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Preparation and Performance Analysis of Urea-Formaldehyde/Cellulose Blended Foam as a Low-Cost, Degradable Water-Absorbing Material for Agricultural Soil Water Retention

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Abstract

In view of the shortcomings of low-cost urea-formaldehyde foams (UFF), such as high brittleness, severe pulverization and shrinkage, poor water absorption, water retention and reusability, which limit their application in agricultural water conservation, this paper adopts water-soluble urea-formaldehyde resin (UF) as the matrix and hydroxypropyl methylcellulose (HPMC) as the toughening agent to construct a "KH550 modified hydrophilic nano-silica (HSN) reinforced + pre-absorbed small particle size superabsorbent resins (SAPs) functionalized" ternary synergistic modification system. Through mechanical foaming technology, a degradable UF/cellulose composite foam water-absorbing material was prepared. Under the optimal ratio, the water absorption capacity of the composite material reached 29.31 g/g, the water retention time exceeded 19 days, and the water absorption recovery rate after 10 water absorption-drying cycles still reached 66%; at the same time, the compressive strength was increased to 0.352 MPa, and the powdering rate was as low as 3.35%, achieving a synergistic improvement in mechanical properties and water absorption performance compared to pure UFF. This ternary system solves the technical problems of traditional UFF's easy cracking and SAPs' swelling-induced collapse of the pore structure. The material preparation process is simple, cost-effective, and biodegradable, and has good application prospects in agricultural soil water retention and water-saving irrigation fields, providing new ideas for the development of green and sustainable water-absorbing materials.

Keywords: Urea-formaldehyde foam; Superabsorbent resins; Hydroxypropyl methylcellulose; Hydrophilic nano-silica; Composite foam; Agricultural soil water retention

1 Introduction

At present, the global water shortage problem is intensifying, and high-performance absorbent

materials are widely used in agricultural water conservation, health care, wastewater treatment, and other fields due to their excellent water absorption and water retention properties [1, 2].

Although the most widely used superabsorbent resins (SAPs) can absorb water more than 1,000 times, they rely on petrochemical raw materials such as acrylamide and acrylic acid, which are difficult to degrade and have high production costs [3]. Although traditional natural absorbent materials (such as pure cellulose and starch) are environmentally friendly, they have defects such as poor mechanical properties and easy swelling and collapse when exposed to water [4], while urea-formaldehyde foams have the advantages of light porosity, degradability and low price [5], but they have problems such as high brittleness, poor water resistance, and formaldehyde residue, which limit their application in the field of water absorption. In this context, the urea-formaldehyde and cellulose-based composite system has gradually attracted attention due to its eco-friendly characteristics, but there are still obvious gaps in existing research, and it is difficult to meet the requirements of comprehensive material performance in agricultural soil water retention and industrial wastewater treatment.

Existing studies have mostly focused on the mechanical modification of urea-formaldehyde/cellulose blends, and the synergistic optimization mechanism of porous structure regulation and water absorption performance is still unclear [6, 7]. Nanoparticles (such as HSN) are prone to agglomeration when used to enhance the properties of materials [8], and it is difficult to meet the needs of high water absorption applications by relying solely on cellulose toughening. At the same time, the interface compatibility, porous structure design and performance synergy optimization path of each component have not been systematically revealed, and the balance between material lightweight and high strength in process regulation is still a technical difficulty.

In view of the above research gaps, in this study, urea-formaldehyde resin was used as the matrix, hydroxypropyl methylcellulose (HPMC) was used as the toughening and hydrophilic modifier, combined with KH550 modified HSN reinforcement and SAPs functionalization, and a multi-stage composite foam absorbent material was constructed through mechanical foaming process.

The innovations of this study are mainly reflected in the following: first, with sustainable

resource utilization and environmental friendliness as the core, mechanical foaming technology is used to prepare absorbent materials by cross-linking degradable cellulose and water-soluble urea-formaldehyde polymers; second, K_{12} is used as the foaming agent and MPS is used as the foam stabilizer to effectively improve the elasticity and strength of the foam system. The third is to build a dual modification system of "HSN reinforced hole wall + SAPs swelling and water locking" to solve the problem of hole structure collapse caused by the swelling of traditional SAPs. Fourth, the hydrogen bonding network enhancement mechanism between HPMC and urea-formaldehyde was revealed, as well as the interaction between the silicon hydroxyl group on the surface of HSN and the carboxylic acid group of SAPs, and the physical limiting effect of HSN on the swelling behavior of SAPs.

In this way, this research achievement not only promotes the high-value utilization of urea formaldehyde and cellulose-based materials, but also provides new ideas and theoretical basis for the development of low-cost, degradable and water-absorbent materials, which is conducive to the sustainable utilization of agricultural soil water retention and water resources.

2 Materials and Methods

2.1 Materials

Anhydrous ethanol of chemical purity, silane coupling agent (KH550) of chemical purity, phosphoric acid (H_3PO_4) of chemical purity, formaldehyde of analytical purity 37%, all were purchased from Haodi Reagent Distribution Co., Ltd. Hydroxypropyl methylcellulose (HPMC) has a viscosity η of 6980 mPa·s, purchased from Shandong Ruitai Chemical Co., Ltd. Urea of chemical purity, purchased from Beijing Chemical Factory. Sodium dodecyl sulfate (K_{12}) of chemical purity, purchased from Gongyi Lanteng Environmental Protection Technology Co., Ltd. Silicone resin polyether emulsion (MPS) of chemical purity, purchased from Suzhou Yanzhihan Trading Co., Ltd. Sodium hydroxide (NaOH) of chemical purity, purchased from Leichen Building Materials Distribution Office in Kaifeng District. Deionized water of chemical purity, made by the laboratory itself. Hydrophilic nano-silica (HSN) with a particle size

of 40-50 nm, purchased from Kelongsheng New Materials Co., Ltd. Superabsorbent resins (SAPs) 160-180 mesh, purchased from Zhenghe Chemical Co., Ltd.

2.2 General Test Methods and Formulas

2.2.1 Determination of liquid absorption capacity

A dry sample with dimensions of 20 mm × 20 mm × 20 mm was placed in a 500 mL beaker, and 300 mL of tap water was poured into the beaker to completely submerge the sample. The temperature of the electric heating forced air constant temperature drying oven was set at 25 °C. The sample was taken out every 4 h, placed on a 100-mesh sieve, and allowed to stand for 10 min to filter out excess moisture; thereafter, the sample was weighed and the data were recorded. Calculations were performed according to Eq.1.

$$W = \frac{W_t - W_d}{W_d} \quad (1)$$

W : Absorption ratio, g/g;

W_t : Mass of the sample after water absorption and expansion, g;

W_d : Mass of the dried sample, g.

The method for measuring the liquid absorption performance of the sample in deionized water and 0.9% NaCl solution is the same, and the data are all the average values of the 3 parallel experiments set for each group of experiments[9].

2.2.2 Determination of water retention performance

A dry sample with dimensions of 20 mm × 20 mm × 20 mm was placed in a 500 mL beaker, and 300 mL of tap water was poured into the beaker. The temperature of the drying oven was set at 25 °C, and the sample was allowed to stand under this condition for 24 h until it reached the water absorption saturation point. Subsequently, the sample was taken out and placed in a drying oven at 25 °C for drying. Every 12 h, the sample was taken out, placed on a 100-mesh sieve, and allowed to stand for 10 min to filter out excess water; the sample was then weighed and the data were recorded.

The corresponding calculation formula is shown in Eq.2.

$$W_b = \frac{W_m}{W_1} \times 100\% \quad (2)$$

W_b : Water retention rate, %;

W_m : The amount of liquid absorbed by the absorbent material at different times, g;

W_1 : The amount of liquid absorbed by the absorbent material in the saturated state, g.

The method for measuring the liquid absorption performance of the sample in deionized water and 0.9% NaCl solution is the same, and the data are all the average values of the 3 parallel experiments set for each group of experiments[10].

2.2.3 Determination of water absorption recovery performance

A dry sample with dimensions of 20 mm × 20 mm × 20 mm was placed in a 500 mL beaker, followed by the addition of 300 mL of deionized water. The temperature of the drying oven was set at 25 °C, and the sample was allowed to stand for 12 h. The water absorption rate was calculated according to Eq. (1). Subsequently, the sample was dried, and the above operation was repeated 10 times, with the water absorption rate calculated for each cycle. All data represent the average values of three parallel experiments conducted for each group [11], and the corresponding calculation formula is shown in Eq.3.

$$Q_n = \frac{W_n}{W_2} \times 100\% \quad (3)$$

Q_n : Water absorption recovery rate, %;

W_n : Maximum water absorption rate of the water-absorbing material on the nth water absorption, g/g;

W_2 : Maximum water absorption rate of the water-absorbing material on the first water absorption, g/g.

2.2.4 Abrasion rate test

A foam water-absorbing sample with dimensions of 20 mm × 20 mm × 20 mm was placed on 400 –mesh sandpaper, followed by the application of a load of 100 g weight. The sample was moved 10 roundtrips on the sandpaper (one way 15 cm).

All data represent the average values of five parallel experiments conducted for each group [12], The powdering rate was calculated according to Eq.4 .

$$P = \frac{M_0 - M_1}{M_0} \quad (4)$$

P : Powder loss rate, %;

M_0 : Initial weight of water-absorbing foam, g;

M_1 : Remaining weight of the water-absorbing material after friction, g.

2.2.5 Compression performance test

The compressive strength was measured according to the method specified in GB/T8813—2008. A sample with dimensions of 50 mm × 50 mm × 50 mm was used, and its surface layer was removed. At least 5 samples were tested to obtain the average value. During the test, the maximum force at which the sample was compressed to 10% of its original height was recorded, and this was used to calculate the compressive strength [13]. The calculation formula is shown in Eq.5:

$$\sigma_m = \frac{F_m}{A_0} \quad (5)$$

σ_m : Compressive strength, MPa;

F_m : Maximum compressive force when the relative deformation is 10%, N;

A_0 : Initial cross-sectional area of the specimen, mm².

2.2.6 Characterization analysis

Scanning electron microscopy (SEM): After drying, the foam absorbent material was fabricated into ~2 mm-thick sheets. Prior to SEM observation, a thin gold layer was sputtered on each sheet, after which morphology observation and pore distribution examination via SEM images at different magnifications.

Fourier transform infrared spectroscopy (FT-IR): In a dry environment, the dried foam absorbent material samples were ground into powder, homogenously mixed with potassium bromide (KBr) at a mass ratio of 1:100, and pressed into transparent pellets. FT-IR spectroscopy was performed in the range of 500/cm to 4000/cm, and the corresponding infrared spectra were obtained. X-ray diffraction (XRD): The absorbent foam

samples, after being dried in a drying oven, were ground into powder. XRD measurements were performed over a scanning range of $2\theta = 10^\circ$ to 60° , at a scanning rate of $5^\circ/\text{min}$, with an output voltage of 40 kV and an output current of 30 mA. Thermogravimetric analysis (TGA): The dried foam absorbent material was ground into powder using a mortar. Approximately 5 mg of the powder was then weighed and placed into an aluminum crucible. TGA was conducted in a temperature range of 50°C to 600°C under nitrogen protection, with a heating rate of $10^\circ\text{C}/\text{min}$.

Raman spectroscopy (RS): The absorbent foam material samples dried in an oven were ground into powder, and the solid powder was uniformly dispersed on a glass slide. Raman spectroscopy measurements were performed using a 532 nm laser (with a $50\times$ objective lens and 0.5 mW power), with an integration time of 10 s per scan, accumulated for $3\times$ consecutive scans. Multi-point scanning was conducted to subtract the background, and the test was carried out in the wavenumber range of 500/cm to 3500/cm to obtain the Raman spectra.

3 Experimental

3.1 Preparation of UF/HPMC Blended Foaming Material

Preparation of UF: First, the formaldehyde solution is placed in a four-necked flask equipped with a condenser, a stirrer, and a thermometer. The temperature is adjusted to 50°C , and the pH is adjusted to 8.5 using NaOH. The first batch of urea is added to the flask to achieve a molar ratio of formaldehyde to urea (F/U_1) of 2.1. The reaction lasts for 30 min. The second batch of urea is then added to the flask, and the $F/(U_1 + U_2)$ ratio is adjusted to 1.6. The temperature is raised to 90°C , and the reaction lasts for 40 min. The pH is adjusted to 5.0 using H_3PO_4 . When the reactants reach a certain viscosity, NaOH is rapidly used to adjust the pH to 8.5, and the temperature is lowered to 60°C . The third batch of urea is added, and the final $F/(U_1 + U_2 + U_3)$ ratio is changed to 1.4. The reaction lasts for 10 min, resulting in the UF pre-polymer solution [14–16].

Preparation of HPMC: Heat 100 mL of deionized water to 85°C [17, 18], add hydroxypropyl methylcellulose (HPMC) and stir at a low speed.

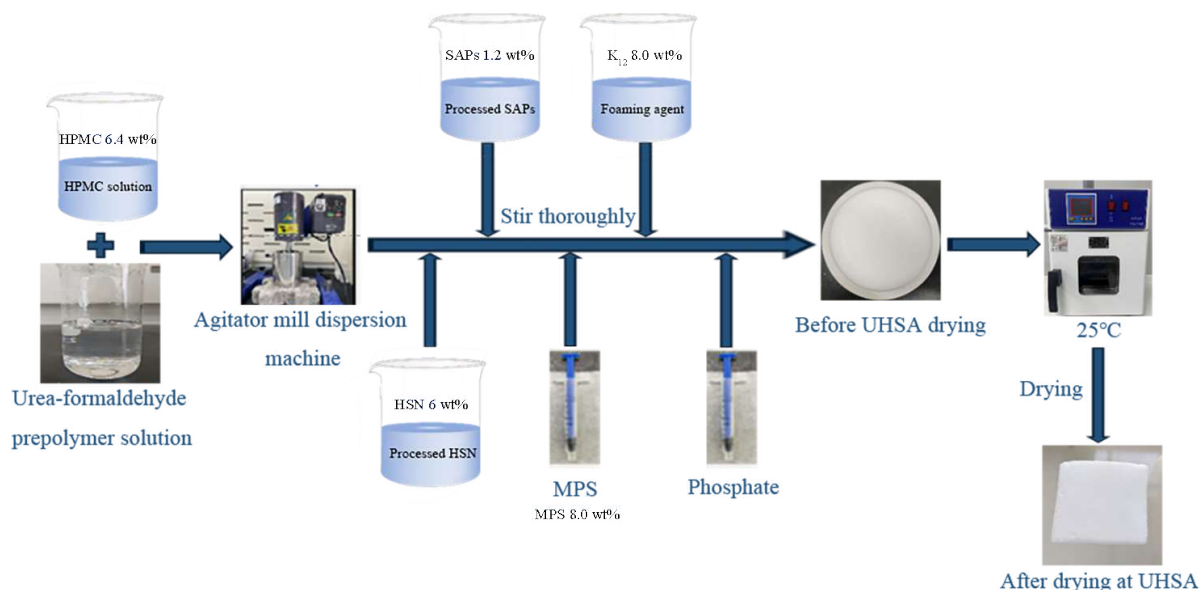


Fig. 1 The overall preparation steps for the ternary modified composite foam (UHSA), along with the optimal ratios of each reagent

After stirring evenly (at this point, take out the mixed gel solution and cure it to obtain the urea-formaldehyde/HPMC cured material, hereinafter referred to as UFH), pour it into a stirring sand-milling disperser. While stirring, add 25 g of the UF prepolymer solution, stir evenly, then add Stabilizer Silicone resin polyether emulsion (MPS) and foaming agent sodium dodecyl sulfate (K_{12}), stir evenly, increase the rotation speed and foam until the foam height no longer changes. Add an appropriate amount of H_3PO_4 (adjust the pH of the system to 5) for curing, and dry at room temperature ($25^\circ C$) to obtain the urea-formaldehyde and cellulose foam water-absorbing material (UFHM).

3.2 Preparation of HSN Modified Foam Absorbent Material

The preparation of HSN foam water-absorbing material is based on the preparation of UFHM, with the addition of HSN. Pre-treatment of HSN: As HSN is prone to agglomeration, it is pre-treated before being added to the foam system. It is added to a beaker containing 50% anhydrous ethanol and 50% deionized water, and KH550 is added. The mixture is then dispersed in an ice water bath using a 500W ultrasonic device

for 1 hour. Preparation of UHMS: The urea-formaldehyde prepolymer solution and HPMC solution are simultaneously added to a stirring sand mill disperser, thoroughly mixed, and then HSN is added and mixed well. The foaming solution and MPS are added for foaming until the foam height no longer changes. An appropriate amount of phosphoric acid is quickly mixed in, and the mixture is poured into a mold for curing. The mold is then placed in a $25^\circ C$ oven to dry, resulting in a dry UHMS sample.

3.3 Preparation of SAPs/HSN Blended Foam Absorbent Materials

The preparation of SAPs/HSN blend foam water-absorbing material (UHSA) was based on the preparation of UHMS, with SAPs added. Pre-treatment of SAPs: Before adding to the foam system, SAPs were pre-treated by ball milling in a vertical planetary ball mill for 24 hours. Then, they were added to a beaker containing deionized water until fully saturated with water. After that, KH550 was added, and the mixture was dispersed using a stirring sand mill until no obvious large particles were present. Preparation of UHSA samples: Urea-formaldehyde prepolymer solution and

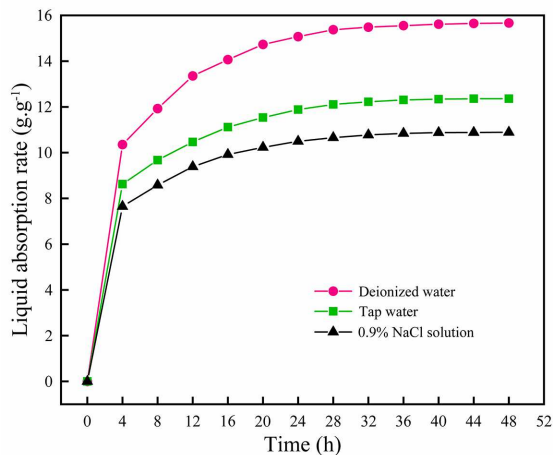


Fig. 2 Liquid absorption performance chart of UFHM

HPMC solution were simultaneously added to a stirring sand mill and thoroughly mixed. Then, HSN and SAPs were added and mixed well. Foaming was carried out by adding foaming agent and MPS until the foam height remained constant. After adding an appropriate amount of phosphoric acid, the mixture was poured into a mold for curing and placed in a 25 °C oven to dry, resulting in dry UHSA samples. As shown in Figure 1.

4 Results and Discussion

4.1 Optimization of Basic UF/HPMC Composite Foam

Through single-factor experiments, the three sets of optimal dosages for HPMC were determined to be 6.0%, 6.4%, and 6.8%; for K_{12} , the three sets of optimal dosages were 8.0%, 8.4%, and 8.8%; and for MPS, the three sets of optimal dosages were 7.6%, 8.0%, and 8.4%. Then, through orthogonal experiments, the optimal blending ratios of the three factors were determined. The three factors that affect the water absorption rate and powdering rate of UFHM were ranked from strongest to weakest as $\text{HPMC} > K_{12} > \text{MPS}$; the three factors that affect the compressive strength of UFHM were ranked from strongest to weakest as $K_{12} > \text{HPMC} > \text{MPS}$; finally, the optimal ratio was obtained: the blending amount of HPMC was 6.4%, the blending amount of K_{12} was 8.0%, and the blending amount of MPS was 8.0%.

The optimal stirring speed under specific conditions was determined to be 3500 r/min, and the performance of the optimal sample of UFHM was analyzed. As can be seen from Figure 2, The experimental results showed that the rates of absorbing ion water, tap water, and 0.9% NaCl solution were 15.55 g/g, 12.22 g/g, and 10.98 g/g respectively; the rate of absorbing ion water by UFHM within a short period of time could reach 10 g/g and continuously increased. This was due to the fact that the water-absorbing material had a connected porous structure and a large number of hydrophilic groups. Under the action of osmotic pressure, water could quickly enter the pores of the water-absorbing material within a short time, and as the water absorption increased, water penetrated into the structure and the interaction between the hydrophilic groups and water strengthened, thereby enhancing the water absorption capacity further. The ring-opening reaction of amino and epoxy groups in the system enabled the pores of the water-absorbing material to remain relatively intact after fully absorbing water [19], and the powdering rate decreased to 7.68%, and the compressive strength increased to 0.244 MPa.

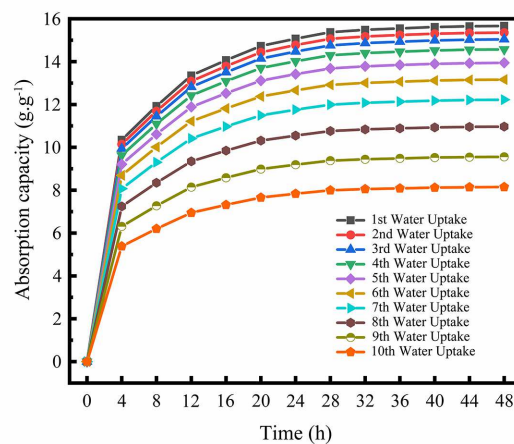


Fig. 3 For the trend chart of water absorption recovery rate

As shown in Figure 3, the liquid absorption rate of UFHM increases rapidly at first and then slowly over time. During the water absorption recovery experiment, this trend still persists. However, the more times of repeated liquid absorption,

the overall water absorption rate decreases. This is because the water-absorbing material has a three-dimensional network structure and a large number of water-absorbing groups. During each drying process, the three-dimensional network structure will be damaged, resulting in the destruction of pores and partial pore deformation and blockage. Moreover, after drying, the macromolecules of the water-absorbing material will also undergo slight degradation due to heat [20].

The addition of HPMC can alter the viscosity of the system, enhancing the strength of the absorbent material. The addition of K_{12} increases the foaming rate of the system, resulting in an increase in the number of pores in the system. The addition of MPS increases the stability of the pores, giving the bubbles good elasticity and reducing the occurrence of bubble collapse, thereby addressing the core pain points of pure UFF, such as its high brittleness and high powdering rate. This has laid a stable porous matrix foundation for subsequent modification.

4.2 HSN Modification for Enhanced Mechanical and Structural Stability

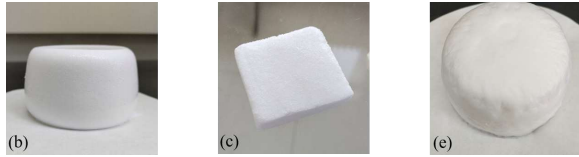


Fig. 4 The optimal sample images for UHMS

Using water absorption rate, compressive strength and powdering rate as evaluation indicators, a single-factor experiment was designed. It was found that the increase in the content of hydrophilic nano-silica would enhance the compressive strength and reduce the powdering rate. This is because the HSN surface contains a large number of unsaturated residual bonds and different bonding states of hydroxyl groups, which can form various non-covalent interactions such as hydrogen bonds and van der Waals forces with the functional groups of the UF prepolymer [21]. However, excessive HSN would lead to a decrease in the water absorption rate. This conclusion was

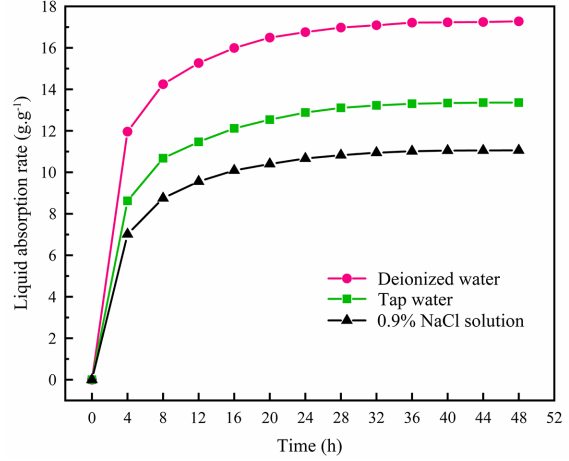


Fig. 5 Aspiration curve of UHMS

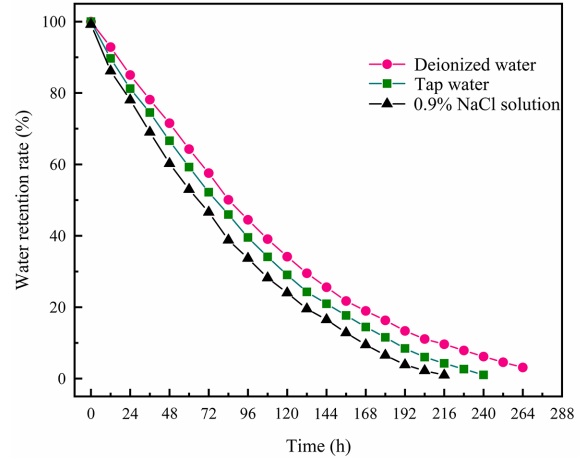


Fig. 6 Water retention curve of UHMS

also confirmed by SEM and FT-IR. Considering all these factors, the optimal content of HSN was determined to be 6%.

The optimal samples of UHMS showed no cracking, shrinking or blistering. As shown in Figure 4. Performance tests were conducted on the optimal samples of UHMS, and the results indicated that: the optimal absorption of anion water by UHMS was 17.28 g/g, the absorption of tap water was 13.36 g/g, and the absorption rate of 0.9% NaCl solution was 11.06 g/g; moreover, the water retention time could reach more than 216 hours; after repeated water absorption 10 times, the water absorption recovery rate still reached about 59%. As shown in Figure 5, 6, 7,

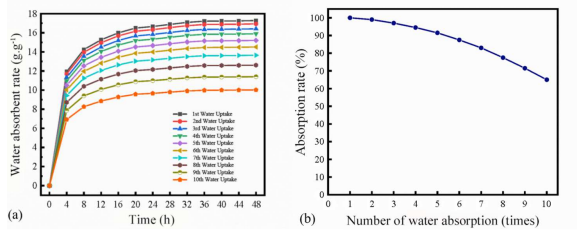


Fig. 7 (a) For the trend chart of water absorption recovery rate, (b) Graph of water absorption recovery rate

demonstrating excellent water absorption recovery performance. The powdering rate decreased to 3.62%, and the compressive strength increased to 0.336 MPa.

Figure 8 shows the SEM images of different treated HSN water-absorbing materials. Figures 8 (a), (b), and (c) are the SEM images of the HSN water-absorbing materials that were not pre-treated, while Figures 8 (d), (e), and (f) are the SEM images of the HSN water-absorbing materials that were pre-treated with KH550. From the figures, it can be clearly seen that the water-absorbing materials after pre-treatment have uniform pore wall surfaces, while the pore wall surfaces of the water-absorbing materials that were not pre-treated are uneven and there are a large number of agglomerations, some of which are agglomerations of HSN and some are agglomerations of UF glue solution. This indicates that the addition of KH550 not only reduces the agglomeration of HSN [22], but also makes the distribution of each component in the urea-formaldehyde foam system more uniform, which is conducive to improving the performance of UHMS.

Figure 9 shows the infrared spectra of the foam water-absorbing material and its components. Through comparative analysis, the characteristic peaks of each component and their interactions in the composite material can be clearly identified. Firstly, after modification with KH550, HSN exhibits a distinct Si-O-Si stretching vibration peak at 1100/cm, and the peak near 944/cm splits into two peaks, which correspond to the Si-OH stretching vibration peaks. This indicates that HSN has been successfully introduced into the composite system while retaining its intrinsic structural characteristics [23]. The broad peak in the infrared spectrum of HPMC at 3200/cm–3600/cm corresponds to the

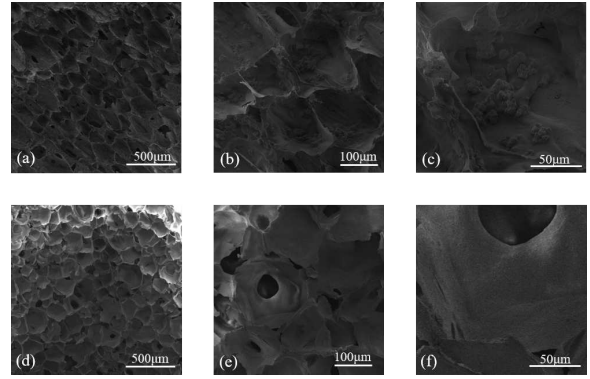


Fig. 8 SEM images of HSN water absorbing materials treated differently: (a), (b), (c) show untreated HSN, (d), (e), (f) show pretreated HSN

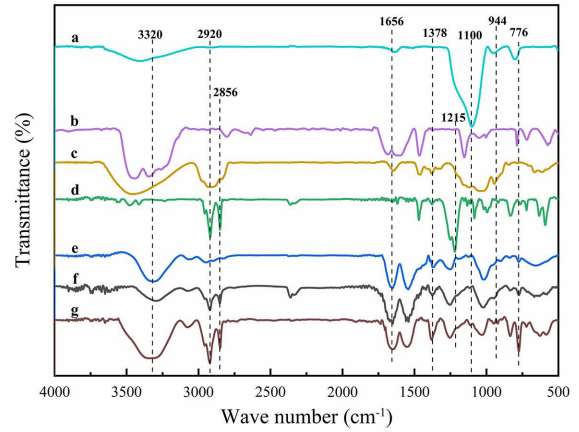


Fig. 9 FT-IR curve: a represents HSN, b represents Urea, c represents HPMC, d represents K12, e represents UF prepolymer, f represents UFHM, g represents UHMS

O–H stretching vibration, and the peaks at 1000/cm–1100/cm are attributed to the C–O–C ether bond vibration, confirming that HPMC contains hydroxyl and ether bond structures. The urea-formaldehyde resin exhibits N–H stretching vibration at 3300/cm–3500/cm and C=O stretching vibration at 1640/cm–1700/cm, indicating the presence of amide bonds and amino groups in its molecular structure. The infrared characteristic peaks of urea are similar to those of the urea-formaldehyde resin, containing N–H and C=O bonds, but the C=O peak of urea appears at 1680/cm–1750/cm with higher intensity. The characteristic peak of K₁₂ at 1200/cm–1300/cm belongs to the S=O stretching vibration of the

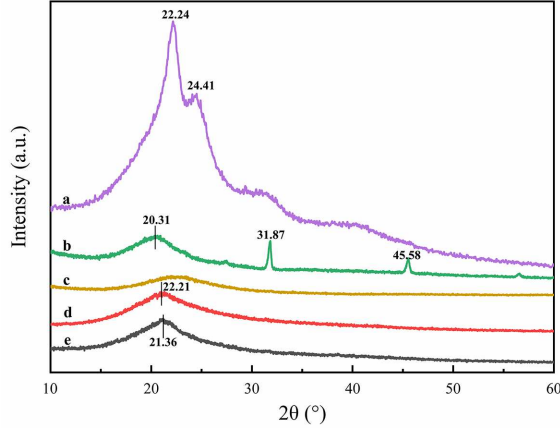


Fig. 10 XRD pattern: a represents UF, b represents HPMC, c represents HSN, d represents UFHM, e represents UHMS

sulfonic acid group, and the C–H stretching vibration at 2800/cm–3000/cm confirms the sulfonic acid-based hydrophobic chain structure of K_{12} . In the infrared spectrum of the foam water-absorbing material, the characteristic peaks of all components are present, indicating that HPMC, HSN, K_{12} , and other additives have been successfully introduced into the composite material.

It is worth noting that the N–H peak of UHMS (3300/cm–3500/cm) is broader than those of the urea-formaldehyde resin and urea, and shifts towards lower wavenumbers, which indicates that the hydroxyl groups of HPMC form hydrogen bonds with the amino groups of the urea-formaldehyde resin. Additionally, the intensity of the Si–O–Si peak (1050/cm–1100/cm) is weakened, suggesting that the hydroxyl groups on the surface of HSN can undergo condensation reactions with the hydroxymethyl ($-\text{CH}_2\text{OH}$) or amino ($-\text{NH}_2$) groups in UF, forming chemical bonds (Si–O–C or Si–O–N). Moreover, the acidic sites on the surface of HSN may play a certain catalytic role in the curing process of the urea-formaldehyde resin, accelerating the cross-linking reaction [24–26]. In conclusion, the infrared spectroscopy analysis indicates that the foam water-absorbing material is successfully composited with all components, and the introduction of HSN further optimizes the structure and comprehensive performance of the material.

As shown in Figure 10, Through the comparative analysis, the crystal structure characteristics of the foam water-absorbing material and the interactions between its components can be obtained. At 22.24° (2θ), a sharp diffraction peak appears in the urea-formaldehyde (UF) resin, indicating the presence of crystalline regions in UF. Diffuse peaks are observed at 24° , 31° , and 41° (2θ), suggesting that no ordered crystal structure is formed after the resin curing, which means UF belongs to a typical amorphous structure. The XRD curve of HPMC exhibits typical amorphous characteristics without obvious sharp diffraction peaks. A broad diffraction peak appears at 20.3° (2θ), and weak peaks are observed at 31.8° and 45.6° (2θ), which are consistent with the disordered structure of its cellulose derivative. HSN shows a broadened amorphous diffraction peak at 22° (2θ), indicating that HSN is mainly amorphous. The surface modification with KH550 did not significantly change its crystal morphology [27]. XRD analysis reveals that the crystal structures of each component in the foam water-absorbing material are retained. HPMC forms a compatible system with the amorphous UF resin through cross-linking reactions, reducing the brittleness of the composite system. This amorphous composite structure endows the material with good processability as well as excellent water absorption and retention performance. Meanwhile, HSN is uniformly dispersed in the UF resin matrix in an amorphous state, forming strong interfacial interactions with the resin. This not only optimizes the mechanical stability of the material, significantly improves its mechanical properties and toughness, but also reduces pore collapse and shrinkage phenomena, which is conducive to maintaining the integrity and damage resistance of the pores. This provides a structural basis for the material's water absorption, water retention, and water absorption recovery performance [28].

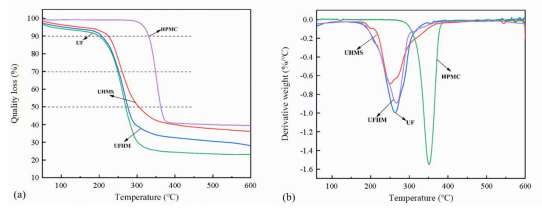
Overall, the composite material maintains amorphous characteristics as a whole, which is beneficial to the formation of a uniform porous structure and further improves the water absorption and retention performance of the material.

Figure 11 shows the thermogravimetric analysis (TGA) (Figure a) and differential thermogravimetry (DTG) (Figure b) curves of HPMC, UF, UFHM, and UHMS. Table 1 presents the

Table 1 TGA and DTG data for UHMS

Sample	T10wt%(°C)	T30wt%(°C)	T50wt%(°C)	CRC 600°C(wt%)	V _{max} (%/°C)
HPMC	330.39	349.08	363.06	39.38	1.54
UF	200.35	248.12	270.33	23.09	1.01
UFHM	207.12	252.05	277.21	28.04	0.89
UHMS	224.18	260.92	305.94	36.01	0.69

Note: T10wt%(°C), T30wt%(°C), T50wt%(°C) represent the temperatures at which a 10%, 30%, and 50% mass loss occurs, respectively; CRC, Carbon residue content; V_{max} represents the maximum decomposition rate.

**Fig. 11** TGA and DTG diagrams of UHMS; (a) TGA chart, (b) DTG chart

TGA and DTG data of HPMC, UF, UFHM, and UHMS. UF exhibits a typical three-stage decomposition behavior. The first stage (50 °C–150 °C) results in a weight loss of approximately 5%, which is attributed to the evaporation of free water and low-molecular-weight polymers. The second stage (250 °C–350 °C) leads to a sudden mass loss (weight loss of approximately 50%) due to chain scission, corresponding to the cleavage of carbamate and ether bonds. The third stage (>350 °C) involves the slow carbonization of residual substances.

Pure HPMC has relatively low thermal stability, with a weight loss of approximately 2% below 300 °C, mainly caused by the cleavage of hydroxyl and ether bonds. After 350 °C, it undergoes vigorous decomposition (weight loss of approximately 65%) with a residual carbon content of 39.38%.

Compared with pure UF (residual carbon content of 23.09%), UFHM has an increased residual carbon content of 28.04% at 600 °C, indicating that the carbonization effect of HPMC enhances the residual carbon content. The thermal stability of UHMS is further improved, with an increased initial decomposition temperature. The filling effect of HSN and the interface strengthening effect of KH550 modifier suppress heat conduction and delay decomposition. At 600 °C, the residual amount of UHMS is as high as 36.01%,

which is significantly higher than that of other samples. This is attributed to the physical barrier effect of HSN and the synergistic carbonization effect of HPMC/HSN [29].

Analysis of the DTG curves shows that the main decomposition peak of pure UF is at 260 °C, with a maximum decomposition rate of 1.01 %/°C; the peak shape is sharp, reflecting rapid segment dissociation. The main decomposition peak of UFHM shifts to 265 °C, with the decomposition rate reduced to 0.89 %/°C and the peak width increased. This indicates that HPMC delays decomposition through molecular entanglement, but uneven decomposition occurs in some regions due to phase separation.

The main decomposition peak of UHMS further shifts to 270 °C, with the lowest decomposition rate (0.69 %/°C) and a broad peak shape. This indicates that HSN is uniformly dispersed to form a thermal barrier, proving that the high-temperature stability of UHMS is enhanced. The synergistic effect of HPMC and KH550-modified HSN significantly optimizes the thermal stability and carbonization ability of the foam water-absorbing material. In the composite system, HPMC enhances the cross-linked network, while HSN provides a physical barrier and synergistic carbonization sites. These two factors jointly delay the decomposition process and increase the residual carbon content, endowing UHMS with better thermal stability.

Through the surface modification of KH550 on HSN, the "structural stability - mechanical properties - hydrophilic water absorption properties" of urea-formaldehyde foam have been synergistically improved: it not only solves the inherent defects of pure urea-formaldehyde foam and HPMC-modified foam, such as easy shrinkage and

cracking, and high powdering rate, but also simultaneously optimizes the material's water absorption and water retention as well as its cycle reuse performance, breaking the core contradiction of traditional modification "enhancing hydrophobicity but not toughness, and enhancing water absorption but not toughness enhancement".

4.3 SAPs Functionalization for Ultra-High Water Absorption and Retention

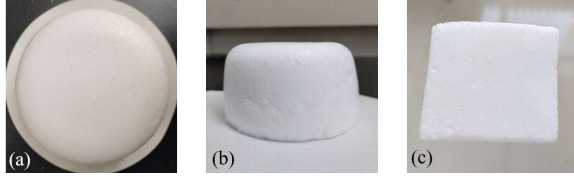


Fig. 12 Sample images of UHSA: (a) (b) Undried sample image, (c) Dried sample cross-sectional image

Using water absorption rate, compressive strength and powdering rate as evaluation indicators, a single-factor experiment was designed. It was found that the increase in the content of SAPs would initially increase the water absorption rate and compressive strength, then decrease them; the powdering rate would initially decrease and then rise. By adding an appropriate amount of water-saturated and small-sized SAPs, through the triple mechanism of "water storage micro-region construction - interface strengthening - pre-release of swelling stress" [30], the water absorption kinetics and anti-powdering performance of the foam material could be significantly optimized. Considering comprehensively, the optimal content of SAPs is 1.2%.

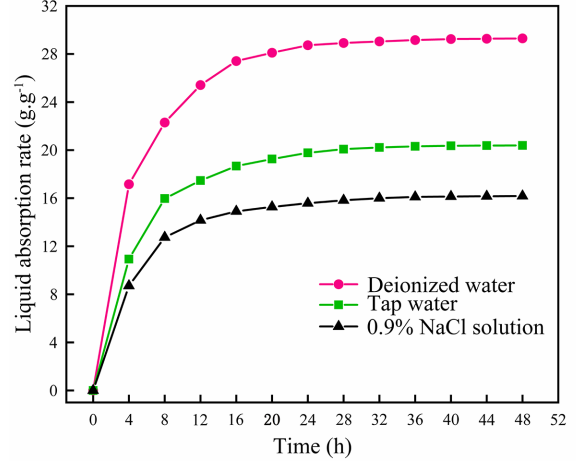


Fig. 13 Aspiration curve of UHSA

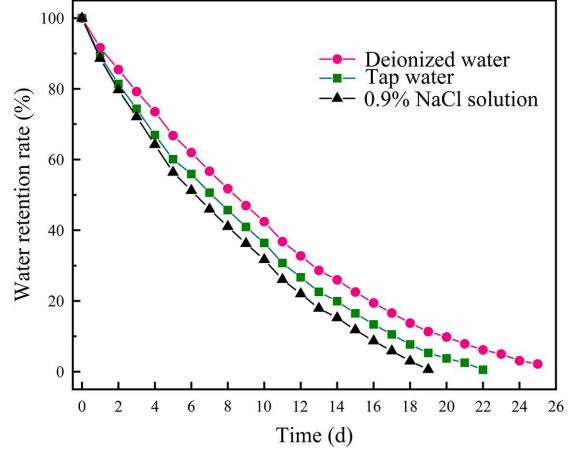


Fig. 14 Water retention curve of UHSA

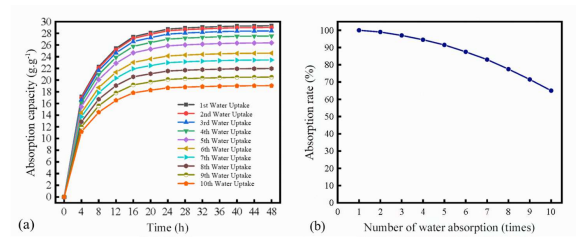


Fig. 15 (a) For the trend chart of water absorption recovery rate, (b) Graph of water absorption recovery rate

The optimal samples of UHSA showed no cracking or collapse. Performance tests were conducted on the optimal samples of UHSA, As shown in Figure 13, 14, 15 and the results indicated that the best rate of UHSA in absorbing anion water was 29.31 g/g, in absorbing tap water was 20.40 g/g, and in absorbing 0.9% NaCl solution was 16.19 g/g; the water retention time could reach more than 19 days, and the good interfacial bonding could prevent water from evaporating preferentially at the interface [31]. After repeated water absorption 10 times, the water absorption recovery rate still reached about 66%, and the appropriate SAPs, through the tripartite synergy mechanism of "water storage - expansion - mechanical adaptation", significantly improved the water absorption recovery performance of UHSA [32]. The powdering rate decreased to 3.35%; the compressive strength increased to 0.352 MPa.

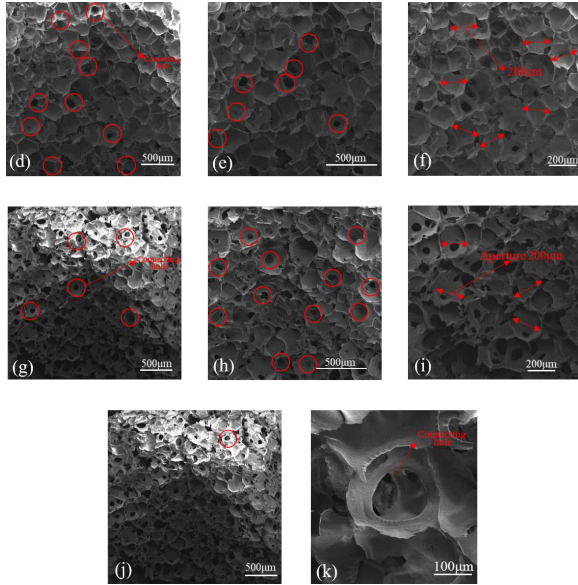


Fig. 16 SEM images of UFHM, UHNS, and UHSA: (d) (e) (f) are UHMS, (g) (h) (i) (j) and (k) are UHSA

Figure 16 shows the scanning electron microscopy (SEM) images of UFHM, UHMS, and UHSA. The differences in pore structures among the three foam water-absorbing materials can be clearly observed via SEM characterization. As presented in Figure 16, (d), (e), and (f) are the SEM images of UHMS, while (g), (h), (i),

(j), and (k) are the SEM images of UHSA. It can be seen from (d) to (i) that as the water-absorbing material changes from UHMS to UHSA, the pores become denser and more uniform, the average pore diameter decreases, and finally stabilizes at approximately 200 µm, with more interconnected pores and smoother pore walls. This indicates that after sequentially adding HPMC, HSN, and SAPs to UF, the mechanical strength of the foam water-absorbing material is gradually enhanced, along with the improvement of water absorption and water retention capacities. From (j) and (k), the internal structure of the interconnected pores can be clearly observed: the pores are intact, the pore walls are smooth, and the pores are interconnected with each other. The uniform dispersion of small-diameter SAPs finely refines the foam pores, forming a multi-level pore structure (micropores-mesopores). Therefore, compared with UHMS, UHSA has a wider pore diameter distribution range.

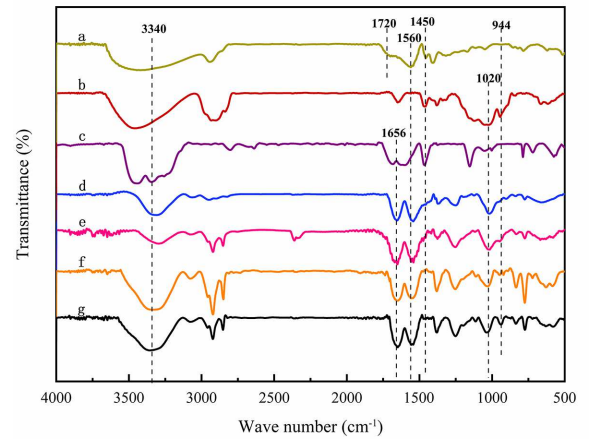


Fig. 17 FT-IR spectra: a represents SAPs, b represents HPMC, c represents urea, d represents UF, e represents UFHM, f represents UHMS, and g represents UHSA

Figure 17 shows the Fourier transform infrared (FT-IR) spectrum of the foam absorbent material. Curves a-g represent SAPs, HPMC, Urea, UF, UFHM, UHMS, and UHSA, respectively. Through comparative analysis of the FT-IR spectra, it can be clearly observed that the introduction of water-saturated small-particle SAPs exerts a significant impact on the chemical structure and interfacial interactions of the material. In curve e (UFHM),

the intensity of the O-H stretching vibration peak at 3340/cm weakens, and the C=N amide bond peak at 1656/cm shifts to a lower wavenumber (1645/cm). This indicates that HPMC reinforces the UF matrix through hydrogen bonding and physical entanglement, without the formation of new chemical bonds.

After the introduction of HSN (curve f, UHMS), the peak at 1645/cm shifts further to 1635/cm, which is attributed to the covalent combination between the amino groups of KH550 and the amide bonds of UF. The peak near 944/cm splits into two peaks, corresponding to the Si-OH stretching vibration peaks, and this is accompanied by the weakening of the peak at 1020/cm (resulting from the hydrogen bonding between Si-OH and UF/HPMC). Additionally, the width of the O-H peak broadens, indicating an increase in free hydroxyl groups. These results prove that HSN modified by KH550 synergistically enhances interfacial compatibility through the covalent connection of amino-amide bonds and hydrogen bonding. The addition of SAPs (curve g, UHSA) triggers multiple interfacial reactions, which are detailed as follows:

Firstly, the characteristic peaks of the carboxyl groups in SAPs (at 1560/cm and 1450/cm) shift to lower wavenumbers (1545/cm and 1435/cm) with an increase in peak width, indicating the formation of ion pairs ($-\text{COO}^- \cdots \text{NH}_3^+$) between these carboxyl groups and the protonated amino groups ($-\text{NH}_3^+$) in UF. Meanwhile, the peak corresponding to the residual carboxyl groups of SAPs at 1720/cm disappears, confirming that an acid-base neutralization reaction occurs between SAPs and the amino groups of UF, which further strengthens the interfacial bonding [33]. The intensity of the O-H peak at 3340/cm decreases significantly, which is attributed to the free water released by SAPs being competitively adsorbed by HPMC/UF, thereby weakening the intrinsic vibration of the cellulose hydroxyl groups. Secondly, the small-particle SAPs balance dispersion and interfacial reaction activity. With a formaldehyde/urea molar ratio of 1.4, the system retains some amino groups for ionic bonding while maintaining the stability of the cross-linked network. Thirdly, KH550-modified HSN buffers the swelling stress of SAPs through the dual effects of covalent bonds (C-NH-Si) and hydrogen bonds, inhibiting the propagation of macroscopic cracks. The chemical

bonding between amino groups and UF improves interfacial compatibility, while the hydrogen bonding between surface hydroxyl groups and SAPs enhances multiphase compatibility. Moreover, an appropriate amount of HPMC buffers the brittleness of UF through hydrogen bonding [34].

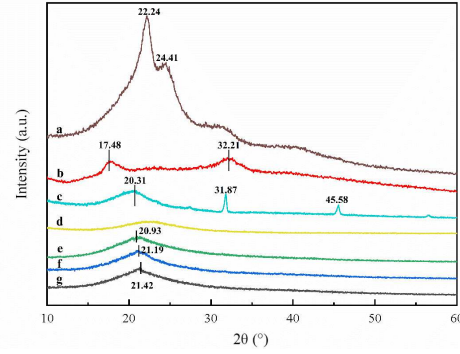


Fig. 18 XRD pattern: a represents UF, b represents SAPs, c represents HPMC, d represents HSN, e represents UFHM, f represents UHMS, g represents UHSA

Figure 18 shows the X-ray diffraction (XRD) patterns of the foam water-absorbing materials. Curves a–g represent UF, SAPs, HPMC, HSN, UFHM, UHMS, and UHSA, respectively. According to the XRD analysis, all three groups of water-absorbing materials exhibit a single broadened diffraction peak at $(2\theta) \approx 21^\circ$. This peak corresponds to the disordered amorphous structure of the cross-linked UF network, and its broadening feature indicates that the material is predominantly in an amorphous phase. With the sequential introduction of HPMC, HSN, and SAPs, the position of this peak shifts to higher angles, and the full width at half maximum increases gradually, revealing the significant regulatory effect of multicomponent composites on the microstructure of the UF matrix. In curve e (UFHM), the peak shifts to 20.93° from that of pure UF ($(2\theta) = 22.24^\circ$). This is attributed to the hydrogen bonds formed between the molecular chains of HPMC and the hydroxyl or amino groups of UF, which restrict the free stacking of UF segments and force the network to contract locally. Meanwhile, the physical entanglement between the ether bonds (C–O–C) of HPMC and the imine bonds (C=N) of UF further inhibits the mobility of the chain

segments, leading to the densification of the amorphous region [35]. After the introduction of HSN (curve f, UHMS), the peak shifts to 21.19° and the peak shape becomes slightly sharper. This indicates that HSN forms covalent grafting bonds ($C=N-Si-O-$) with the imine bonds of UF via the amino groups introduced by KH550, constructing a rigid inorganic-organic interface and partially restoring the short-range order of the UF chain segments. The addition of SAPs (curve g, UHSA) significantly induces structural reconstruction; the peak further shifts to 21.42° and becomes slightly sharper again, while maintaining a highly disordered characteristic [36]. This phenomenon can be explained as follows: Firstly, the carboxyl groups ($-COO^-$) of SAPs form an ionic cross-linked network with the protonated amino groups ($-NH_3^+$) of UF through electrostatic interactions. The strong bonding forces force the UF chain segments to twist and deform, destroying the original local order. Secondly, after being pre-absorbed to saturation, the volume expansion of SAPs exerts anisotropic tensile stress on the UF network, causing non-uniform expansion of the intersegmental distance and intensifying the broadening of the amorphous peak. Thirdly, the pretreated SAPs with high specific surface area adsorb HSN particles through hydrogen bonds ($Si-OH \cdots COO^-$), breaking the local ordered structure induced by KH550 and promoting the monodisperse embedding of HSN at the UF/SAPs interface. This forms an “inorganic-organic-ionic” multiscale interpenetrating network, which further suppresses the formation of long-range order.

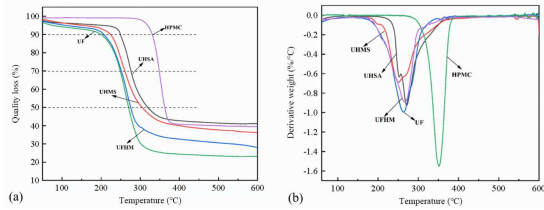


Fig. 19 TGA/DTG diagram of UHSA: (a) TGA diagram, (b) DTG diagram

Figure 19 presents the thermogravimetric analysis (TGA) (Figure a) and differential thermogravimetry (DTG) (Figure b) curves of HPMC, UF, UFHM, UHMS, and UHSA. Table 2 shows

the TGA and DTG data of HPMC, UF, UFHM, UHMS, and UHSA. By comparing the TGA/DTG curves of urea-formaldehyde foam composite materials (UFHM, UHMS, and UHSA) with those of their components (HPMC and UF), the influence of water-saturated small-particle SAPs on the thermal decomposition behavior and interfacial stability of the materials can be clearly determined.

Pure UF exhibits a typical three-stage decomposition behavior: the first stage ($50^\circ C-150^\circ C$) corresponds to the evaporation of adsorbed water; the second stage ($220^\circ C-350^\circ C$) involves the cleavage of imine bonds and decomposition of amino groups; the third stage ($400^\circ C-600^\circ C$) is the oxidation of residual carbon, with a residual carbon content of 23.09 %.

HPMC shows a two-stage decomposition process: the first stage ($50^\circ C-200^\circ C$) is the evaporation of physically adsorbed water and release of bound water; the second stage ($240^\circ C-400^\circ C$) is the decomposition of the cellulose main chain, with a final residual carbon content of 39.38 %. The thermal decomposition of UFHM is divided into three stages: in the initial weight loss stage ($50^\circ C-150^\circ C$), the hydrogen bonds formed between the hydroxyl groups of HPMC and the amino groups of UF inhibit water adsorption. In the main decomposition stage, the DTG peak shifts to a higher temperature ($265^\circ C$) with an increased peak width, indicating that HPMC delays the dissociation of the UF network through physical entanglement. The residual carbon content of UFHM increases to 28.04 %, and the maximum decomposition rate is $0.89\%/^\circ C$, which is attributed to the synergistic barrier effect of the carbonization layer of HPMC and the residual carbon of UF. The thermal stability of UHMS is further optimized: the main decomposition peak shifts to $270^\circ C$, corresponding to the decomposition of UF imine bonds and the interface of HSN grafting. The residual carbon content increases to 36.01 %, and the maximum decomposition rate decreases to $0.69\%/^\circ C$. For UHSA, the thermal decomposition process is as follows: the initial weight loss stage ($50^\circ C-200^\circ C$) is caused by the release of bound water remaining from the water-saturated state of SAPs and the exothermic dissociation of ionic bonds ($-COO^- \cdots NH_3^+$); the main decomposition stage ($240^\circ C-400^\circ C$) involves the decomposition of the UF network, with the DTG

Table 2 TGA and DTG data for UHMS

Sample	T10wt%(°C)	T30wt%(°C)	T50wt%(°C)	CRC 600°C(wt%)	V _{max} (%/°C)
HPMC	330.39	349.08	363.06	39.38	1.54
UF	200.35	248.12	270.33	23.09	1.01
UFHM	207.12	252.05	277.21	28.04	0.89
UHMS	224.18	260.92	305.94	36.01	0.69
UHSA	249.88	276.97	320.49	41.13	0.9

Note: T10wt%(°C), T30wt%(°C), T50wt%(°C) represent the temperatures at which a 10%, 30%, and 50% mass loss occurs, respectively; CRC, Carbon residue content; V_{max} represents the maximum decomposition rate.

main peak shifting from 270 °C to 275 °C and an increase in peak intensity, which corresponds to the breakage of ion pairs of carboxylic acid groups in SAPs and the cleavage of the polyacrylic acid main chain; in terms of residual carbon behavior, the residual carbon content of UHSA significantly increases to 41.13% (compared with 36.01% for UHMS), and the maximum decomposition rate recovers to 0.92 %/°C.

This phenomenon is attributed to a triple mechanism: Firstly, the carbonization of SAPs generates a porous carbon layer, which forms an interpenetrating barrier structure with the residual carbon of UF/HSN; Secondly, the hydrogen bonds (Si-OH...COO⁻) between HSN and SAPs at high temperatures are converted into Si-O-C covalent bonds, enhancing the continuity of the residual carbon network [37]; Thirdly, the high dispersion of small-diameter SAPs optimizes the pore distribution of the residual carbon and improves the antioxidant capacity. Overall, UHSA exhibits better thermal stability.

4.4 Synergistic Modification Mechanism of the Ternary System

The ternary system of "HPMC toughening - HSN pore wall reinforcement - SAP functionalization" achieves performance breakthroughs through multi-dimensional synergy: The HPMC molecular chains form hydrogen bond networks with UF, enhancing the flexibility of the matrix and reducing the powdering rate; KH550-modified HSN is uniformly dispersed on the pore walls, using silanol groups to construct a cross-linked structure, strengthening the pore wall strength and inhibiting cracking and shrinking; Pre-absorbed SAPs form local hydrogel zones,

strengthening water absorption and retention through physical adsorption and capillary action, and the hydrogen bond interaction between HSN and SAPs prevents the collapse of the pore structure. The three elements work together to solve the inherent defects of traditional urea-formaldehyde foams, such as high brittleness and insufficient water absorption. While maintaining low cost and degradability, it achieves a balance between mechanical strength and water absorption retention, and the nitrogen element released by the urea-formaldehyde in the material also gives it potential value for slow-release fertilizer, with the core adapting to the water retention needs of agricultural soil.

5 Conclusion

This study used low-cost biodegradable urea-formaldehyde resin as the matrix, and through the tri-component synergistic modification of HPMC for toughening, KH550 for modifying HSN for reinforcement, and pre-absorption SAPs for functionalization, a UF/cellulose composite foam water-absorbing material suitable for agricultural water retention applications was successfully prepared. The optimal proportion of each component was determined. Under the optimal ratio, the composite material has excellent water absorption, water retention, mechanical properties and recyclability: the water absorption rate of deionized water is 29.31 g/g, the water retention time exceeds 19 days, the water absorption recovery rate after 10 cycles is 66%, the compressive strength is 0.352 MPa, the powdering rate is 3.35%, and the performance has been comprehensively improved compared to pure urea-formaldehyde foam. The preparation process of this material is simple, the raw material cost is

low, and it is biodegradable. It has good application prospects in agricultural water-saving and water retention, and soil improvement, providing a new strategy for the development of green and sustainable polymer water-absorbing materials.

Data Availability No datasets were generated or analysed during the current study

Declarations

Competing Interests The authors declare no competing interests.

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