%, respectively, that shows its good degradation performance. This study provides a low-cost and simple method for the design of anti-UV aging, high-performance and degradable WPC, which has broad application prospects in packaging, construction and other fields.

KEYWORDS: Salix, PLA, PBAT, hydrolysis, ultraviolet radiation.

INTRODUCTION

Resin raw materials used in the wood plastic industry are mostly non-degradable, and the waste is difficult to degrade in nature, thus endangering the ecological environment (Durmaz

et al. 2022, Zhao et al. 2021). Polylactic acid (PLA) is a thermoplastic resin with high strength and modulus, good biodegradability and environmental friendliness. In recent years, use of PLA instead of non-degradable resin to prepare high-strength and degradable wood-plastic composites has gradually become a research hotspot. However, PLA has defects such as high cost, hardness, toughness and poor heat resistance, which limits its application (Dylan et al. 2022). Poly(butylene adipate-co-terephthalate) (PBAT) is a synthetic co-polyester with excellent degradability, good high temperature resistance and good tensile strength. It is often used to enhance the heat resistance and toughness of polymers (Radhakrishnan et al. 2022). Bianchi et al. (2023) prepared a PLA/PBAT blends by melt blending method. Thermogravimetric analysis showed that the thermal stability of PLA was improved by PBAT. The stiffness and strength decrease with the increase of PBAT content, the ductility was significantly increased. The shape memory properties of PLA were reduced by PBAT due to the low interfacial adhesion in the blends. Tong et al. (2022) prepared PLLA/PDLA/PBAT ternary composites by melt blending as well. The results showed a good comprehensive performance at 6%, of the PBAT content. The mass loss rate and elongation at break at Vicat softening temperature (VST), alkaline (pH=12) were 166°C, 21.6% and 4.40%, respectively. Compared with the pure PLLA sample, the VST, mass loss rate and elongation at break were increased by nearly 5%, 13% and 20%, respectively. The heat resistance, hydrolysis resistance and toughness of PLA was enhanced by PBAT, but the tensile strength decreases.

With the deepening of the research field of wood-plastic composites, some achievements have been made in the research on the interface characteristics and performance optimization of single resin-based wood-plastic composites, but there are relatively few studies on multi-resin-based wood-plastic composites (Mahesha et al. 2018, Sabirova et al. 2019).

WPC products are mostly used in outdoor environments, and are vulnerable to the combined effects of environmental factors such as high and low temperature, oxygen, humidity, ultraviolet light, microbial corrosion and acid-base erosion during use (Patrick et al. 2022). The material has surface cracking, brittleness, discoloration and mechanical properties degradation. With the increasing degree of aging, its performance is gradually reduced, which seriously shortens the service life of wood plastic products (Sara et al. 2021). It was found that adding inorganic nanoparticles to WPC can effectively improve its aging resistance (Małgorzata et al. 2018). As an inorganic modified material, titanium dioxide (TiO_2) has good UV shielding and thermal stability. The addition of TiO_2 to WPC can endow it with ultraviolet radiation resistance, and further realize the functionalization and high value of the material (Luo et al. 2018). The effect of accelerated weathering degradation on the properties of polylactic acid (PLA)/polycaprolactone (PCL) blends and PLA/PCL/titanium dioxide (TiO_2) nanocomposites was introduced by Luyt et al. (2021). The results showed that the presence of TiO_2 delayed the degradation of PLA and PCL. Thermogravimetric analysis confirmed that PLA and PCL affected the thermal degradation of each other, and TiO_2 played a role in the thermal degradation of PLA and PCL. Both PLA/PCL and PLA/PCL/TiO_2 significantly reduced the tensile properties by weathering exposure and TiO_2 incorporation.

At the same time, degradable WPC is a new type of environmentally friendly material. It contains a large number of modifiers that delay aging and degradation, and cannot be rapidly

degraded after losing use value (Rapa et al. 2019). Therefore, the disposal of WPC after abandonment is still worthy of attention. PLA/PBAT films were prepared by Wang et al. (2023). The 30-day aging behavior of the films under air irradiation, water, ultraviolet irradiation, water and ultraviolet irradiation was investigated. The results showed that the carbonyl index (CI) of PLA/PBAT film in water decreased from 3.84 to 1.36 under UV irradiation. The oxygen carbon ratio (O/C) decreased to 0.49 at 30 d. It shows that the oxygen-containing functional groups have broken bonds, showing a rapid aging process. This is mainly due to the combined effect of hydrolysis and photolysis, which increases the contact area of PLA/PBAT films and accelerates the aging process. Correa-Pacheco et al. (2019) prepared PLA and PBAT extruded fibers with cinnamon essential oil (CEO) and explored their degradation properties. The results showed that with the addition of PBAT and CEO, the Young's modulus of the blends decreased and the elongation at break increased. During the water degradation, the weight loss rate of the fibers was less than 5%. Microscopic cracks are formed on the surface of the fibers, especially PLA fibers in an alkaline medium at 37°C.

In order to problems mentioned above, degradable and aging-resistant WPC were studied and the aging resistance, composite mechanism and degradation law of WPC were explored. This research meets the application requirements of degradable WPC in different environments.

MATERIAL AND METHODS

Experiment material

PLA (3251D) was provided by Nature Works, USA and PBAT (C1200) by BASF, Germany. Salix wood-flour (the particle size is 40~60 mesh) was supplied by Man Lai Township Or-dos City Inner Mongolia, China. Coupling agent (KH550) was provided by Nanjing Chuang Shi Chemical Auxiliary Co., Ltd, China. Stearic acid was supplied by Xilong Science Co., Ltd, China and ethanol (95 wt%) by Tianjin Feng Chuan Chemical Reagent Technology Development Co., Ltd, China. Nano-TiO_2 was supplied by Beasley New Materials (Suzhou) Co., Ltd, China, NaOH by Tianjin Hui hang Chemical Technology Co., Ltd, China.

Preparation of wood-plastic composites

The design density of WPC was 1.2 g/cm^3 for designed size of 15×15×4 mm. The total mass of WPC was created by the sum of PLA, PBAT, salix powder and nano-TiO_2 (Tab. 1). The mass ratio of salix powder to resin (PLA and PBAT) was 1:1. The fixed amount of coupling agent (KH550) was 3 wt%, the fixed amount of stearic acid was 0.6 wt%, the fixed amount of nano-TiO_2 was 2 wt% (wt.% was the percentage of the total mass of WPC).

Tab. 1: Formula of nano-TiO_2 modified PLA/PBAT/salix composite.

Samples	Hydrolysis time	PLA	PBAT	Salix powder	Nano-TiO_2	Stearic acid
WPC-0	0	25	25	50	2	0.6
WPC-1	48 h	25	25	50	2	0.6
WPC-2	96 h	25	25	50	2	0.6
WPC-3	182 h	25	25	50	2	0.6
WPC-4	384 h	25	25	50	2	0.6

KH550 was hydrolyzed with 95 wt% ethanol, mixed with modified wood flour and nano-TiO_2 respectively, and dried for later use. The temperature of the double roller of the mixer was raised to 170°C and 180°C, and PLA, PBAT, modified wood powder and modified nano-TiO_2 were added in turn for uniform mixing. Finally, the mixture was chopped and put into the mold, which was formed by hot pressing and cold pressing. The hot pressing temperature, pressure and time were 185°C, 5 MPa and 7 min, respectively. The cold pressing pressure and time were 7 MPa and 10 min.

The specimens were cut and put into the ultraviolet aging test box. The aging test was carried out according to GB/T14522-2008. The irradiation intensity was 0.82 W/m^2, the constant temperature was 32°C, and the exposure section was dried for 8 h and condensed for 4 h.

NaOH and deionized water were used to prepare a degradation solution with a pH=11. On this basis, the effects of hydrolysis time 0.48 h, 96 h, 192 h and 384 h on the composites were investigated. The obtained WPC were named WPC-0, WPC-1, WPC-2, WPC-3, WPC-4, respectively.

Equipment and instruments

According to GB/T17657-2013, the static bending strength and modulus of elasticity were tested by Electronic universal testing machine (Model, WDW-20A, Jinan Tianchen Machinery Manufacturing Co., Ltd., China). According to GB/T1040-2006, the tensile strength was tested by a Mechanical testing machine (Model, AG-IC, Shimadzu Instruments (Suzhou) Co., Ltd. China). According to GB/T1043.1-2008, the impact strength was tested by a Pendulum impact tester (Model, ZBC7151-B, Meters Industrial Systems Co., Ltd. China). Microstructure images of samples were obtained by scanning electron microscopy (Phenom Pro, Funa Scientific Instruments (Shanghai) Co., Ltd. Holland). A sample used to study the effects of ultraviolet (UV) radiation was exposed to an UV aging instrument (Q8-UV3, Q-Panel company, USA). The pH value of the hydrolysate was measured by a pH meter (model, STARTER3100, China).

Experimental method

Mass loss rate test

Before the hydrolysis experiment, the sample was accurately weighed, and then placed in the hydrolysate for a period of time, taken out, cleaned with distilled water, and then placed in a blast drying oven for 12 h, taken out and weighed, and calculated the mass loss rate. The mass loss rate is calculated according to Eq. 1:

$$C = （W_1\text{-}W_2） / W_1 \times 100 \quad (\%) \qquad （1）$$

where: C-mass loss rate (%); W_1 - initial mass of sample before degradation (g); W_2 - mass of sample after degradation (g).

Water absorption test

Before the hydrolysis experiment, the sample was accurately weighed, and then placed in the hydrolysate for a period of time, taken out, and dried the WPC surface water stains, weighed, and calculated the water absorption rate. The water absorption rate is calculated according to Eq. 2:

$$M = (Q_2\text{-}Q_1) / Q_1 \times 100 \quad (\%) \qquad (2)$$

where: M-water absorption (%); the initial mass of Q_1 sample before water absorption (g); Q_2-the mass of the sample after soaking in the solution for a period of time (g).

RESULTS AND DISCUSSION

Ultraviolet resistance properties

When the addition amount of TiO_2 was 0 or 2%, the changes of static bending strength and elastic modulus of WPC before and after UV aging for 240 h were shown in Fig. 1. After UV irradiation for 240 h, static bending strength and elastic modulus of WPC with 0% TiO_2 addition decreased by 22.2% and 23.6%, respectively. The static bending strength and elastic modulus of WPC with 2% TiO_2 addition decreased by 2.8% and 4.5%, respectively, and the decrease was small. The anti-ultraviolet aging properties were increased by 19.4% and 19.1%, respectively, compared with those without TiO_2. This is because WPC is endowed with the ability to absorb and scatter ultraviolet light by TiO_2. The UV aging resistance is improved, which slows down the aging rate of the material to a certain extent. But it does not completely eliminate the damage caused by ultraviolet light.

Adding appropriate amount of TiO_2 to WPC, the nanoparticles were uniformly dispersed in the composite system without agglomeration (Liu et al. 2019). The van der Waals force and hydrogen bonding between nanoparticles are weak, and the mechanical properties of the material are enhanced. TiO_2 has the characteristics of absorbing and shielding ultraviolet light. It is mainly absorbed by electronic transition. Collect most of the ultraviolet radiation energy. It is then released in the form of energy or heat without damaging the material properties (Xu et al. 2015). Comprehensive analysis, when the amount of TiO_2 is 2 wt%, after 240 h of ultraviolet aging, the static bending strength and elastic modulus of WPC are 40.67 MPa and 3560 MPa, respectively, which can still meet the requirements of use, and the anti-ultraviolet aging effect is the best.

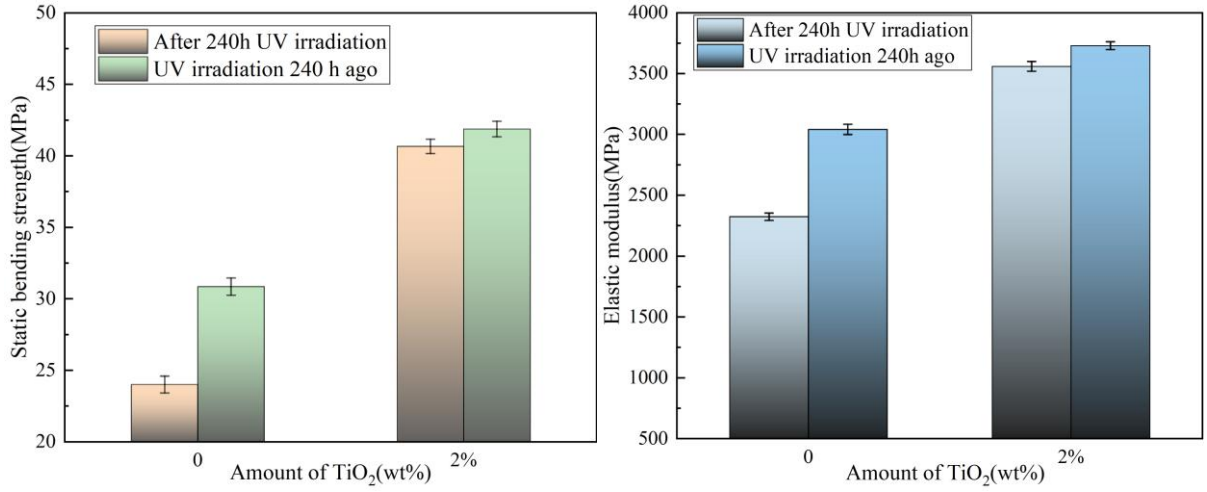

Fig. 1: Effect of different TiO_2 content on static bending strength (a) and elastic modulus (b).

Mechanical properties

The change trend of mechanical properties of WPC after water degradation is shown in Fig. 2.

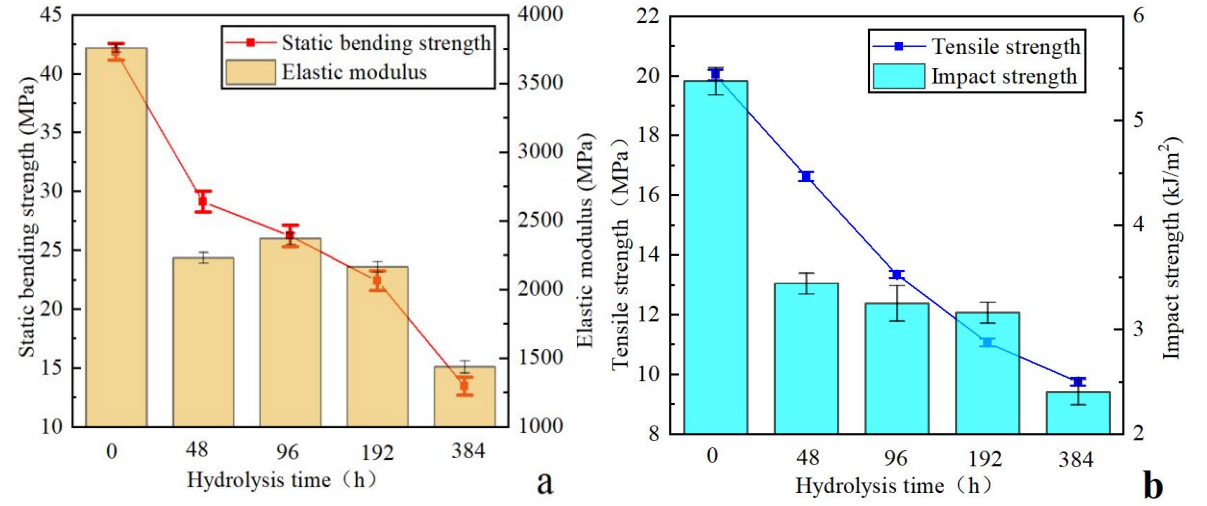

Fig. 2: Effect of hydrolysis time on static bending strength and elastic modulus (a), effect of hydrolysis time on tensile strength and impact strength (b).

It can be seen from Fig. 2 that the mechanical properties of WPC decreased with the extension of hydrolysis time. When the time is 384 h, the static bending strength of WPC was 13.16 MPa, the elastic modulus was 1436 MPa, the tensile strength was 9.75 MPa, and the impact strength was 2.40 kJ/m^2. Compared with unhydrolyzed, the above properties decreased by 68.3%, 61.5%, 51.9% and 57.4%, respectively. This is because the ester bonds in the PLA molecular chain are easily degraded by alkaline solution. The acidic oligomers produced by PLA degradation can be neutralized by OH^- in the alkali solution. The autocatalytic effect of PLA hydrolysis was promoted. After hydrolysis of PBAT, terephthalic acid and adipic acid react with OH^-, which promotes the forward hydrolysis reaction. The degradation time was prolonged,

and a large number of holes and defects appear in the material, resulting in interface debonding and mechanical properties decreasing (Ratanawilai et al. 2022, Tasdemir et al. 2020).

Water absorption and quality loss rate

The change trend of water absorption and mass loss rate of WPC after hydrolysis time is shown in Fig. 3. It can be seen from Fig. 3 that the mass loss rate and water absorption rate of WPC increased with the extension of hydrolysis time. When the degradation time was 384 h, the mass loss rate and water absorption rate were 6.1% and 22.4%, respectively. This is due to the prolonged degradation time, PLA and PBAT hydrolysis to produce a large number of soluble oligomers and small molecules. The movement and diffusion of water molecules were accelerated, and the degradation rate of WPC was increased (Durmaz et al. 2020). At the same time, lignin, hemicellulose and pectin were dissolved and diffused into the solution, resulting in serious mass loss of WPC.

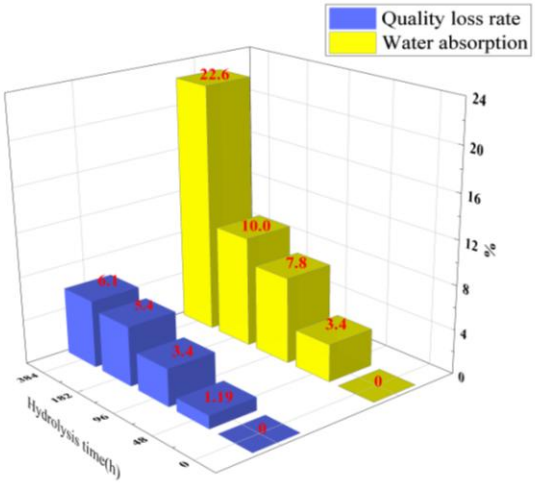

Fig. 3: The effect of hydrolysis time on the water absorption and mass loss rate of WPC.

The hydrolysis of PLA and PBAT promotes the formation of microcracks and penetration cracking inside the material, which promotes the diffusion of water molecules. At the same time, the content of hydrophilic terminal carboxyl group and terminal hydroxyl group provides more adsorption points for water molecules, and the water absorption of the composites increases. Wood flour has the characteristics of dry shrinkage and wet expansion (Jiang et al. 2020). It will expand and deform after absorbing water, which will produce stress on the resin matrix, resulting in deformation and gap inside WPC. With the passage of time, water molecules continue to diffuse into the interior of the material, and the water absorption rate gradually increases.

pH value of hydrolysate

The change trend of pH value of hydrolysate is shown in Fig. 4. It can be seen that the pH value of the hydrolysate decreased with the extension of degradation time.

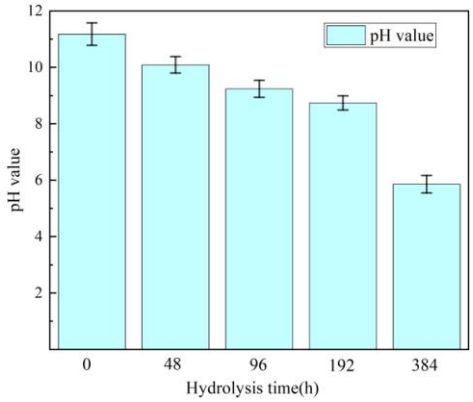

Fig. 4: The effect of hydrolysis time on the pH value of hydrolysate.

When the time was 384 h, the pH value of the hydrolysate decreased to 5.86. This is because PLA is an aliphatic polyester, and the ester bonds on the molecular chain are hydrolyzed to produce a large amount of soluble lactic acid and other acidic small molecules. The alkaline of the hydrolysate was neutralized by chemical reaction with OH^- in the solution (Matthews et al. 2018). PBAT will form acid after reacting with OH^- in the hydrolysis process, and the formed acid will be further decomposed into $RCOO^-$ under the action of OH^-. The pH value of the solution was further reduced. With the extension of degradation time, the OH^- in the solution was gradually consumed by the acidic degradation products. The amount of H^+ increases, and the solution is weakly acidic (El-fattah et al. 2018).

Scanning electron microscope analysis

The surface morphology of WPC at different degradation time is shown in Fig. 5. The surface is smooth and flat without cracks and gullies when the immersion time is 0 h. When the immersion time rises to 48~96 h, cracks and gullies gradually appear on the surface (Fig. 5b,c).

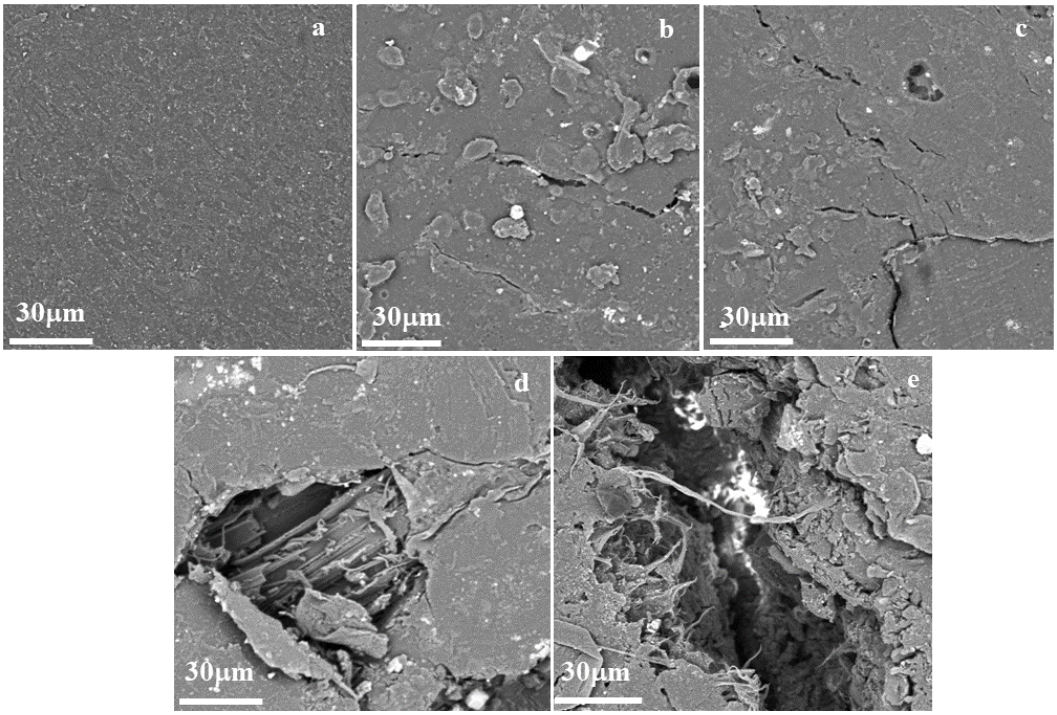

Fig. 5: The microstructure of surface of WPC with different hydrolysis time: a) 0, b) 48 h, c) 96 h, d) 192 h and e) 384 h.

At 192~384 h, the cracks and holes on the surface of the material gradually become larger and deeper (Fig. 5d,e). A large number of wood fibers were separated from the resin and wrapped and exposed. During the hydrolysis process, the resin molecular chain is eroded by water molecules and breaks to form a small molecular chain, resulting in the destruction of the inherent entanglement and crosslinking between the polymer molecular segments (Petchwattana et al. 2017, Machado et al. 2016) Therefore, this damage is expanded with time, resulting in increased cracks and holes in WPC and decreased mechanical strength.

CONCLUSIONS

In this work, degradable wood-plastic composites with ultraviolet radiation resistance and high thermal stability were prepared by compression molding. After adding nano-TiO_2 to the WPC composite system, the UV absorption and scattering ability of WPC increased, and the UV aging rate slowed down. With the extension of hydrolysis time, the mechanical properties decreased significantly, the mass loss rate and water absorption rate increased, and the pH value of the hydrolysate decreased. When the degradation time was 384 h, the static bending strength, modulus of elasticity, tensile strength and impact strength of WPC decreased by 68.3%, 61.5%, 51.9% and 57.4%, respectively; the mass loss rate and water absorption rate were 6.1% and 22.4%, respectively; the pH value of the hydrolysate decreased from 11.0 to 5.68. SEM analysis showed that the corrosion of aqueous solution made the smooth surface of WPC become rough,

the interfacial compatibility between the internal components became worse, and a large number of holes and cracks appeared, and the degradation degree was further deepened.

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ZHITAO LEI, JIE LIU, ZHIYUAN ZHANG, QI LI*
[1]COLLEGE OF MATERIAL SCIENCE AND ART DESIGN
INNER MONGOLIA AGRICULTURAL UNIVERSITY
HOHHOT 010018, CHINA
[2]INNER MONGOLIA KEY LABORATORY OF SANDY SHRUBS FIBROSIS AND ENERGY DEVELOPMENT AND UTILIZATION
HOHHOT 010018, CHINA
*Correspondent author: li_qi555@163.com

XUESONG ZHAO*
COLLEGE OF MECHANICAL AND ELECTRICAL ENGINEERING
INNER MONGOLIA AGRICULTURAL UNIVERSITY
HOHHOT 010018, CHINA
*Correspondent author: zhao_xuesong555@163.com