

Periodic Hygrothermal Properties of *Salix* Liquefaction Products / Isocyanate Rigid Foam Materials

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Under specific circumstances, the liquefaction product of biomass polyol can be converted into hard polyurethane foam. In this study, the rigid foam material (RPUF) was created by reacting the liquefaction product of *Salix psammophila* with isocyanate. The mechanical properties, chemical structure, cell structure, and thermal stability of RPUF were characterized by mechanical properties tests, Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA). The impact strength, compressive strength, and flexural strength of RPUF after hygrothermal treatment showed a downward trend. FTIR showed that the change of temperature and humidity did not affect the chemical structure of RPUF. SEM showed that the destruction of cell structure of RPUF was increased with the extension of time. TGA showed that the initial decomposition temperature of RPUF was reduced by periodic hydrothermal treatment, and the residual carbon rate decreased with the extension of treatment time.

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INTRODUCTION

Polyurethanes (PU) are polymers containing urethane (-NHCOO-) repeating structural units. Polyurethane foams can be divided into rigid polyurethane foams (RPUF) and soft polyurethane foams (FPUF). RPUF can be used in construction because of its excellent thermal insulation properties, and FPUF can be used in the home (mattress) industry because of its softness (Akindoyo *et al.* 2016; Wang *et al.* 2021). Polyurethanes are generally made of diisocyanates (*e.g.* MDI) and petroleum-based polyols as the primary raw materials. More and more attention has been paid recently to the technology of preparing polyurethane from bio-based polyols.

As an environmentally friendly material, biomass material has the advantages of avoiding pollution; it is easily degradable and renewable. Bio-based polyols prepared by liquefaction of wood and straw could be used instead of petroleum-based polyols to prepare polyurethane materials (Zhang *et al.* 2007; Fidan and Ertaş 2020; Sun *et al.* 2020). They could solve the problems of shortage of fossil fuels and have the potential for the production and synthesis of polyurethane materials. However, rigid polyurethane materials are often impacted by hot and humid environments in the fields of construction and insulation, resulting in aging of the materials and reduced performance (Ostrogorsky *et al.* 1986; Liu *et al.* 2005).

RPUF are often used in buildings and insulation materials. It is important to study the aging process of RPUF under different temperatures and humidity to save energy and improve people's life quality (Liu *et al.* 2005; Sienkiewicz *et al.* 2019). After a no-load accelerated temperature and humidity aging test, the compressive strength of RPUF does not decrease considerably. The compressive strength dramatically decreases following the load-accelerated temperature and humidity aging test (Yan *et al.* 2012). Thus, the mechanical properties of RPUF are affected by temperature and humidity. Under the condition of temperature and humidity change, the increase of humidity will promote the hydrolysis of ester groups, so that the RPUF density becomes smaller, the thermal conductivity increases, and the thermal insulation performance decreases. With increased temperature, the elongation at break decreases rapidly (Peng *et al.* 2015). However, the test conditions are too simple to reflect the specific effects related to the usage of RPUF. The aging process of materials exposed to weather can be better simulated by cyclic changes in temperature and humidity. This method has been utilized in research on wood aging. With the change of temperature and humidity, the mechanical properties of wood will be reduced, but there is almost no chemical change of wood (Qing *et al.* 2017; Kytka *et al.* 2022). Therefore, it is of certain significance to study the mechanical strength and chemical structure of RPUF under the condition of alternating temperature and humidity.

In this study, *Salix psammophila* was used as raw material for polyol liquefaction, and then the liquefaction products/isocyanate rigid foam material (RPUF) was prepared by adding MDI and foaming. The cyclic hygrothermal degree was simulated to establish six temperature and humidity change stages. The relationship between hygrothermal effect and mechanical strength change at different stages and different cycles was analyzed based on the microscopic morphology of cells' morphology and chemical structure change, and the response mechanism theory of hygrothermal effect of microporous materials under the coupling effect of moisture, heat, and force was constructed. This research provides a theoretical basis for improving the aging resistance of polyurethane foam materials in the future, and it also provides a new type of biomass-based polyurethane insulation material for heating cities in northern China.

EXPERIMENTAL

Materials

The original *Salix psammophila* was taken from Xinminpu Village, Dalate Banner, Ordos City (China), Inner Mongolia Autonomous Region, and the trunk of 1000 mm in length was cut from 20 mm above the root. The main components of *Salix* are shown in Table 1. The PEG-400 was supplied by Tianjin Fengchuan Chemical Reagent Technology Co., Ltd. (Tianjin, China). The glycerol was supplied by Tianjin wind boat chemical reagent technology Co., Ltd. (Tianjin, China). The concentrated sulfuric acid was supplied by Shanghai Lynn Technology Development Co., Ltd. (Shanghai, China). The montmorillonite (OMMT) was supplied by Shandong Yousuo Chemical Technology Co., Ltd. (Shandong, China). The isocyanate (MDI), tin isooctanoate, and trimethylolpropane (TMP) were supplied by China Pharmaceutical Group. (Beijing, China).

Preparation of *Salix* liquefaction products

The original *Salix psammophila* was cut into a section of 2 cm by a cutter and put into a grinder to prepare *Salix* wood powder. Polyethylene glycol, glycerol, and *Salix* wood

powder were mixed in three flasks with a mass ratio of 64 : 16 : 20. The three flasks were immersed in an oil bath for liquefaction reaction, with liquefaction time of 140 min (OH = 345 mg KOH/g), liquefaction temperature of 170 °C, and stirring rate of 300 r/min (Zhou and An 2015).

Table 1. Chemical Compositions of *Salix*

Component	Wt%
Ash	2.87
Cold water extract	9.24
Hot water extract	10.01
1% NaOH extract	32.11
Benzyl alcohol extract	6.20
Holocellulose	72.71
Hemicellulose	22.81
Lignin	19.72

Preparation of Salix liquefaction / isocyanate rigid foam products (RPUF)

The manufacturing process of rigid polyurethane is shown in Fig. 1. The mass ratio of *Salix* liquefaction product, isocyanate, stannic octoate, montmorillonite, distilled water, and trimethylolpropane was 100 : 110 : 18 : 12 : 2 : 2. According to the one-step method, various foaming additives were added into the *Salix* liquefaction product, and isocyanate was added after uniform stirring. After about 15 s small white bubbles appeared, and they were poured into the 200 × 100 × 50 mm mold. After molding, the foam material was solidified in a 75 °C drying oven for 60 min and then aged at room temperature for 24 h (Han *et al.* 2020). The prepared foams (foam density 0.084 g/cm³ Zhou *et al.* 2017) were cut into specified sizes according to the national standard measurement methods of different intensities and dried in an oven at 50 °C for further use.

Methods

Stage hygrothermal test

The hot and humid environment of northern China (Hohhot) was simulated. The relative temperature was selected as 5 °C in low temperature, 14 °C in medium temperature, 25 °C in medium temperature, and 40 °C in medium temperature. The relative humidity was selected as 35% in low humidity, 50% in medium and low humidity, 60% in medium humidity, 70% in medium and high humidity, and 85% in high humidity. The six stages are shown in Table 2. The changes of temperature and humidity in each stage were warming and dehumidifying, warming and dehumidifying, isothermal humidification, isohumidifying warming, cooling and dehumidifying, and cooling and dehumidifying. The constant temperature and humidity box equipment (Shanghai Yiheng Scientific Instrument Co., Ltd., Shanghai, China) was used for treatment, and each stage was treated for 48 h. The changes of mechanical strength and chemical structure after each stage were studied.

Table 2. Hygrothermal Aging Conditions

Stages	Stage 1	Stage 2	Stage 3	Stage 4	Stage 5	Stage 6
Relative Temperature and Humidity	5 °C 65%	14 °C 50%	25 °C 70%	25 °C 85%	40 °C 85%	14 °C 35%

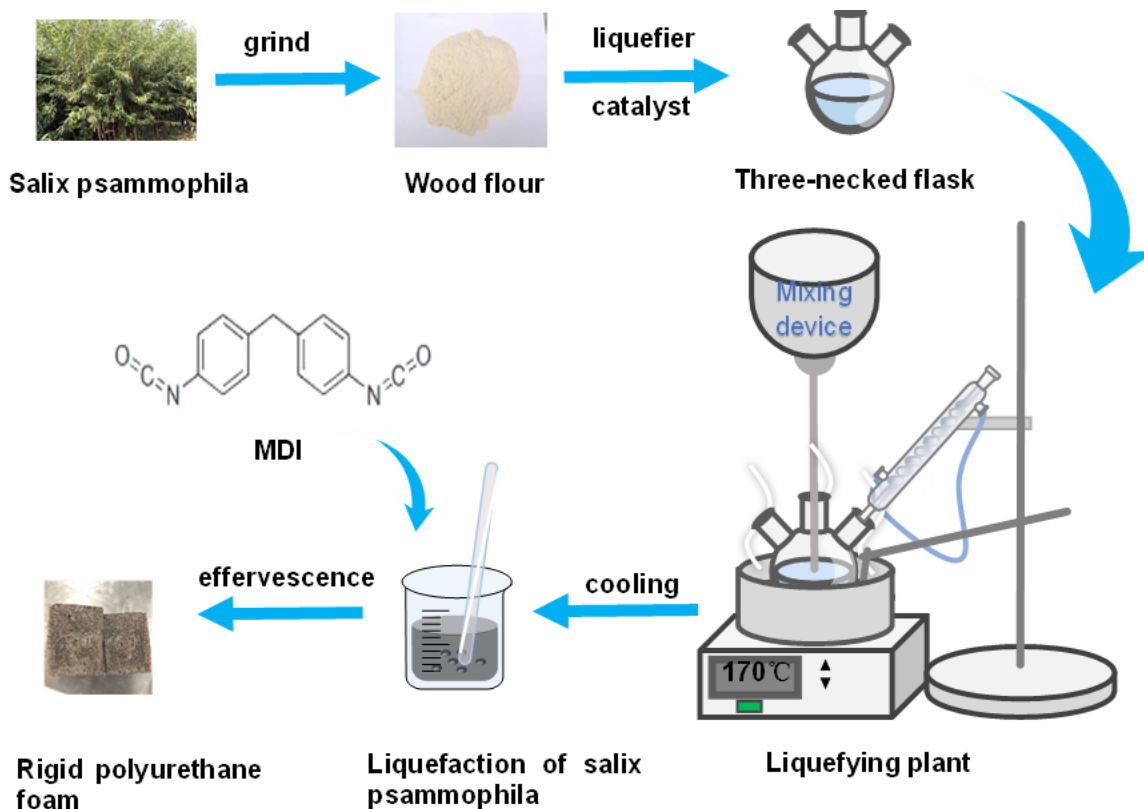


Fig. 1. Manufacturing flow chart of rigid polyurethane

Cycle hygrothermal test

Six stages were set as one cycle, and the material was treated for four consecutive cycles. The treatment durations of each cycle are shown in Table 3. After each cycle, the changes of mechanical strength, pore morphology, chemical structure and thermal decomposition were tested.

Table 3. Hygrothermal Time of Cycle

Cycle	Cycle 1	Cycle 2	Cycle 3	Cycle 4
Processing time	12 d	24 d	36 d	48 d

Physical strength measurement

Impact strength was measured according to GB/T 1043-1993. Compression strength was measured according to GB/T 8813-2020 with a compression rate of 2 mm/min. Bending strength refers to GB/T 8812.2-2007, compression rate 5mm/min. Averages were obtained based on 5 samples per group.

Fourier transform infrared (FTIR) characterization

A TENSOR 27 Fourier transform infrared spectrometer (Bruker, Germany) was used. The powder sample was pressed with potassium bromide, and the scanning range was from 500 to 4000 cm^{-1} .

Scanning electron microscopy (SEM)

Using S-4800 field emission scanning electron microscope (Hitachi Japan), the sample was adhered to the copper table with conductive double-sided adhesive, and the surface of the sample was vacuum sprayed with gold at a voltage of 10 kV.

Thermogravimetric (TG) characterization

Tests were conducted with a DT-60 thermal analyzer (Shimadzu, Japan), powder sample, nitrogen atmosphere, with a heating rate of 10 °C/min. The heating range was from 50 to 800 °C.

RESULTS AND DISCUSSION

Effect of Stage Hygrothermal on Mechanical Strength

Effect of stage hygrothermal treatment on impact strength

The changes of material impact strength under different stages of treatment are shown in Fig. 2. The impact strength of the material decreased slightly from the first stage of warming and dehumidifying to the second stage, because at relatively low temperature, the activity of water molecules was low, so the energy change of the material was small when it was impacted. From the second stage of warming and humidification to the third stage, the temperature and humidity increased in a small range, and the impact strength decreased significantly, with a decrease of about 32.6%. This is because the surface of polyurethane foam material was aged, so the impact strength of the material decreased. From the third stage to the fourth stage, when the temperature remains unchanged, the humidity increases by 15%, and the decrease of impact strength is not obvious. This may be because although the humidity increases in a small range, the area of contact water molecules on the material surface is saturated at 25 °C, so the aging degree of the material is not obvious in a short time.

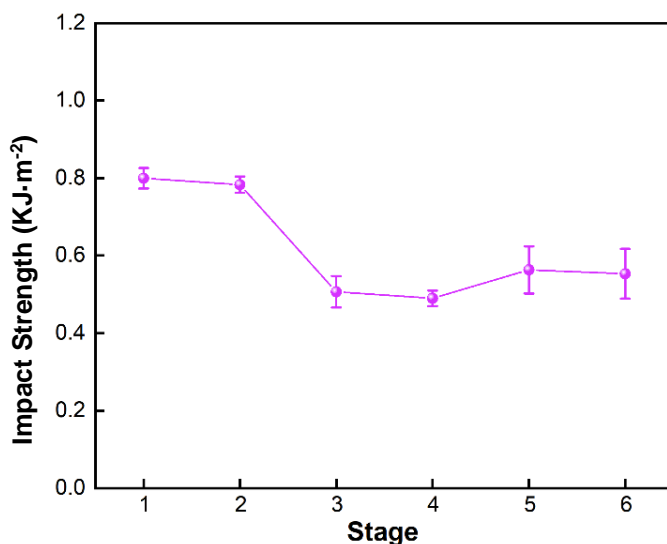


Fig. 2. RPUF impact strength change curve at different stages

From the fourth stage to the fifth stage, the impact strength increased, and the recovery rate was approximately 12.2%. When the temperature rose to 40 °C, it was already in the middle and high temperature stage. As the temperature increased, the degree of hydrogen bonding increased and the surface of the material became viscoelastic. From the fifth to sixth stages, the impact resistance of the material surface did not change significantly.

Effect of the stages of hygrothermal treatment on compressive strength

The change in compressive strength under different stages of treatment is shown in Fig. 3. The compressive strength of the material decreased slightly from the first stage to the second stage. From the second stage of warming and humidification to the third stage, the compressive strength of the foam material decreased by 15.2%, which was attributed to the simultaneous increase of temperature and humidity, which led to the aging of the foam. As a consequence, it was easy to cause large-scale extrusion of bubbles in the foam, so the compressive strength decreased significantly. From the isothermal humidification in the third stage to the fourth stage, the compressive strength of the material did not change significantly; from stage 4 to stage 5, the compressive strength of the material rebounded with a recovery rate of 12.5%, because the high temperature can induce a thermal aging reaction inside the polyurethane and make the crosslinked network structure more closely integrated. From stage 5 to stage 6, the compressive strength decreased by 5.6%, because the material was in a relatively dry environment at low temperature and humidity, which would cause the material to change from toughness to brittleness.

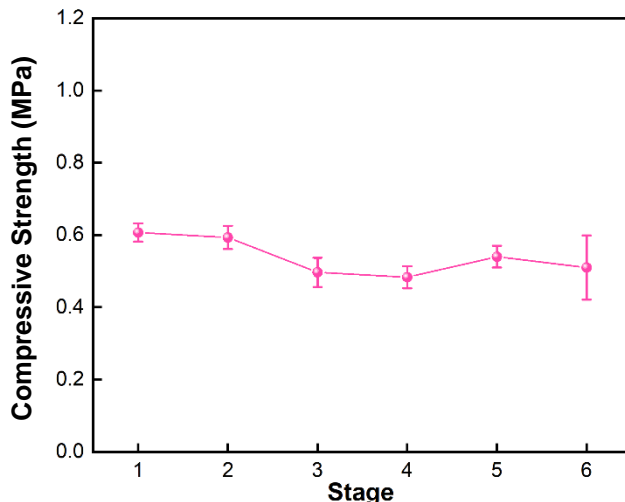


Fig. 3. RPUF compression strength change curve at different stages

Effect of stages of hygrothermal treatment on bending strength

The change of bending strength of materials under different stages of treatment is shown in Fig. 4. The overall change trend of material bending strength was similar to that of impact and compression strength. From the first stage to the second stage, the bending strength decreased by 11.5%. From the second stage of warming and humidification to the third stage, the bending strength continued to decrease by 16.7%. From the third stage of isothermal humidification to the fourth stage, the bending strength did not change. From the fourth stage to the fifth stage, the bending strength increased by 37.8%, and the increase

was large. This was because when the temperature rose to the middle and high temperature stage, the toughness of the material was enhanced under the bending load, resulting in a larger bending fracture value. From stage 5 to stage 6, the bending strength decreased by 8%.

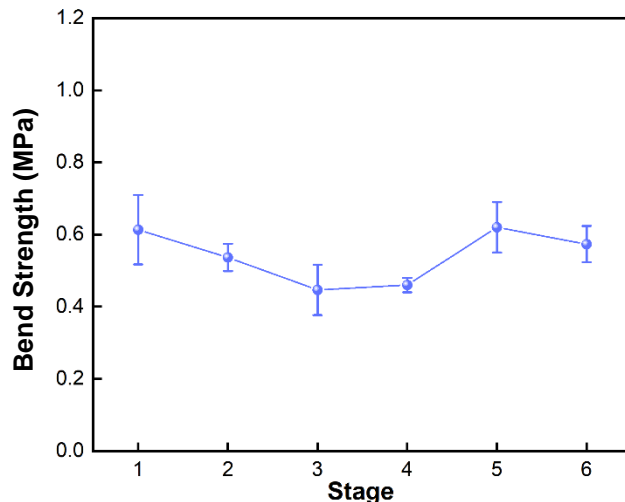


Fig. 4. RPUF bending strength change curve at different stages

Effect of Stage Hygrothermal on Chemical Structure

The infrared spectra of RPUF at different stages are shown in Fig. 5. The trend of material spectra was basically similar at different stages. In the second and third stages, the transmittance of -OH group was large at 3200 to 3700 cm^{-1} , which may be due to the large amount of water molecules contacted on the surface of the material in a relatively high humidity environment at 25 °C and 70% and 85% relative humidity, respectively.

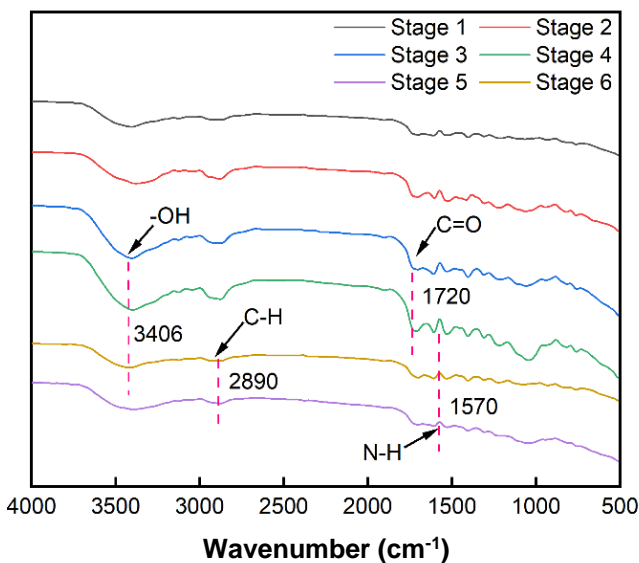


Fig. 5. FT-IR of RPUF at different stage

Under phases 1, 5, and 6, the intensity of C-H stretching vibration peak on-CH₂ and-CH₃ was weakened near 2890 cm⁻¹. The C=O near 1720 cm⁻¹ represents the basic carbon structure of polyurethane rigid foam. The absorption peaks of C=O stretching vibration are enhanced in the third and fourth stages, indicating that the hydrolysis reaction of ester group is stronger (Li *et al.* 2020). In the fifth and sixth stages, the N-H stretching vibration peak of carbamate at 1570 cm⁻¹ weakened (Zieleniewska *et al.* 2016). In the infrared spectrum, the strength change of each characteristic peak reflects the mechanical strength change at different stages, but no new characteristic peak was found, indicating that there was no new chemical structure change.

Effect of Cyclic Hygrothermal Treatment on Mechanical Strength

Effect of cyclic hygrothermal treatment on impact strength

The change of impact strength of materials in dry state and four cycles is shown in Fig. 6. The impact strength of the material showed a downward trend after four cycles (48 d). After the first cycle, the impact strength of the material decreased the most, from 0.89 to 0.55 KJ/m², and the strength decreased by 38.2%. With the extension of aging time, the impact strength continued to decline after the second cycle, and the strength loss was only 9.1%. After the third cycle, the impact strength rebounded slightly, with a rebound rate of 4%; after the fourth cycle, the impact strength decreased by 11.5%. After four cycles of alternating hydrothermal treatment, the impact strength decreased by 48.3%. The above analysis shows that the impact strength of *Salix* liquefiable product/isocyanate foam material treated with alternating hydrothermal cycles did not always keep a downward trend, and there was a slight recovery process after the third cycle. The impact strength recovery decreased with the extension of treatment cycle.

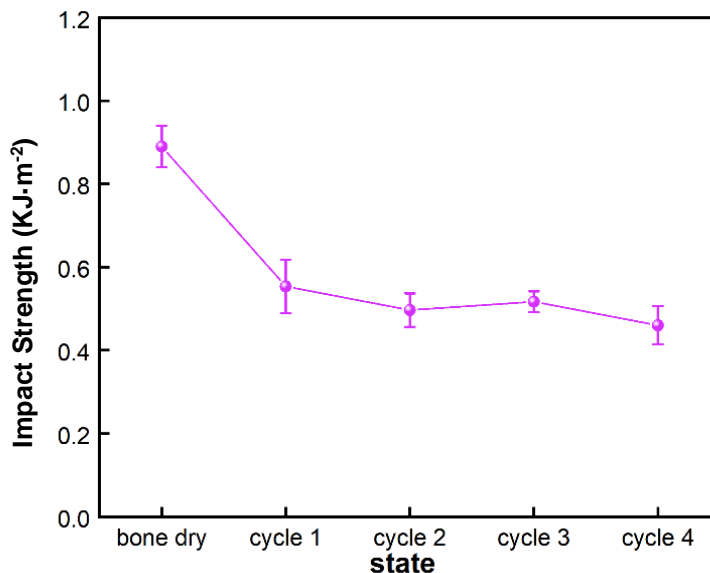


Fig. 6. Impact strength curve of RPUF in different states

Effect of cyclic hygrothermal treatment on compressive strength

Figure 7 shows the changes of the compressive strength of the material after dry state and four cycles of treatment. The compressive strength of the material decreased after different cycles of hot and humid alternate aging. After the first cycle, the compressive strength decreased greatly to 0.51 MPa, which was 35.4% lower than that in the dry state.

After the second cycle, the compressive strength decreased slowly, and the compressive strength decreased to 0.48 MPa, which was 5.9% lower than that of the first cycle. After the third cycle, the compressive strength continued to decrease, and the compressive strength decreased to 0.45 MPa, which was 6.2% lower than that of the second cycle. After the fourth cycle, the compressive strength decreased to 0.39 MPa, which was 13.3% lower than that of the third cycle. After four cycles of alternating hydrothermal treatment, the compressive strength decreased by 50.6%. The above analysis shows that the effect of alternating hydrothermal cycle treatment on the compressive strength of *Salix* liquefaction products/isocyanate foam materials exhibited a declining trend.

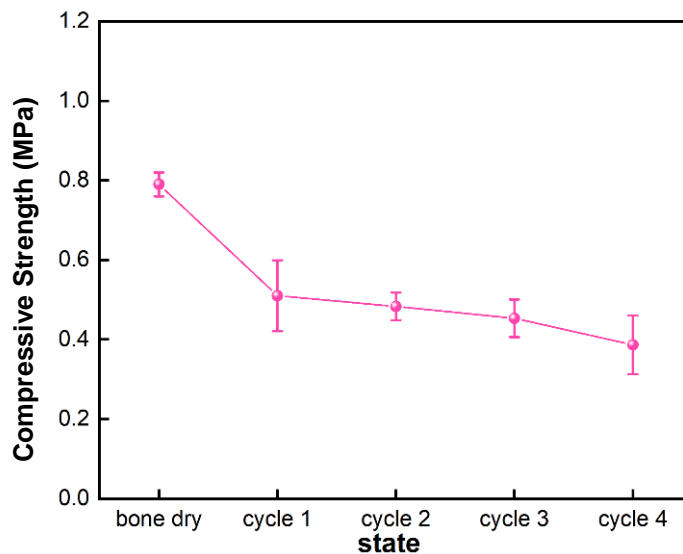


Fig. 7. Compressive strength curve of RPUF in different states

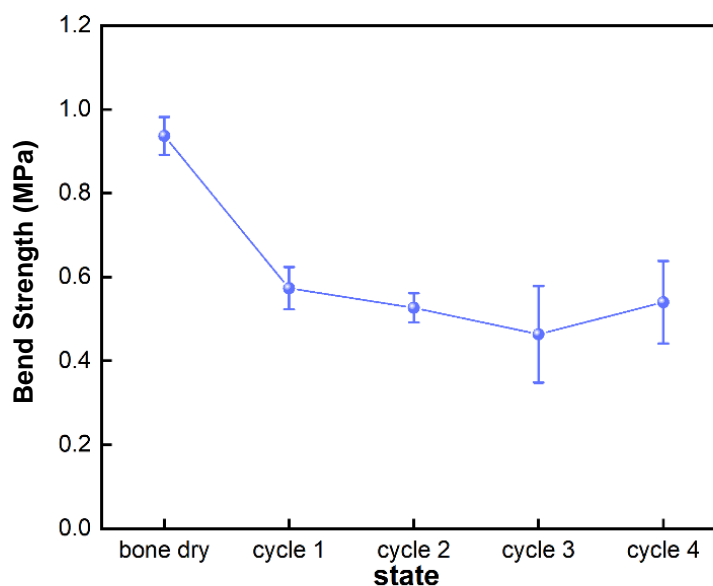


Fig. 8. Bend strength curve of RPUF in different states

Effect of cyclic hygrothermal treatment on bending strength

The change of bending strength of materials after the dry state and four cycles of treatment is shown in Fig. 8. From the dry state to the third cycle, the bending strength of the material maintained a downward trend. After the first cycle, the bending strength decreased greatly from 0.94 to 0.57 MPa, and the bending strength loss was 39.4%. After the second cycle, the bending strength decreased to 0.53 MPa, and the bending strength loss was 7.01% compared with the first cycle. After the third cycle, the bending strength decreased continuously to 0.46 MPa, which was 13.2% lower than that of the second cycle. After the fourth cycle, the bending strength rebounded by 17.4%. After four cycles of alternating hydrothermal aging, the bending strength of polyurethane foam materials showed a downward trend, which was 42.6% lower than that in the dry state. The above analysis shows that the effect of alternating hot and humid cycles on the flexural strength of *Salix* liquefaction product/isocyanate foam material did not always maintain a downward trend, but there was a small recovery process after the fourth cycle. After the third cycle, the compressive strength decreased by 51.1%.

Effect of Cyclic Hygrothermal on Chemical Structure

The infrared spectra of the materials after dry state and four cycles are shown in Fig. 9. The water evaporated in the dry state, and the vibration of -OH group at about 3500 cm^{-1} moved to the high position. With the extension of aging cycle, the peak type of -OH group broadens, but the transmittance decreased. The C-H group vibrations associated with -CH₂ and -CH₃ near 2950 , 2920 , and 1400 cm^{-1} gradually decreased (Servay *et al.* 2000; Stuart 2004), and so did the C=O stretching vibration absorbance near 1720 cm^{-1} decreases (Larsen *et al.* 2009). Although periodic damp heat does not make *Salix* liquefaction product / isocyanate foam material to form a new chemical structure, it will affect the strength of each characteristic peak, which will result in a mechanical strength difference.

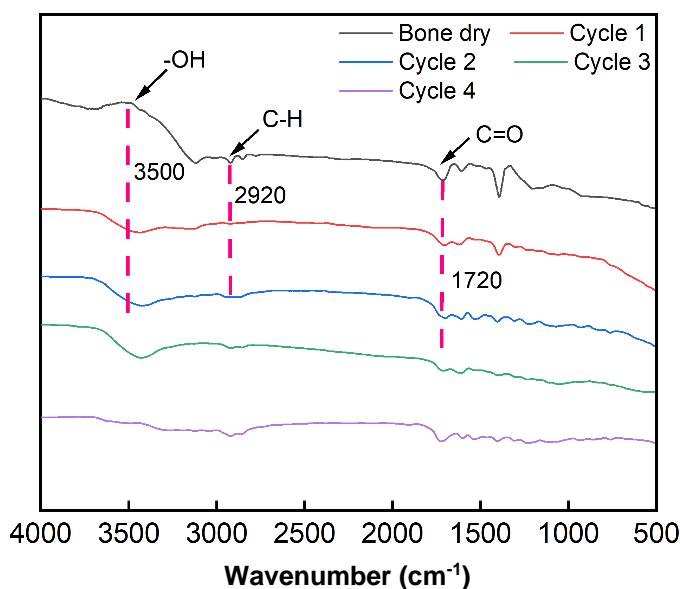


Fig. 9. FT-IR of RPUF in different states

Effect of Cyclic Hygrothermal on Foam Structure

The mechanical strength of RPUF is closely related to the structure of the cell, and the thinner wall and smaller spine result in lower compressive strength (Czlonka *et al.* 2018; Liu *et al.* 2019). The dry state and the SEM of the material after four cycles of treatment are shown in Fig. 10. It can be seen from Fig. 10a that the majority of cells was closed. Figure 10b shows that the pore structure of polyurethane foam was relatively complete and the surface was smooth in the dry state. With the extension of hydrothermal treatment time, the foam aging, degradation, wrinkles occurred on the surface of the bubble hole, some thin hole wall fell off (10c). With the continuation of processing time, the cell surface wrinkles seriously (10d). With continuation of treatment to the third cycle, the bubbles burst (10e). By the end of the fourth cycle, the foam spine degraded severely and fractured (10f).

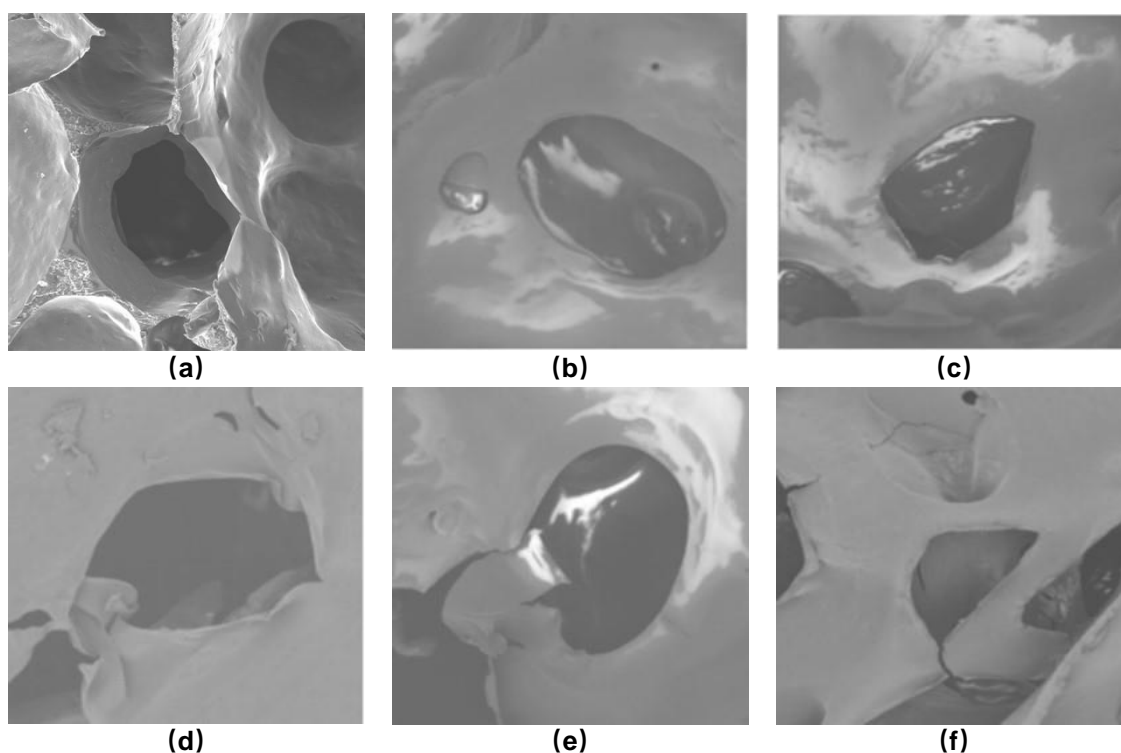


Fig. 10. SEM of RPUF in different cycles

Effect of Cyclic Hygrothermal Treatment on Pyrolysis Behavior

TGA results corresponding to the pyrolysis of the material after dry state and four cycles of treatment are shown in Fig. 11. The related parameters of thermal decomposition are shown in Table 4. The pyrolysis process of RPUF under dry condition and after four cycles of treatment was mainly divided into four stages. The first stage is 25 to 250 °C, and this process is mainly volatilization of bound water (Wang *et al.* 2022). The second stage is 250 to 480 °C, in this process, the fourth cycle weight loss rate was higher, because with the extension of aging time in RPUF system hydrogen bond fracture, macromolecular main chain pyrolysis to produce more CO₂, MDI, and alcohol (Hablott *et al.* 2008). The third stage is 480 °C to 700 °C; this stage mainly the decomposition of carbon residue layer (Yarmohammadi *et al.* 2018). The thermal stability of the third and fourth cycle materials was very different from that of other materials. The fourth stage is 700 to 800 °C. In the

dry state, the first cycle, and the second cycle the weight loss rate remained in a relatively stable range, while the third cycle and the fourth cycle weight loss rate continued to decline. The initial decomposition temperature of RPUF decreased significantly after different cycles of treatment, but the temperature was higher after the fourth cycle treatment when the weight loss was 50%. With the extension of treatment time, the final carbon residue rate of RPUF decreased after different cycles, which was due to the degradation of hard segments in RPUF system caused by the extension of aging time, which led to the reduction of final residues (Bradai *et al.* 2022).

Table 4. TG Measurement Data of RPUF

State	Initial Weight Loss Temperature (°C)	Temperature at 50 % Weight Loss (°C)	Carbon Residue at 800 °C (%)
Bone dry	50.48	334.1	32.39
Cycle 1	46.44	334.49	31.53
Cycle 2	38.05	329.65	30.41
Cycle 3	36.1	325.94	28.13
Cycle 4	25.96	336.52	24.44

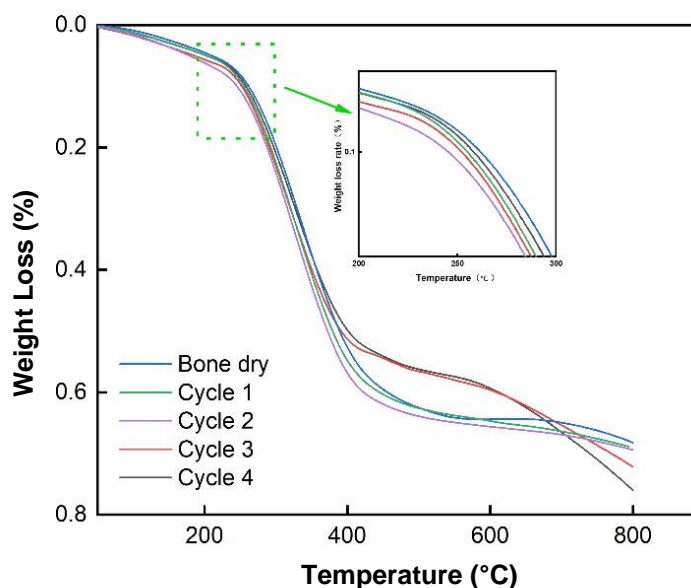


Fig. 11. TG curve of RPUF in different states

CONCLUSIONS

1. The mechanical properties analysis of rigid polyurethane foam (RPUF) under different stages of elevated temperature and relative humidity treatments showed that the impact strength, compressive strength and bending strength of RPUP decrease first and then increase under different stage conditions. As shown by Fourier transform infrared analysis (FT-IR), the spectra of RPUF at different stages were basically similar. After different temperature and humidity treatment stages, RPUF did not induce any chemical changes.

2. The mechanical properties analysis of RPUF after dry state and four cycles of treatment showed that from dry state to the end of the cycle, the impact strength, compressive strength, and flexural strength showed a decreasing trend overall, but the impact toughness and flexion did not maintain a decreasing tendency. Impact strength was restored after the third cycle and bending strength after the fourth. The FT-IR results showed that the cyclic hygrothermal effect did not cause the formation of new chemical structure of RPUF, but it affected the strength of each characteristic peak and caused the mechanical strength gap. Scanning electron microscopy (SEM) results showed that the foam structure in dry condition was relatively complete. With the extension of the thermal treatment time, the aging of foam occurred degradation, the foam hole wrinkled, a part of the thin hole wall fell out, and even the foam holes and foam spine fractures occurred. The thermogravimetry analysis (TGA) showed that the initial decomposition temperature of RPUF was reduced by the cyclic hydrothermal treatment, and the residual carbon rate decreased with prolongation of the treatment duration.

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