

Article

Oxalic Acid Pretreatment of Cotton Straw Enhances Its Salt Adsorption and Water Retention Capacity—A Soil-Amending Strategy for Saline Soil

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Abstract

Straw return is a potential practice for adsorbing salt and retaining moisture in saline–alkali soils. However, adverse climate conditions such as prolonged drought and cold winters shorten the effective structural turnover of returned straw biomass in soils. Furthermore, the rigid crystalline cell walls and recalcitrant lignin components of undecomposed plant residues lower the adsorption capacity towards salt. Here, we report the pretreatment of neutral oxalic acid to destroy the dense crystalline structure of cotton straw cellulose. Through laboratory experiments, combined with the changes in the structural and chemical properties of cotton straw, the optimal oxalic acid pretreatment (OAC) conditions were determined. Subsequently, the application effectiveness of OAC was evaluated via pot experiments and field trials. The optimal conditions of OAC were 0.2% dosage, 60 °C, and 24 h, displaying a maximum increase in salt absorption and water retention capacities of cotton straw materials, through exposing the hydroxyl network of cellulose and chemically hydrolyzing recalcitrant lignin. In the indoor potted plant experiments, the feasible application of oxalic acid pretreatment can be regarded as an active barrier, increasing soil moisture by 16–43% and reducing total salts by 23–26% in the topsoil (0–20 cm) within a 45-day laboratory incubation. Additionally, the OAC pretreatment had negligible adverse impacts on soil microbial communities. Moreover, some plant-beneficial microbes (e.g., *Sphingomonadaceae* and *Gemmatimonadaceae*) were stimulated, with their relative abundance increasing by 26–40% and 27–63%, respectively. Ultimately, under the pretreatment of oxalic acid-modified cotton straw salt-absorbing water-retention agent (OAC-SR), cotton seedling emergence rates, plant height, and biomass all increased to varying degrees across different concentrations of saline–alkali soil (0.05–1.0%) in the field. Then OAC-SR can be potentially applied to the process of cotton straw return to facilitate the turnover of straw structure in soil, enhance the salt-adsorption and water-retention capacities of returned straw, and provide a low-salt microenvironment for crop growth. This study demonstrates a further low-carbon and in situ applicable route to accelerate the destruction of cotton straw structure, thereby alleviating crop salt damage and promoting the green circular development of saline–alkali soil remediation.

Keywords: oxalic acid pretreatment; cotton straw; saline soil; salt absorption



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1. Introduction

Global land systems include over 950 million hectares of saline–alkali soil [1]. The comprehensive utilization of saline–alkali land can effectively relieve world hunger and maintain food security [2,3]. In the northwest of China, Xinjiang, 20 million hectares of saline–alkali land are gradually amended to increase the production [4]. How to utilize these saline–alkali soil resources is crucial for ensuring sustained increases in grain production [5]. Although some methods of saline–alkali soil remediation (e.g., gypsum, desulfurization steel slag, biochar) have been applied to improve soil fertility [6–8], the relatively high cost and unknown secondary pollution risk limit their sustainable application [9]. The current methods for green improvement of saline–alkali land still mainly focus on conventional physical techniques such as underground pipe drainage and flooding irrigation for salt leaching, as well as organic or inorganic soil amendments [10,11]. Crops with a certain salt tolerance, such as cotton, are widely cultivated annually in Xinjiang, China. And the area of cotton cultivation has exceeded 2.5 million hectares, with an annual 17 million tons of cotton straw [12]. Cotton straw returned to soils is an important utilization method to provide favorable organic nutrition for crop growth [13]. Meanwhile, the decomposed straw residues can increase the salt adsorption and water retention capacities of soils to alleviate crop salt damage [14]. However, the adverse climate conditions of Xinjiang (i.e., drought, scarce rainfall, and long, cold winters) extend the effective turnover time of returned cotton straw. This leads to the accumulation of cotton straw residues with low decomposition degree in soils, increasing the risks of pests and diseases and lowering the adsorption capacities of residues [15,16]. Meanwhile, during the decomposition of cotton straw, the structurally diverse plant residues accumulate in the soil to adsorb and immobilize salt ions [17]. Additionally, the released organic micromolecules are incorporated into soil graded aggregates and further improve soil water retention [17,18]. Finding simple and effective pretreatment of straw plays a crucial role in facilitating the turnover of straw residues and enhancing its salt adsorption capacity in soil.

Bioaugmentation and biostimulation methods have been applied to enhance the structural conversion of straw returned to soils [19,20]. The cellulase applications at different doses can also demonstrate the promoting effect of straw decomposition by 6–28% within 45 d constant-temperature cultivation [19]. The combined application of nitrogen fertilizer and straw-decomposing inoculants displayed significant acceleration (by 7–23%) of straw decomposition in the winter wheat–summer maize continuous cropping system [21]. It's worth noting that the biological reactions in the soils are dependent on optimal temperature and moisture conditions [22,23]. Duan et al. found that preparing straw-based biochar under anaerobic conditions can increase soil moisture and reduce the salt content of the soil profile by salt adsorption of the biochar [24].

Moreover, chemical treatments (e.g., diverse acids, alkali, and persulfate) have been gradually developed to destroy the recalcitrant lignocellulosic cross-structure in the fields of bioenergy conversion or environmental remediation [25–28]. The pretreatment of urea and NaOH (USH) achieved a 37% enhancement in the decomposition of maize straw in the field within six months [29]. The USH (7%/0.1%) can interact synergistically to cleave rigid hydrogen bond linkages of recalcitrant lignin and hemicelluloses, and break the crystalline structure of cellulose [29,30]. It was reported that the pretreatment with 0.2–1.0% HNO₃ for 1–20 min at 120–160 °C could increase the enzymatic hydrolysis digestibility of straw [31]. Cai et al. developed a 0.1% persulfate (PS) pretreatment for maize straw to obtain a 29% increment in the decomposition of straw incorporated into soils within 181 days [32]. The mechanism of PS pretreatment is believed to destroy the dense crystalline structure of cellulose and break the aryl C–O bonds of lignin [32]. Furthermore, these pretreated straw residues exhibited high adsorption capacity for maintaining soil

moisture and organic nutrient content. The above advantages contribute to plant growth and production increase [32]. Previous research found that oxalic acid, as an organic acid and a plant root exudate, can activate and release salt ions in soil [33,34]. Meanwhile, oxalic acid can dissociate the cross-linked hydrogen bonds of cellulose and lignin. The oxalic acid was also detected during the decomposition of rice straw and was responsible for P solubilization from phosphate and udaipur rock phosphate [35]. It was reported that oxalic acid pretreatment of straw under ball milling obtained a sufficient amount of total reducing sugar by increasing the depolymerization of straw [36]. Under the optimal oxalic acid pretreatment conditions (210 °C, 3.6% acid concentration, and 16.3 min) for wheat straw, xylose was solubilized in the liquid phase, causing an increase in the cellulose and lignin content of the biomass, and the ethanol yield obtained was 59% [37]. Compared to inorganic acids (0.25% *v/v* H₂SO₄, HCl, H₃PO₄) and alkali (0.25% *w/v* NaOH), oxalic acid leads to the highest glucose yield from enzymatic hydrolysis (84.2%) and the lowest formation of furans under the optimal liquid hot water conditions at 160 °C. It can be used as a superior promoter for increasing sugar recovery and saving energy in liquid hot water pretreatment [38].

Preliminary experimental results indicated that the adsorption capacity of straw residues may be increased with straw decomposition. Despite the expected promotion of straw decomposition, the naturally formed saline–alkali land requires the adoption of suitable green methods to consolidate the benefits of cotton straw return under the climate conditions of a harsh drought and long cold winters in Xinjiang, China. Although oxalic acid pretreatment has a facilitating effect on the bioconversion process of straw applied in the biomass refining manufacture, the adopted practices under high temperature (>100 °C) and high concentration (>1%) conditions may be unsuitable for the farmland. Then, in this study, we aimed to develop the environmental impact of safe reagents. Based on the actual temperature and dosage ranges applicable to farmland in Xinjiang, China, screening tests were conducted to determine the optimal conditions for oxalic acid pretreatment of straw under controlled indoor conditions (Figure 1). Considering that the reaction of pretreatment proceeds relatively slowly under indoor temperatures (20–35 °C), we appropriately extended the reaction pretreatment time to several days. The effects of oxalic acid pretreatment on the chemical structure and salt adsorption of cotton straw were analyzed to screen applicable dosages for cotton straw returned to soils. Field experiments of the selected oxalic acid pretreatment were conducted to evaluate soil salt ion distribution and water retention in soils. Meanwhile, the regulatory effects of oxalic acid pretreatment on the soil microbial communities were also investigated to identify the ecological safety. This study aims to provide an efficient method to alleviate crop salt damage through in situ oxalic acid pretreatment of cotton straw incorporated into soil.



Figure 1. The roadmap of the experimental design (the numbers from 1 to 6 in the figure represent the experimental design steps).

2. Materials and Methods

2.1. Reagents and Materials

Oxalic acid was purchased from Tianjin Fuchen Chemical Reagent Co., Ltd., Tianjin, China. Sodium chloride, potassium chloride, magnesium chloride, and calcium chloride were purchased from Chemical Reagent Co., Ltd., Tianjin, China. Bromide potassium was purchased from Tianjin Hengchuang Zhongda Technology Development Co., Ltd., Tianjin, China. All the chemical reagents listed above are of analytic grade (AR). Deionized water was produced by a laboratory water purifier. The cotton seeds selected for this experiment were Xinluzao 68, which tolerates soil salt concentrations up to 0.6%, supplied by Xinjiang Jinfengyuan Seed Industry Co., Ltd. (Shihezi city, China).

Fresh cotton straw was collected from the farmland in Shihezi city, China (85.7093 E, 44.4330 N), naturally air-dried, and cut into segments about 1.0 cm in length. Then the dried cotton straw was rinsed with deionized water and further dried in a forced-air drying oven at 50 °C for 14 h. The dried cotton straw was crushed with a high-speed grinder and sieved through a 10-mesh screen. The obtained cotton straw powders were stored in the resealable plastic bags for future experiments.

The saline–alkali soil was collected from farmland in Shihezi city, China (86.0144 E, 44.4813 N), naturally air-dried in the shade, then sieved through a 20 mm mesh for subsequent use. The soil exhibited a pH of 8.05, an electrical conductivity of 2.66 mS/cm, and a total salt content of 17.17 g/kg.

2.2. Optimization of the Preparation Conditions for Oxalic Acid-Modified Cotton Straw Salt-Absorbing Water-Retention Agent (OAC-SR)

A total of 20.0 g of cotton straw powder was placed in a 250 mL conical flask with 200 mL of deionized water. A total of 0.2 g oxalic acid (1% of the cotton straw mass) was added to the reaction flask. The reactions were conducted at 60 °C with different reaction durations (0.5, 1.0, 1.5, 2, 3, 4, 5 d). We extend the pretreatment time to 5 d, aiming to compensate for the decrease in reaction rates caused by the insufficient temperature by keeping it as close as possible to room temperature. After reaction, the pretreated cotton straw was filtered and rinsed with deionized water until neutral. Then the solid materials were dried at 50 °C for 36 h and stored in ziplock plastic bags for subsequent determination of straw adsorption capacity and physicochemical structures.

Based on the determined processing time (Figure 1), 20 g of cotton straw powder and 200 mL of deionized water were placed in a 250 mL conical flask in sequence. Oxalic acid at different concentrations (0.1%, 0.2%, 0.5%, 1%, 2%, and 5% of the cotton straw mass) was added to the reaction flasks, respectively [36–38]. Considering the negative impact of high-concentration oxalic acid on soil physical and chemical properties, the application of oxalic acid should be controlled within a moderate and appropriate range. The flasks were processed at a constant temperature of 60 °C for the determined duration. The following operation was the same as described above paragraph.

Based on the determination of the processing time and the amount of oxalic acid, 20 g cotton straw powder and 200 mL deionized water were placed in a 250 mL conical flask in sequence, with a fixed amount of oxalic acid and the same processing time under different temperature conditions (i.e., 20 °C, 30 °C, 40 °C, 50 °C, 60 °C, 70 °C). The pretreatment temperature design was based on the daily temperature range in Xinjiang, China, and temperature requirements for material chemical reactions (Figure S1). Although the chemical reaction effect can be enhanced with rising temperature, we design slightly above the normal temperature range (50 °C, 60 °C, and 70 °C) to select the appropriate temperature range to minimize energy consumption and application costs while ensuring effectiveness. The following operation was the same as described above paragraph.

The salt absorption performance of the cotton straw materials prepared under different conditions was tested. The pretreatment effect of oxalic acid under these conditions was evaluated by the Na^+ adsorption amount. The specific experimental steps were as follows: 0.6 g of cotton straw material was added to a 50 mL conical flask with 30 mL of 4 g/L NaCl solution. Each treatment had three replicates. The mouths of the conical flasks were sealed with a sealing film. The reaction flasks were shaken at 180 r/min at 25 °C for 48 h and stood for 12 h. The 48 h reaction time has been identified as the time within which the straw materials can reach a state of salt adsorption saturation in the preliminary experiments. A total of 5 mL of supernatant was collected in a volumetric flask and diluted to 50 mL with deionized water. The diluted supernatant was passed through a 0.22 μm needle-type water filter membrane. The Na^+ content of the filtered supernatant before and after the adsorption experiment was measured by an ion chromatograph (Thermo Fisher Scientific, Co., Ltd., Shanghai city, China). The Na^+ adsorption amount of straw material was calculated according to Formula (1).

$$A = \frac{(C_1 - C_2) \times V \times k \times 10^{-3}}{m} \quad (1)$$

In the formula, A (mg/g) represents the adsorption amount (mg/g); C_1 ($\mu\text{g/mL}$) is the initial concentration of Na^+ in the reaction solution before adsorption; C_2 ($\mu\text{g/mL}$) is the concentration of Na^+ in the reaction solution after adsorption; V (mL) is the volume of the NaCl solution in the adsorption experiment; k is the dilution factor, and 10^{-3} is used to convert μg to mg ; m (g) is the mass of the cotton straw added to the reaction solution.

Based on the salt absorption performance of straw materials, the optimal conditions for OAC-SR preparation were determined. Additionally, the physical structures and chemical compositions of the cotton straw materials were characterized.

2.3. Verification of the Salt Absorption and Water Retention Capacity of OAC-SR

2.3.1. Na^+ Adsorption Capacity of Materials

To verify the effect of organic acid modification on cotton straw, the absorption salt comparison test was conducted. Four groups were set up: 1 cm uncrushed cotton straw (A), crushed original cotton straw (B), cotton straw treated with deionized water (C), and OAC-SR (D) as the adsorbent. The salt absorption performance test was carried out as per Section 2.2. The method for testing Na^+ content was the same as that for calculating the adsorption amount, following Formula (1).

2.3.2. Water Adsorption Capacity of Materials

A total of 1.00 g of cotton straw material was accurately weighed and placed in a 100-mesh nylon mesh bag. It was immersed in a beaker containing 200 mL of deionized water. The sample was allowed to stand to fully absorb water and reach adsorption equilibrium. Then, it was drained until no water droplets fell, and weighed. The water absorption capacity of the straw material is calculated according to Formula (2).

$$Q = \frac{m_2 - m_1}{m_0} \quad (2)$$

In the formula, Q (g/g) represents the water absorption capacity of cotton straw materials, m_0 (g) is the mass of the cotton straw, m_1 (g) is the total mass of the cotton straw material and nylon net before submerged in the liquid, and m_2 (g) is the total mass of the cotton straw material and nylon net after saturated with the liquid.

2.3.3. Water Retention Comparison Experiment

A 1.0 g sample of dry cotton straw material with six replicates was accurately weighed, placed into a 100-mesh nylon mesh bag, and immersed in a beaker filled with 25 mL of deionized water to shake at 25 °C for 24 h [13,29]. After reaching equilibrium, withdrawn materials were allowed to stay until no droplet was formed, and then they were weighed in order to calculate the relative weight gain. Then the total mass of the sample was dried at 25 °C and measured at 1, 2, 3, 4, 5, and 6 h, respectively. The water retention rate was calculated using Formula (3).

$$R_W = \frac{m_2 - m_3}{m_2 - m_1} \times 100\% \quad (3)$$

In the formula, R_W represents the water retention rate of cotton straw. The m_1 (g) and m_2 (g) are the same as those in Formulas (1) and (2), and m_3 (g) is the sample mass at the specific sampling time mentioned above.

2.4. Adsorption Experiments

2.4.1. Adsorption Condition Test

The effects of the initial NaCl concentration, dosage of cotton straw, adsorption time, and pre-treatment temperature on the salt absorption performance of cotton straw were investigated. The untreated raw cotton straws after crushing (CK) were used as the control, and OAC-SR was used as the test sample. Firstly, the initial NaCl concentrations were set at 1 g/L, 2 g/L, 4 g/L, 8 g/L, 10 g/L, and 20 g/L to compare the Na⁺ adsorption capacity of cotton straw materials. The setting of salt concentration is based on the range of salt content in saline–alkali soil of Xinjiang, China (Table S1). Secondly, the dosage of cotton straw was set at 0.03 g, 0.15 g, 0.3 g, 0.6 g, 1.2 g, and 1.8 g to test the effect of material dosage. Thirdly, the adsorption time was set at 1 h, 2 h, 5 h, 10 h, 15 h, 24 h, 48 h, and 72 h, with samples taken at regular intervals to determine the adsorption amount at different times. Finally, the pre-treatment temperatures were set at 20 °C, 30 °C, 40 °C, 50 °C, and 60 °C to investigate the effects of temperature. The specific test steps and calculation formulas used were the same as those in Section 2.2.

2.4.2. Base Cation Adsorption Experiment

The cations in saline–alkali soils are mainly Na⁺, K⁺, Mg²⁺, and Ca²⁺. To clarify the adsorption effects of OAC-SR on cations, single solutions of 1.0 g/L NaCl, KCl, MgCl₂, and CaCl₂, as well as mixed solutions, were prepared for the adsorption experiments. The following processes regarding cation adsorption were the same as those in Section 2.2.

2.4.3. Soil Extraction Solution Adsorption Experiment

The pH, conductivity, and total salt content of the soil were 9.27, 5.92 mS/cm, and 28.3 g/kg, respectively. 40.0 g of saline–alkali soil was weighed into a 250 mL volumetric flask, and 200 mL of deionized water was added at a water-to-soil ratio of 5:1. The volumetric flask was shaken in an oscillator at 180 r/min for 30 min, and the mixture was allowed to stand for 2 h. The supernatant in the flask was collected and filtered to remove floating residues in the extract. Following the procedure described in Section 2.3, the obtained soil extract was used as the initial mixed ion solution and diluted 2, 4, 6, 8, and 10 times to investigate the effects of different ion concentrations in the adsorption experiments.

2.4.4. Soil Phase Adsorption Experiment

Six types of soils were tested to verify the application scope of OAC-SR materials (Table S2). The basic properties of the soils are shown in Table S1. OAC-SR and raw cotton

straw (CK) were used as test materials. The salt (NaCl) concentration was set at 0.5%, 1%, and 2% of the soil mass. The cotton straw content was 4% of the soil mass. 5.0 g of soil was mixed with the cotton straw and transferred to a 50 mL beaker, followed by the addition of 12.5 mL of deionized water. Each treatment had three replicates, resulting in a total of 108 samples. After a 48 h adsorption process, the mixtures were left to stand for 12 h. 1 mL of supernatant was removed and added to a volumetric flask, followed by the addition of deionized water to make up to 10 mL. The supernatant sample was filtered through a 0.22 μm syringe filter. The Na^+ content was determined by an ion chromatograph instrument (Thermo Fisher Scientific, Co., Ltd., USA).

2.5. Indoor Potted Plant Experiment

Soil with low salt content (0.04%) was collected from the farmland in Shihezi city, Xinjiang, China (85.7093 E, 44.4330 N). Soils with salt contents of 0.05%, 0.1%, 0.5%, and 1% were obtained through adding salt quantitatively in the laboratory. Soil salt content levels were based on the range of salt content in the farmland of Xinjiang, China (Table S1). Meanwhile, preliminary indoor experiments indicated that cotton seeds exhibited minimal germination when soil salinity exceeded 1.0%. Therefore, the soil salinity range for this potted plant experiment is set between 0.05% and 1%. Salt-baked soil from the farmland was used as a control. In the above different salt content soils, three treatment groups were set up: no landfill of cotton straws (CK), landfill of raw cotton straws (CU), and landfill of cotton straws treated by oxalic acid (CT). Each treatment group was repeated three times. According to the amount and pattern of cotton straw returned to the field, a series of pot experiments were set up [39,40]. In Xinjiang, China, the amount of cotton straw was from about 6 tons to 12 tons per hectare. Additionally, the depth of the straw returned to the field was generally about 10 cm in the tillage layer. Then, the 1 cm thick (approximately 8 g in weight) cotton straws were filled at the 9 cm position of the long tube cup. The 10 cotton seeds were planted in each pot, according to providing regular and quantitative water. The pot experiment was conducted in an incubator maintained at a constant temperature of 28 °C and humidity of 67%. After 7 days, the germination of cotton was counted. After the basic situation of cotton germination was statistically analyzed, 3 cotton seedlings were planted in each pot. Soil from the upper and lower layers of the cotton straw layer in each treatment group was collected to measure soil moisture and Na^+ contents after 50 days of incubation. Two cotton plants were randomly selected from each pot to investigate plant height and dry weight.

Soil samples from the upper layer above the cotton straw layer, with initial salt contents of 0.1%, 0.5%, and 1.0%, were collected and placed in 10 mL plastic centrifuge tubes, respectively. The upper soil refers to the soil layer above the straw layer, ranging from 1 to 10 cm below the surface. The lower soil layer refers to the soil below the straw layer. The samples were then immediately placed in a -80 °C ultra-low temperature refrigerator for preservation prior to testing. The preserved soil samples were shipped on dry ice to Beijing Novogene Co., Ltd. (Beijing, China) for soil microbial analysis.

The kit machine (MP Biomedical, Santa Ana, CA, USA) was used to extract DNA from the 0.5 g sample. After obtaining the PCR products, the electrophoresis was performed using a 2% concentration of agarose gel for detection. The PCR products that passed the detection were purified using magnetic beads. The constructed libraries were quantified using Qubit and Q-PCR. After the libraries were qualified, PE250 sequencing was performed on the NovaSeq6000 (S4 Novogene, Beijing, China) with the Silva database and the Unite database. The quantified raw data were analyzed using QIIME2 software (Version 2023.5, China). Diversity index analysis was further performed based on the OTUs results of the sample groups.

2.6. Field Experiment

The field micro-area verification experiment was conducted at the Teaching Experimental Field of Shihezi University (86.0024 E, 44.3120 N) from April to November 2024. The annual precipitation and evaporation in this area are 175.5 mm and 1514.9 mm, respectively. The soil pH, electrical conductivity, and total soil salt content were 8.04, 1.68 mS/cm, and 10.4 g/kg, respectively.

Each experimental plot had an area of 6.0 m² (3.0 m in length and 2.0 m in width), with three treatments: no addition of cotton straws (CK), addition of raw cotton straws (CU), and addition of oxalic acid-prepared cotton straw (CT). Each treatment had three replicates through a randomized block design. The cotton straws were crushed into 2 cm-long segments. 12.6 kg of cotton straw was spread in each treatment plot at a soil depth of approximately 20 cm. In the CT plots, an oxalic acid solution was prepared at a concentration of 0.2% relative to the straw mass (with a water-to-oxalic acid mass ratio of 1:100), and was evenly sprayed onto the surface of the cotton straws. The cotton straw was then covered with soil for burial. The irrigation and fertilization in the field follow local routine field management practices. After a 45-day field experiment, soil samples were collected from the 0–20 cm and 20–40 cm layers using the five-point sampling method. The soil pH, electrical conductivity, soil moisture content, and total soil salt content were measured after mixing and homogenizing the samples.

2.7. Data Analysis

The initial data obtained from the experiment were organized by Excel 2021 (Microsoft, Redmond, WA, USA). A one-way ANOVA analysis of variance was conducted for the results of the cotton straw adsorption experiment for Na⁺ and water absorption (including the pH, electrical conductivity, and soil Na⁺ content of soils under different landfill treatments) by SPSS 24.0 statistical software (IBM Corporation, Armonk, NY, USA). The confidence interval is determined by the confidence level and standard error according to the *p*-values (*p* < 0.05). In the field micro-area experiment, a one-way analysis of variance was performed among different landfill treatments, and independent-samples *t*-tests were conducted on the soil indicators of surface soil samples (0–20 cm) and subsurface soil samples (20–40 cm). The obtained data were analyzed, and visualization plots were generated by Origin 2021 (OriginLab Corporation, Northampton, MA, USA).

The crystallinity index of cotton straw was calculated using Equation (4).

$$CrI = [(I_{002} - I_{am})/I_{002}] \times 100\% \quad (4)$$

The I_{002} is the maximum intensity of the 002 peak at about $2\theta = 22.5^\circ$, and the I_{am} is the lowest intensity of the peak at about $2\theta = 18.5^\circ$.

The analysis of X-ray photoelectron spectroscopy (XPS) was based on the XPSPEAK41 software (Raymund Kwok, Hongkong, China) and Gauss model (Carl Friedrich Gauss, German), and the binding energy values of chemical functional groups were matched from the NIST X-ray photoelectron spectroscopy database (SRD 20), Version 5.0, U.S. Department of Commerce (<https://srdata.nist.gov/xps/EnergyTypeElement>). This website was accessed on 24 October 2025.

3. Results

3.1. Optimization of OAC-SR Preparation Conditions

A controlled-variable experiment was designed to systematically investigate the effects of oxalic acid dosage, pretreatment time, and temperature on the Na⁺ adsorption capacity of cotton straw materials (Figure 2). The Na⁺ adsorption capacity of oxalic acid-pretreated

cotton straw displayed a trend of initial increase followed by a decrease, and eventually stabilized to 3.45 mg/g (Figure 2a). With the increase in pretreatment time, the Na⁺ adsorption capacity of oxalic acid-pretreated cotton straw was increased from 4.07 mg/g to 5.39 mg/g ($p < 0.01$). For the 1.0 d pretreatment cotton straw, the adsorption capacity reached a maximum value of 5.39 mg/g (Figure 2a). With the increase in pretreatment time, the Na⁺ adsorption capacity of oxalic acid-pretreated cotton straw gradually maintained a range of 3.09–3.68 mg/g.

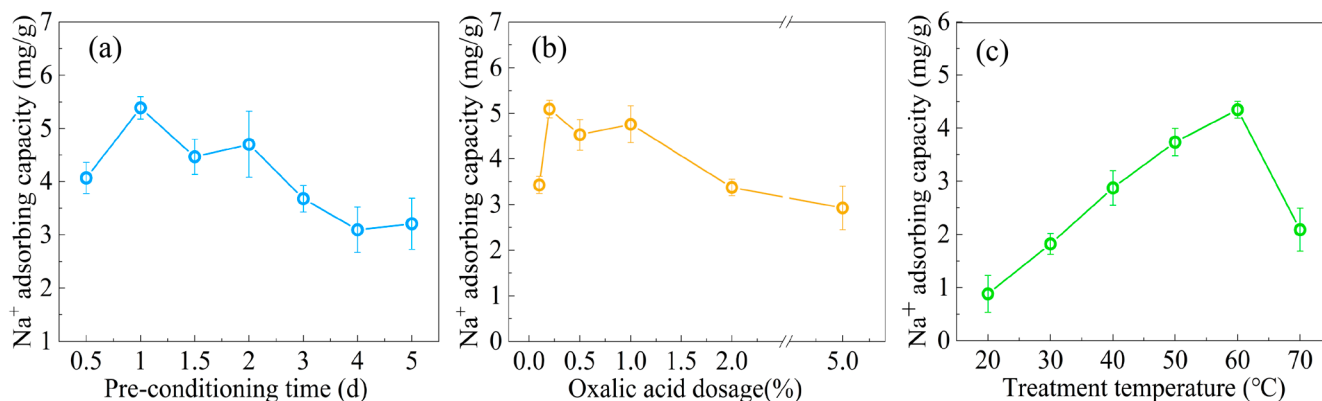


Figure 2. The effects of different pretreatments on Na⁺ adsorption of cotton straw. (a) Pre-conditioning time; (b) oxalic acid dosage; (c) pretreatment temperature.

With the enhancement of the oxalic acid dosage, the Na⁺ adsorption capacity of pretreated cotton straw materials was increased from 3.53 mg/g to 5.10 mg/g ($p < 0.05$), then decreased to 2.93 mg/g with further increases in dosage (Figure 2b). And the maximum adsorption capacity was 5.10 mg/g under the 0.2% oxalic acid pretreatment (based on cotton straw mass).

As the pretreatment temperature rose, the Na⁺ adsorption capacity of oxalic acid-pretreated cotton straw displayed a trend of initially increasing from 0.88 mg/g to 4.34 mg/g, followed by a decrease to 2.08 mg/g (Figure 2c). The oxalic acid-pretreated cotton straw obtained the maximum Na⁺ adsorption capacity of 4.34 mg/g at a pretreatment temperature of 60 °C. Overall, the optimal pretreatment conditions were a pretreatment duration of 1.0 d, an organic acid dosage of 0.2% (relative to cotton straw mass), and a temperature of 60 °C (Figure 2).

3.2. The Capacity of Salt Absorption and Water Retention of OAC-SR

The Na⁺ adsorption capacity of OAC-SR was 4.08 mg/g. Compared with crushed raw cotton straw and uncrushed raw cotton straw, the Na⁺ adsorption capacity of OAC-SR was increased by 2.9 and 6.2 times, respectively (Figure 3). Under identical adsorption conditions, there was no significant difference in salt adsorption between cotton straw pretreated with deionised water and raw cotton straw (Figure 3a). And the OAC-SR displayed a water absorption capacity of 6.98 g/g for the peak of absorption equilibrium, representing a 1.5-fold increase in moisture adsorption compared to that of raw cotton straw (Figure 3b). The surface of the pretreated cotton straw displayed some uniformly distributed salt particles by magnified 10,000 times (Figure 3a).

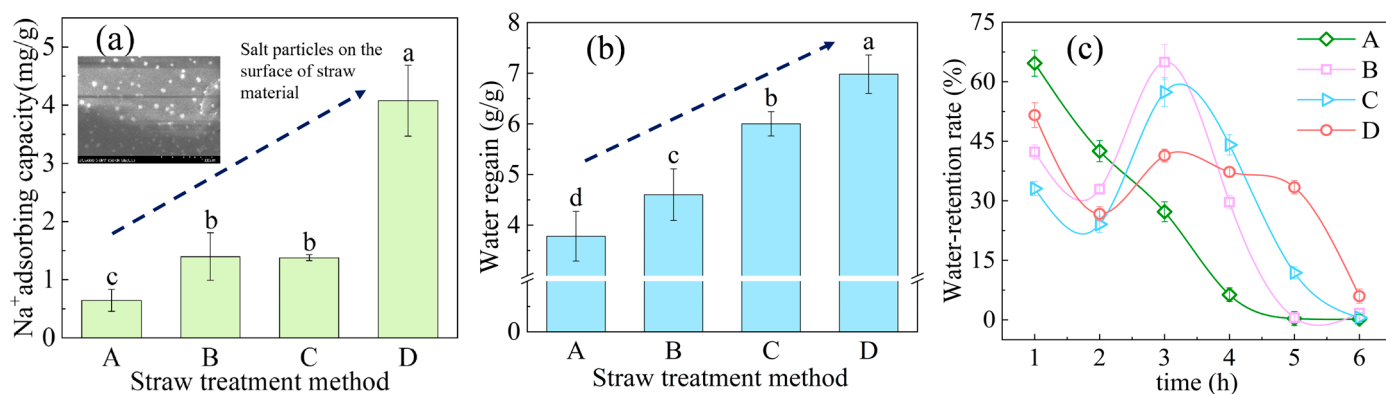


Figure 3. Effects of different pretreatments on the salt adsorption and water retention capacities of cotton straw. (a): Na⁺ adsorbing capacity; (b): water regain; (c): water-retention rate. In the figures, A represents the uncrushed cotton straw (1 cm length) treatment; B represents crushed cotton straw treatment; C represents deionized water pretreatment; D represents oxalic acid pretreatment. Different lowercase letters indicate significant differences between different treatments ($p < 0.05$).

Compared to that of uncrushed cotton straw, the water absorption capacity of OAC-SR was increased by 1.8-fold (Figure 3). Significant differences were observed between uncrushed cotton straws and crushed raw cotton straws ($p < 0.05$). It indicated that both physical and chemical structure changes significantly facilitated the water absorption capacity of cotton straw. Compared to that of the other pretreated straw materials, OAC-SR exhibited more stable water retention performance (Figure 3b). Within 2 h, the moisture contents of all pretreated cotton straw were decreased substantially, implying that water molecules adhering to the surface evaporated extensively, causing a sharp drop in material moisture content (Figure 3c). After 3 h, the water retention rates of both crushed and deionized water-pretreated cotton straw were increased compared to those of the other groups. Additionally, the water retention capacity of all pretreated cotton straw showed significant differences: OAC-SR exhibited a water retention rate of 33%, representing a 60 to 98-fold increase compared to other groups (Figure 3c). In summary, OAC-SR demonstrated markedly superior salt absorption, water uptake, and water retention capabilities.

3.3. Effects of Oxalic Acid Treatments on the Structure and Properties of Cotton Straw

The marked roughness and diverse small pores were displayed on the surface of OAC-SR (Figure 4). The unchanged microstructure of pretreated cotton straw after adsorption of salt implied the stability of materials and potential adherence to the adsorption mechanism (Figure 4) [13,29]. The Na element on the surface of OAC-SR materials after adsorption showed visible increments, shielding the highlights of the carbon element (Figure 4e,f). Meanwhile, the structural destruction of oxalic acid pretreatment on cotton straw contributed to increases in the specific surface area, pore volume, and pore diameter of OAC-SR materials by 24.3%, 60.1%, and 294% ($p < 0.05$), respectively (Figure 5a,b). This demonstrated the advantages of adsorption with changes in the OAC-SR materials' microstructures.

The FTIR spectrum of cotton straw exhibited a broad band at 3345 cm^{-1} , where the stretching vibration peak originates from O-H groups in alcohol, phenol, and carboxylic acid, primarily presented in cellulose and lignin components (Figure 5c). The band at 3345 cm^{-1} of the OAC-SR group displayed a reduced trend, indicating partial degradation of cellulose and lignin of pretreated cotton straw (Figure 5c), corresponding to the disruption of intermolecular hydrogen bonds within the cellulose molecules (Figure 5c) [29]. The band at 1740 cm^{-1} (C=O) in the OAC-SR group also displayed a decrease, consistent

with the structural changes of lignin-cross-linked hemicellulose or cellulose in pretreated cotton straw [29,32].

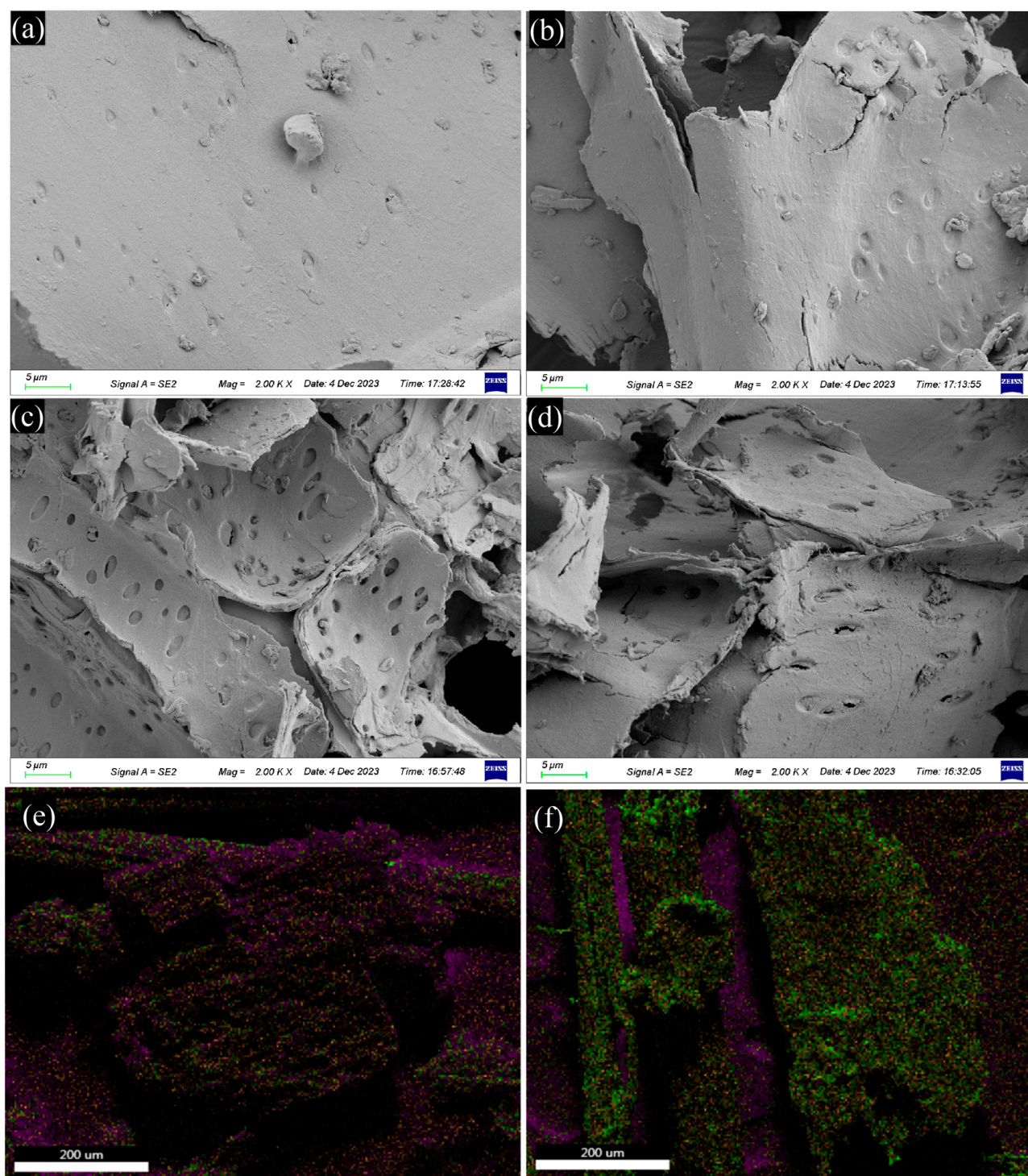


Figure 4. SEM images of crushed raw cotton straw (a), Pure water treatment cotton straw (b), OAC-SR (c), OAC-SR after adsorption of Na⁺ (d), OAC-SR mapping images before (e) and after (f) Na⁺ adsorption of straw materials. OAC-SR is the oxalic acid-modified cotton straw salt-absorbing water-retention agent. The red color and green color represented the surface C and Na elements distribution of cotton straw in (e,f).

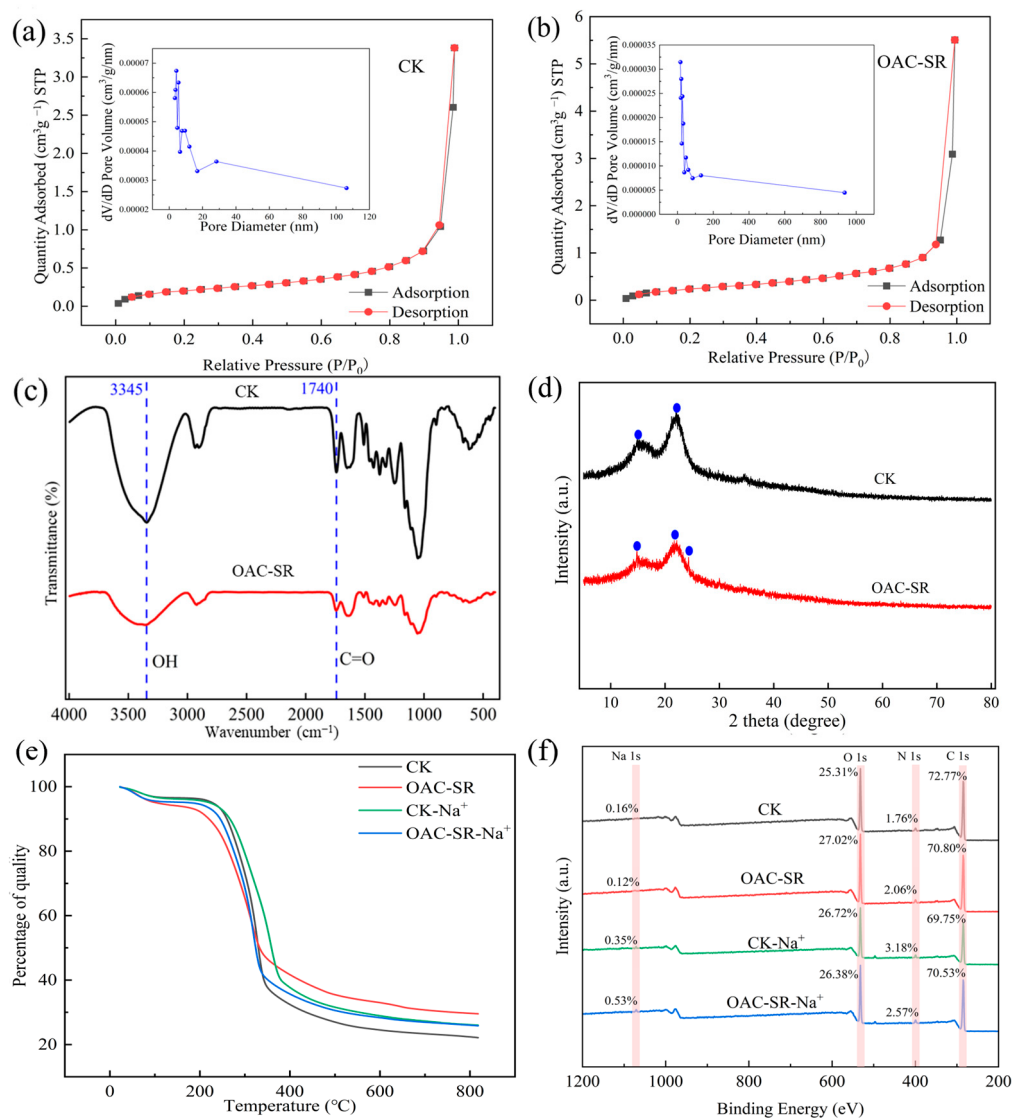


Figure 5. BET isotherms for (a) CK and (b) OAC-SR. (c) FTIR spectra and (d) XRD patterns of OAC-SR and CK. (e) TG-DSC curves and (f) XPS full spectra of OAC-SR, CK, OAC-SR- Na^+ , and CK- Na^+ . CK represents the pure water treatment cotton straw; OAC-SR represents the oxalic acid-modified cotton straw salt-absorbing water-retention agent.

The characteristic absorption peak at $2\theta = 22.5^\circ$, according to the crystalline region of cellulose, became weak after oxalic acid pretreatment, associated with the destruction of the internal crystalline structure of cotton straw (Figure 5d) [13,27]. The CrI value of the OAC-SR group was decreased from 46.7% to 34.2%, indicating the destruction of the crystalline region of cellulose in the pretreated cotton straw [13]. The proportion of stable components in oxalic acid-pretreated straw was increased by 7–8% before and after adsorption (Figure 5e), corresponding to the $\text{C}=\text{O}$ stretching vibrations at $1720\text{--}1750\text{ cm}^{-1}$ within the acetyl ester chain mainly from lignin (Figure 5c). Notably, oxalic acid pretreatment enhanced the surface C-O content of cotton straw relative to crushed untreated straw (Figure 5f). The fraction of C-OH/C-O-C on the surface of pretreated cotton straw increased from 24.6% to 40.1%, implying the more hydrophilic functional groups exposed on the surface of straw (Figure 6) [29]. The surface Na content of cotton straw was increased with salt absorption (Figure 5f and Figure S2). Compared to that of crushed untreated cotton straw materials, oxalic acid-pretreated straw displayed a 35% higher surface Na

content after salt adsorption, consistent with previous comparative salt adsorption test results (Figure 3).

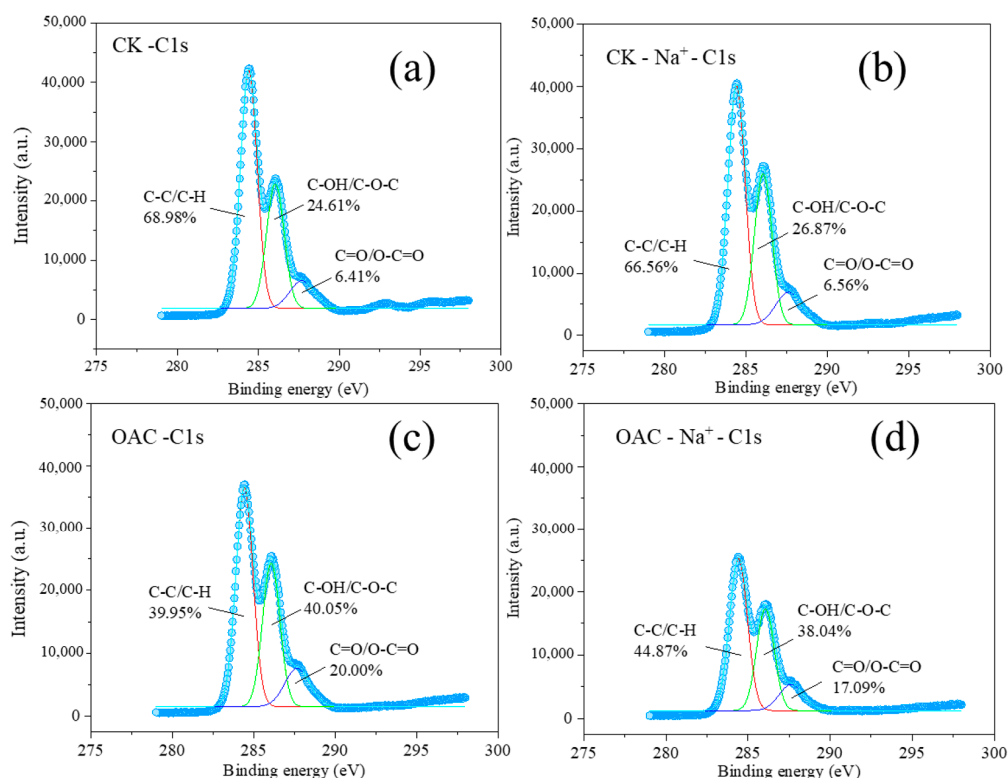


Figure 6. The fractions of C-C/C-H, C-OH/C-O-C, and C=O/O-C=O on the surface of straw materials from XPS spectra C1s region. The CK and CK-Na⁺ represent the pure water treatment cotton straw before and after adsorption of salt, respectively. The OAC and OAC-Na⁺ is the oxalic acid pretreated cotton straw before and after adsorption of salt, respectively. (a,b) represent the XPS spectra C1s region for the CK group before and after the adsorption of Na⁺, respectively. (c,d) represent the XPS spectra C1s region for the OAC group before and after adsorption of Na⁺, respectively.

3.4. Adsorption Capacities of Materials Studies

3.4.1. Effect of Adsorption Conditions on Na⁺ Adsorption of OAC-SR

The cotton straw materials of the OAC-SR and CK groups displayed an overall increasing trend in Na⁺ adsorption with rising initial NaCl concentration (Figure 7a). Additionally, the OAC-SR demonstrated a from 8.2 to 9.9-fold higher adsorption capacity for Na⁺ than that of the CK group (Figure 7a). Similarly, as the cotton straw material dosage increases, the Na⁺ adsorption capacity of materials first enhanced and then reached a plateau (Figure 7b). The maximum Na⁺ adsorption capacity of OAC-SR was 3.09 mg/g, which was 60.10% higher than that of the CK group at a dosage of 1.0%. These results further demonstrated that the salt adsorption capacity of OAC-SR was independent of the material dosage.

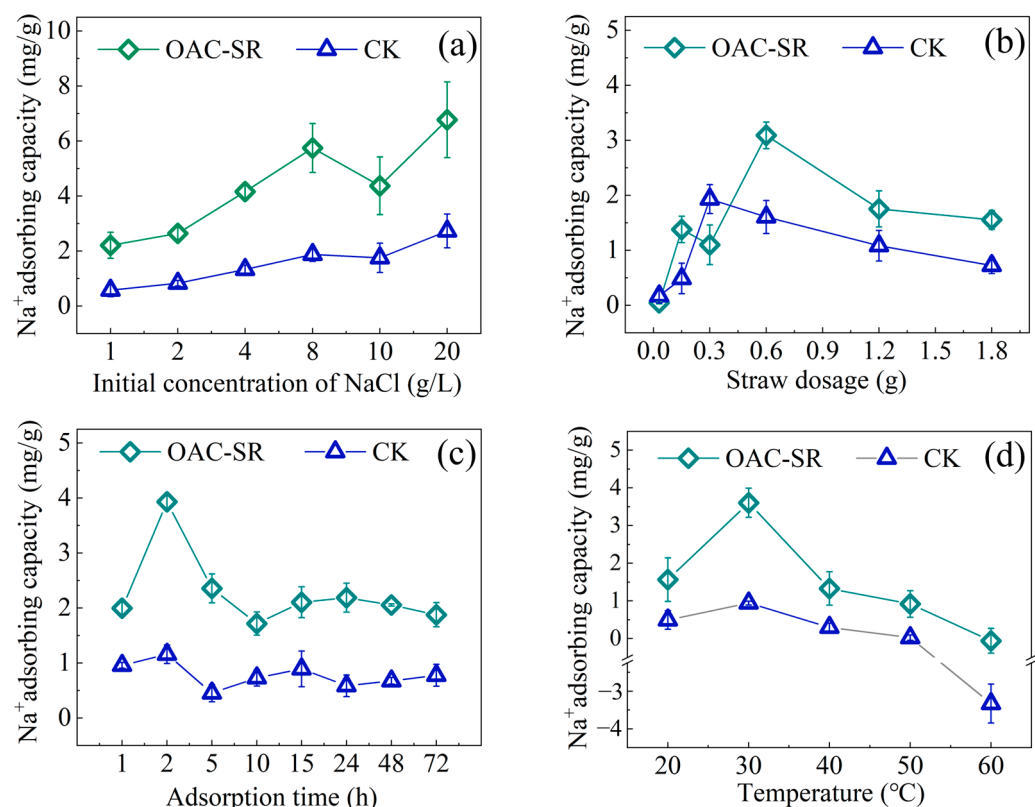


Figure 7. Effect of different conditions ((a): initial NaCl concentration, (b): straw dosage, (c): adsorption time, (d): adsorption temperature) on the adsorption performance of cotton straw materials pretreated by organic acid. CK represents the pure water treatment cotton straw; OAC-SR represents the oxalic acid-modified cotton straw salt-absorbing water-retention agent.

With increasing adsorption time, the Na⁺ adsorption capacity of cotton straw materials under different pretreatments exhibited an overall trend of initial increase, subsequent decrease, and eventual stabilization (Figure 7c). Within 2 h, the Na⁺ adsorption capacity of OAC-SR increased from 2.00 mg/g to 3.93 mg/g. The maximum adsorption capacity of OAC-SR was 2.39-fold higher than that of CK. In the temperature range of 20–30 °C, as the temperature increased, the Na⁺ adsorption capacity of straw materials showed an increasing tendency (Figure 7d). The maximum adsorption capacity of OAC-SR at 30 °C was 2.83-fold higher than that of the CK group. On the contrary, when the temperature was above 40 °C, high temperature was disadvantageous to the Na⁺ adsorption capacity of the straw materials. The result indicated that the Na⁺ adsorption process in straw materials was an exothermic reaction. Then, by measuring the field temperature, the salt absorption capacity of cotton straw may be predicted.

3.4.2. Effects of Cations and Dissolved Organic Matter on Na⁺ Adsorption of OAC-SR

In the mixed-ion system, the adsorption capacity for Na⁺, K⁺, Mg²⁺, and Ca²⁺ showed a reduced tendency compared to that of cotton straw materials in single-ion systems (Figure S2). It demonstrated the competitive effects of different ions on the cotton straw materials. The adsorption capacities of OAC-SR were at least 60.7% higher ($p < 0.01$) than those of the CK group in the single-ion or mixed-ion systems.

To investigate the effects of dissolved organic matter from soil solutions on the adsorption capacity of cotton straw materials, we collected natural saline–alkali soils from farmland to extract and dilute with deionized water to obtain the different concentrations of soluble matrix (Section 2.4.3). As shown in Figure 8, for both the OAC-SR and CK groups, the adsorption capacity of cotton straw towards different ions (i.e., Na⁺, K⁺, Mg²⁺, and

Ca^{2+}) displayed a decreasing trend with increasing dilution ratio. Compared to that of CK, the OAC-SR materials showed at least 54.7% higher adsorption values towards four cations. This result implied that the dissolved organic matter extracted from saline-alkali soils solution had an adverse effect on the adsorption of OAC-SR towards various cations. It further confirmed that salt ion adsorption occupies the adsorption sites of straw materials [29,32].

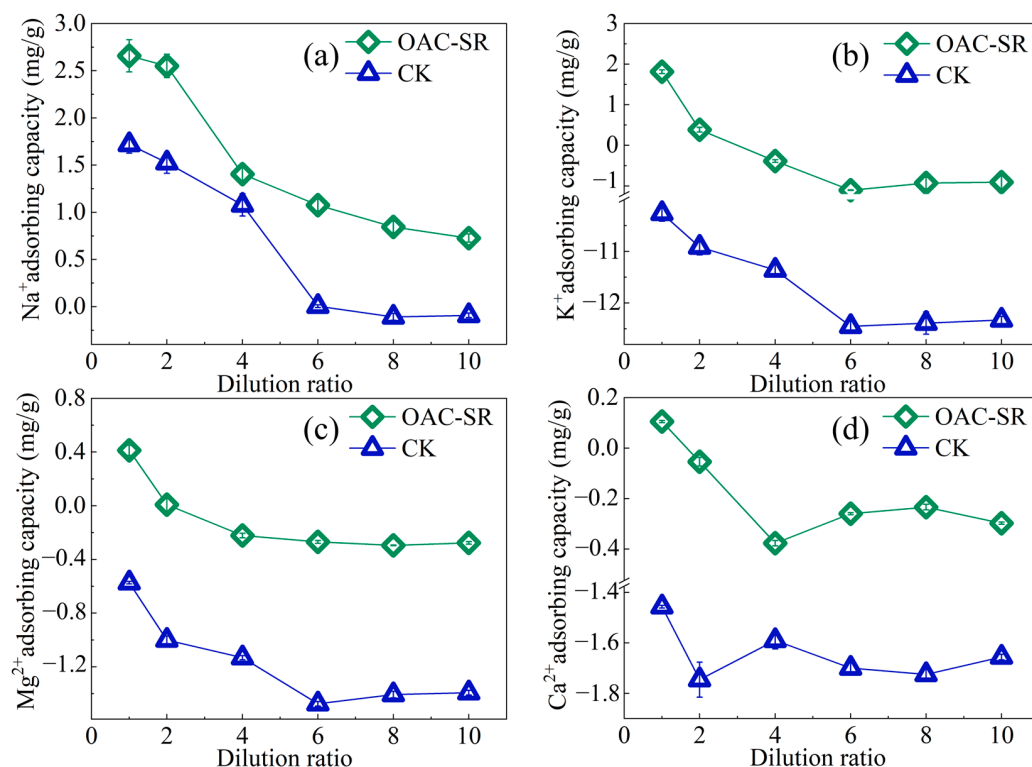


Figure 8. Effect of organic acid pretreatments of cotton straw on adsorption of different ions ((a): Na^+ , (b): K^+ , (c): Mg^{2+} , (d): Ca^{2+}) from saline soil leachate. The X-axis represents the dilution factor of the solution. The soil extract solution was obtained by a water-to-soil ratio of 5:1, as the initial mixed ion solution; then, it was diluted 2-, 4-, 6-, 8-, and 10-fold to investigate the effects of different ion concentrations.

The Na^+ adsorption capacity of OAC-SR in six soils was investigated to identify the effects of different soil physicochemical characteristics (Figure S3). For the 0.5%, 1.0%, and 2.0% salt content of six soils, the Na^+ adsorption capacity of OAC-SR consistently exceeded that of the CK group (Figure S3). Additionally, with the increasing NaCl content in the six soils, OAC-SR displayed an increase by 0.78–2.65-fold for Na^+ adsorption across six soils of three salinity levels. The result further confirmed that the salt adsorption advantage of pretreated cotton straw was independent of soil physicochemical characteristics.

3.5. Salt Barrier Effect of OAC-SR on the Salt Distribution in Soils

Through soil column experiment, the salt distribution in soil was analyzed to investigate the transport and distribution of salt ions under the adsorption barrier effects of OAC-SR materials (Table 1).

Table 1. Soil pH, conductivity, and Na⁺ content in the upper (0–10 cm depth of soil) and lower layers (10–30 cm depth of soil) of the cotton straw landfill layer.

Name	pH		Conductivity (mS/cm)		Na ⁺ Content (mg/g)	
	Upper	Lower	Upper	Lower	Upper	Lower
CK	7.70 ± 0.09 a	7.69 ± 0.07 a	2.37 ± 0.03 a	2.76 ± 0.06 b	0.14 ± 0.01 a	0.44 ± 0.01 a
CU	7.69 ± 0.15 a	7.67 ± 0.09 a	2.37 ± 0.03 a	2.88 ± 0.02 a	0.13 ± 0.02 a	0.48 ± 0.03 a
CT	7.68 ± 0.09 a	7.72 ± 0.04 a	2.31 ± 0.04 a	2.59 ± 0.03 c	0.14 ± 0.01 a	0.38 ± 0.02 b

Note: The values in this table are all means ± standard errors. Different letters in the same column indicate significant differences ($p < 0.05$). CK indicates no landfill, CU indicates landfill of raw straw, and CT indicates landfill of oxalic acid pretreated cotton straw. Different lowercase letters indicate significant differences between different treatments ($p < 0.05$)

The OAC-SR-amended group (CT) displayed no significant effect on soil pH, compared with the untreated cotton straw group (CU). Additionally, according to electrical conductivity and Na⁺ content, the addition of untreated and pretreated cotton straw materials caused no significant difference from the CK group (Table 1). On the contrary, both electrical conductivity and Na⁺ content in the upper and lower soil layers of CT groups were decreased by 2.53% to 13.6% ($p < 0.05$), compared to that of the CK group. Although electrical conductivity and Na⁺ content decreased in the upper soil column after OAC-SR burial, the electrical conductivity and Na⁺ content in the lower soil layer were significantly lower than the CK and CU groups. The OAC-SR materials reduced salt ion content in the subsoil beneath straw residues and provided an opportunity for Na⁺ retention during leaching processes. These results verified the barrier effects of OAC-SR materials on the mobility of salt ions in the soils.

3.6. Effects of Indoor Potted Plant Experiments

3.6.1. Effect of OAC-SR Addition on Soil Salinity in Potted Plants

The effect of OAC-SR incorporation on soil salinity in the upper layer above the cotton straw interlayer was more pronounced across different soil salinity levels (Figure 9a). With the increase in salt content in soils, the Na⁺ concentration in the upper soil layer of the OAC-SR group (CT) was decreased by 23.3–26.0% ($p < 0.05$) compared to that of the CK group. Meanwhile, the Na⁺ concentrations of CK and CU showed no significant difference above the 0.5% salt content in the soils (Figure 9a). On the contrary, for saline soils below 0.5%, the addition of untreated or pretreated cotton straw to the saline–alkali soils displayed a negligible effect on Na⁺ adsorption. It further implied the weak physical surface adsorption of cotton straw materials on the low concentration of salt ions. Combined with the results of the soil column experiment and mixed-ion system (Figure 8 and Table 1), it demonstrated that the pretreated cotton straw had a better barrier effect, dependent on more adsorption sites (Figures 4–6) [13,29].

In natural saline–alkali soils with subsoil salinity levels of 0.1% and 1.0%, the Na⁺ content in the lower soil of the OAC-SR group was reduced by 13%, 36%, and 5% ($p < 0.05$), respectively, compared to that of the untreated cotton straw group (Figure 9b). The addition of untreated cotton straw to the 0.1–1.0% saline soils did not change the Na⁺ content in the lower soil. The OAC-SR group showed a decrease of 7.69% and 20.01% for 0.1% and 1.0% saline soils compared to the CK group. These results further demonstrated the effects of an active barrier of OAC-SR materials on the transportation of Na⁺ ions by adsorbing and fixing upper-layer salts in the soils.

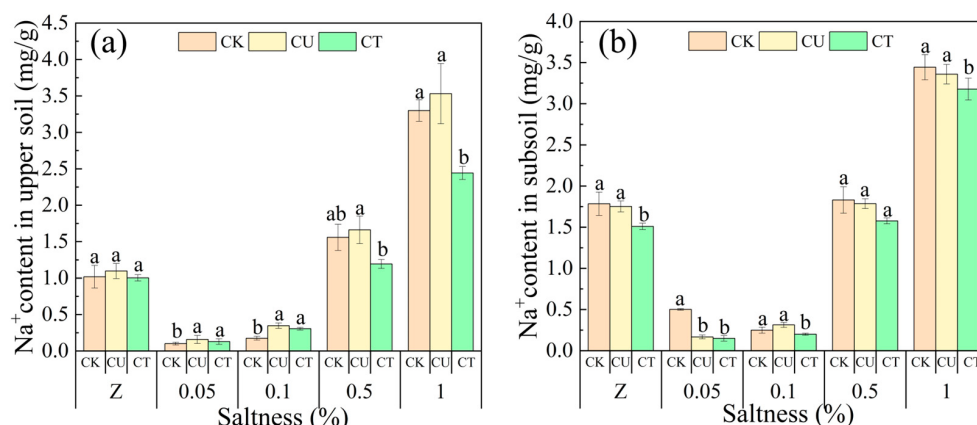


Figure 9. Effect of oxalic acid pretreatment of cotton straw on soil salinity in the upper (a) and lower (b) soil layers in pots. Note: CU indicates the addition of raw cotton straw; CT indicates the addition of oxalic acid pretreated cotton straw; CK indicates the control group without the addition of cotton straw; Z indicates the natural saline soils without the addition of salt ions. The upper soil refers to the soil layer above the straw layer, ranging from 1 to 10 cm below the surface. The lower soil layer refers to the soil below the straw layer. Different lowercase letters indicate significant differences between different treatments ($p < 0.05$).

3.6.2. Effect of OAC-SR Addition on Soil Moisture in Potted Plants

The soil moisture content in the upper layer of natural saline–alkali soil and straw-layered substrates with salt contents of 0.05%, 0.1%, and 1.0% was significantly increased by 16–43% ($p < 0.05$) compared to the topsoil moisture content of the CK group (Figure S4a). In soil with a salt content of 0.5%, although the upper soil moisture content of the CT group did not reach a significant difference, it still showed a marked increase compared to the upper soil moisture content of the CK group (Figure S4a). In natural saline–alkali soil and subsoil with a salt content of 1.0%, no significant differences were observed in soil moisture content among the three groups. The subsoil layer with a salt content of 0.5%, the soil moisture content of the OAC-SR group was 1.52- and 1.43-fold higher ($p < 0.05$) than that of CK and CU groups, respectively (Figure S4b). Combined with the increase in surface hydrophilic functional groups (C-OH/C-O-C) on the surface of pretreated cotton straw (Figures 5 and 6), it further verified that the exposed hydrophilic groups of OAC-SR can adsorb and retain the moisture in the soils.

3.6.3. Effects of OAC-SR Addition on Soil Microorganisms in Potted Plants

Figure 10 illustrates the distribution of bacteria (Figure 10a–c) and fungi (Figure 10d–f) in topsoil containing 0.1%, 0.5%, and 1% salt content. In the from 0.1% to 1.0% salinity soils, the proportion of OTUs in the topsoil of the OAC-SR (CT) showed a 29–52% increment depending on the salt content ($r = 0.956$), compared to that of the CK group. Additionally, the Chao1, Shannon, and Simpson indices for bacteria in the 0.1% to 1.0% salinity topsoil of the OAC-SR (CT) were increased from 1.12% to 21.25%, respectively (Table S3). As shown in Figure 10g, among the top 10 bacterial genera, the relative abundance of *Sphingomonadaceae* and *Gemmatimonadaceae* in the upper soil layer of the CT was from 26% to 63% higher ($p < 0.05$) than that of the CK and CU groups.

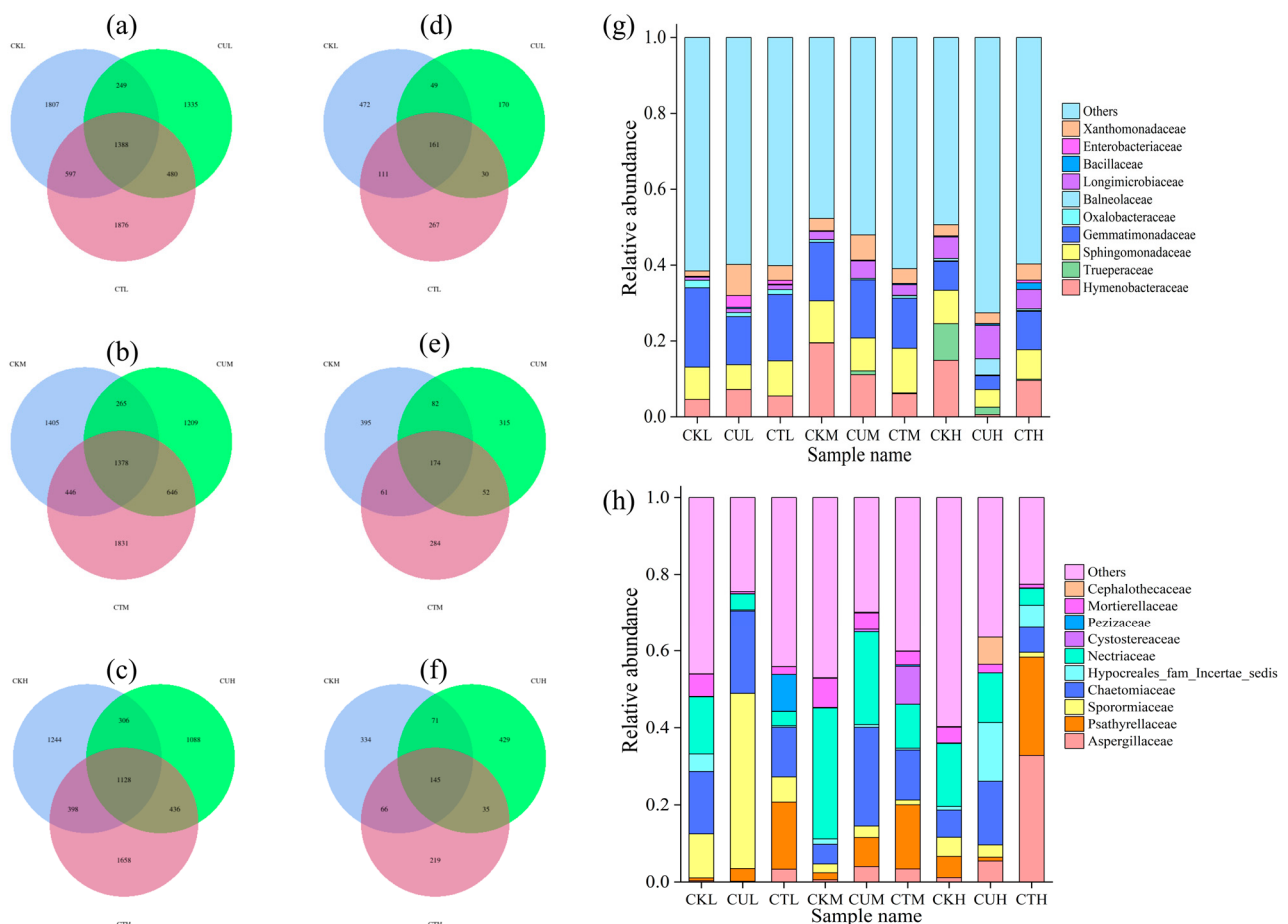


Figure 10. OTUs Venn plots of bacteria (a–c) and fungi (d–f) in upper soil layers with salt content of 0.1% (a, d), 0.5% (b, e), and 1.0% (c, f); relative abundance distribution of TOP10 bacteria (g) and fungi (h). (Note: CK, CU, and CT represent no cotton straw landfill, raw straw landfill, and oxalic acid pretreated cotton straw landfill, respectively. L, M, and H represent soils with 0.1%, 0.5%, and 1.0% salinity, respectively).

The OTUs of fungi displayed similar results in the from 0.1% to 1.0% salinity soils (Figure 10d–f). The proportion of OTUs in the topsoil of the OAC-SR (CT) was increased by 9% to 48% compared to that of the CK and CU groups. The diversity index of fungi (i.e., Chao1, Shannon index, and Simpson index) showed a diverse change with the salt content of soils. Under low saline soils (0.1%), the diversity index of fungi for the CT group displayed a from 17% to 33% increment compared to that of the CK and CU groups (Table S4). When the soil salt content reached 1.0%, the diversity indices of fungi in the topsoil for the CT group decreased from 22% to 33%. As shown in Figure 10h, among the top 10 fungal genera, *Psathyrellaceae* relative abundance in the topsoil of the CU group was increased by 81%, 55%, and 96% in soils with 0.1%, 0.5%, and 1.0% salinity, compared to that of the CK group. On the contrary, compared to that of the CK group, the relative abundance of *Chaetomiaceae* in the upper soil layer of the CT group was decreased from 40% to 60%, depending on the range from 0.1% to 1.0% in the salt content of the soils ($r = 1.00$, $p = 0.002$). It indicated that the addition of OAC-SR may reduce the bioactivity of some fungal populations as salt content increases during the 50-day incubation.

3.7. The Field Verification of OAC-SR

As shown in Figure 11a,b, no significant differences in soil pH and electrical conductivity were observed between treatment groups at the 0–20 cm depth. Compared to the CK, the CT group displayed a significant 6% reduction in soil pH at the 20–40 cm depth

($p < 0.05$). This may contribute to the addition of oxalic acid pretreatment to destroy the structure of cotton straw and increase the adsorption sites of soils (Figures 5–7). It was noteworthy that the CT group exhibited a substantial difference in electrical conductivity between the 0–20 cm and 20–40 cm soil layers, associated with the 18% decrease in electrical conductivity in the 0–20 cm layer.

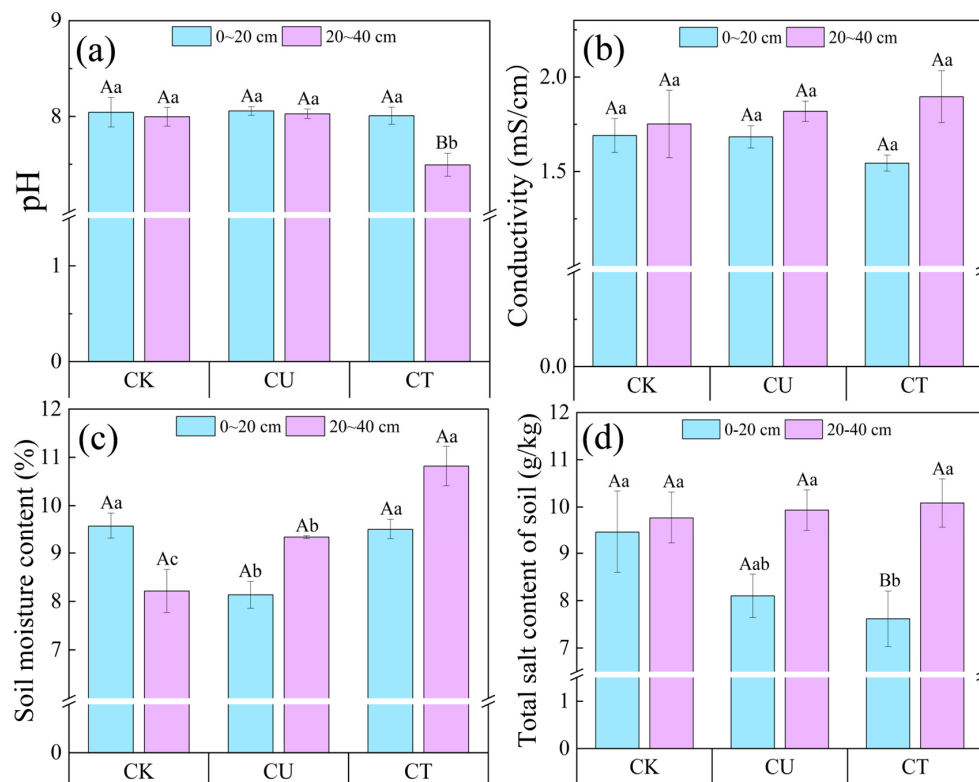


Figure 11. Effect of oxalic acid pretreatment of cotton straw on soil moisture in the upper (a) and lower (b) layers. (Note: CK, CU, and CT represent no cotton straw addition, raw straw addition, and oxalic acid pretreated cotton straw addition, respectively.) The upper layers were 0–20 cm depth of soil, and the lower layers were 20–40 cm depth of soil. (a) PH, (b) Conductivity, (c) Soil moisture content, (d) Total salt content of soil. The different uppercase and lowercase letters indicate statistical significance between different treatments at the $p < 0.05$ level for 0–20 cm depth of soil (uppercase letters) and 20–40 cm depth of soil (lowercase letters), respectively.

The moisture content of 0–20 cm topsoil in the CU group was significantly reduced by 15.1% ($p < 0.05$) compared to that of the CK group (Figure 11c). Meanwhile, significant differences ($p < 0.05$) were observed in soil moisture content at the 20–40 cm depth across all treatment groups. Compared to the CK group, the soil moisture content of the CT group was increased by 11% for CU and 24% for the CT group, respectively. It is noteworthy that the moisture content of the 20–40 cm soil layer was relatively higher than that of the 0–20 cm layer in the CU and CT groups. The increase in soil moisture content is identical to the result of potted plants and the exposure of hydrophilic functional groups on the surface of pretreated cotton straw (Figures 5, 6 and S4).

The total soil salinity of the CT group in the 0–20 cm layer was significantly reduced by 19% and 6% ($p < 0.05$), respectively, compared to that of the CK and CU groups (Figure 11d). Under identical burial treatments, the CT group displayed a 24% reduction ($p < 0.05$) in soil total salt content for the 0–20 cm soil layer compared to that of the 20–40 cm soil layer. Combined with the structural characteristics of pretreated cotton straw (Figures 4–6), these results further demonstrated the stronger surface adsorption function of OAC-SR materials towards the slat of the 0–20 cm soil layer than that of the CK or CU groups.

Obviously, in soils with salt contents of 0.1%, 0.5%, and 1.0%, the germination rate, plant height, and plant dry weight of the cotton plants for the CT group showed overall significant increases ($p < 0.05$), compared to those of the CK and CU groups (Figure S5). Additionally, the increments from the OAC-SR materials were dependent on the salt content ($r > 0.92$, $p < 0.05$). Meanwhile, the soil organic carbon content of the CT group was increased by 66.7% compared to that of the CU group (Figure S6). The release of organic nutrients from OAC-SR materials can provide a more beneficial microenvironment for the turnover of other inorganic elements, promoting plant growth (Figures S5 and S6). These results further indicated that the OAC-SR materials can alleviate the salt damage to crops and promote cotton growth by mediating the soil salt distribution and moisture content.

4. Discussion

In this study, the effects of oxalic acid pretreatment on cotton straw adsorption associated with pore-expanding, salt-absorbing, and water-retaining performance in diverse saline–alkali soils were evaluated. The practical effectiveness of the pretreated materials in promoting the emergence and growth of cotton seedlings was further verified through the experiments of soil columns, pot experiments, and field trials (Figures 9, 11, S5–S7, and Table 1). The results demonstrated that OAC-SR pretreatment caused a significant increase in the specific surface area, salt-ion adsorption capacity, and water-retention performance of cotton straw materials (Figures 5, 9, 11, and Table 1). In addition, a functional shift from “salt suppression” to “growth promotion” was achieved through a carboxylated interface of pretreated cotton straw incorporation to saline–alkali soils (Figures S4 and S5). The retention of salt and moisture, and the regulation of microbial communities in saline–alkali soil further alleviated the salt damage of the crop and promoted the growth of cotton seedlings (Figures 11 and S5) [2].

4.1. Analysis of Pore Expansion, Salt Absorption, and Water Retention Properties of OAC-SR Materials

The simultaneous amplification of pore volume, Na^+ adsorption, and water retention in OAC-SR materials was combined with a mild-temperature “soft-etching” route for superficial structures and a subsequent carboxyl-rich interfacial reconstruction (Figures 4–6). Unlike conventional biochar that demands $\geq 400^\circ\text{C}$ to conduct porosity [22–24], 0.2% oxalic acid at 60°C selectively cleaved $\beta\text{-O-4}$ aryl-ether bonds in lignin while leaving the cellulose crystalline skeleton broken (Figures 4 and 5) [29,32]. The specific surface area of pretreated cotton straw was enhanced by 24% and the maximum pore volume was doubled (Figure 5a,b), providing physical transmission and load channels that lower capillary blockage for both water and ions [41–44].

The chemisorption of Na^+ of straw material is governed by weak electrostatic attraction [29,32]. The $\text{O}=\text{C}-\text{O}$ proportion from XPS C1s was increased from 6.4% to 20.0% after 24 h oxalic acid pretreatment (Figure 5). Additionally, a new Na 1s peak at 1071.2 eV appeared after adsorption, which was identical to that of the sodium oxalate standard. The structural characteristics under investigation yielded an increase in salt adsorption in different solution systems (Figures 8 and S3). This result was an order of magnitude higher than that of untreated cotton straw [45]. The high selectivity of OAC-SR material was preserved in Na^+ , K^+ , Mg^{2+} , and Ca^{2+} co-existed systems, and retained 61% of its single-Na uptake, whereas raw straw lost 92% (Figure 8). It further illustrated the advantage of specific weak electrostatic attraction with the structural changes of pretreated cotton straw (Figures 4–6).

Water retention of materials is achieved through a dual-layer mechanism [2,46]. The first layer arises from chemical adsorption: the negatively charged carboxylated surface forms strong hydrogen bonds ($\text{O}-\text{H} \cdots \text{O}$) with polar water molecules [47]. The second layer

is physical: the opened interconnected pores generate capillary condensation described by the Kelvin equation, significantly delaying desorption [48]. Under harsh evaporation (at 50 °C), OAC-SR still retained 33% of its initial water after 5 h, 98-fold higher than raw straw and three-fold higher than water-washed straw (Figure 3), reaching the performance level of polyurethane–straw composites but without micro-plastic residues [49,50]. At a field-relevant scale, a 2 cm OAC-SR interlayer increased 0–20 cm soil moisture by 24%, comparable to a biochar amendment applied at three times the carbon input (Figure S4) [51], highlighting the high water-regulation efficiency of the oxalic acid route. The more hydrophilic functional groups (C-OH/C-O-C) on the surface of pretreated cotton straw were increased from 24.6% to 40.1% (Figure 6), and the increase in specific surface area and pore volume through destruction of rigid cell structure (Figures 4 and 5) contributed to the higher adsorption capacity of OAC-SR [13,29,46].

The “mild-temperature soft-etching and interfacial carboxylation” strategy endowed OAC-SR materials with a tri-functional character: expanded pores for rapid water/ion transport, specific-COONa sites for selective Na⁺ locking, and a dual-layer water retention network (Figures 4–6) [46]. The increase in Na elements on the surface of pretreated cotton straw further demonstrated the adsorption capacity of straw materials (Figure 4e,f). Oxalic acid pretreatment destroyed the dense crystalline structure of straw (Figure 5d) and exposed more surface hydrophilic hydroxyl functional groups (Figure 5c), which made the material surface structure rougher (Figure 4) and increased the adhesion capacity of salt ions (Figure 3). The specific surface area of cotton straw was also increased after oxalic acid pretreatment. Reorganization of the physical structure of cotton straw contributed to the exposure of surface chemical functional groups in the pretreated cotton straw (Figures 4 and 5). Meanwhile, the increased C=O functional groups on the pretreated straw surface were more conducive to the adsorption of salt ions, forming weak adsorption forces (Figures 5f and 6). Further, the NaCl crystalline diffraction peak was observed on the pretreated straw material of adsorption salt (Figure 5d). Additionally, the increase in thermally stable substances derived from salt in the pretreated cotton straw confirmed its salt adsorption capacity (Figure 5e). Combined with the increase in Na elements on the surface of pretreated cotton straw (Figures 4e,f and 5d), it further confirmed the surface adsorption and pore filling mechanism of the pretreated straw materials. These advantages were achieved under mild conditions and without external energy or synthetic monomers, offering an environmentally benign and scalable alternative to both alkaline biochar and synthetic plastic resins for saline-soil remediation [46,51].

4.2. The Effects of OAC-SR Addition on the Physicochemical Properties of Saline–Alkali Soil

The OAC-SR addition to saline soils developed a 2 cm “active barrier” that simultaneously re-partitioned water, intercepted Na⁺, and buffered alkalinity in the 0–40 cm profile (Figure S4 and Table 1). At the proportion of 10 g·kg^{−1} incorporation, OAC-SR increased 20–40 cm soil moisture and decreased 0–20 cm EC value (Figure 11). Guan et al. applied acidified biochar to improve saline–alkali soils, achieving similar results [52]. However, biochar requires preparation under anaerobic and high-temperature conditions [53], resulting in increased energy consumption and carbon input compared to the OAC-SR pretreated process. Simultaneously, biochar materials exhibit alkaline properties, and long-term application will increase soil pH [52]. In contrast, oxalic acid pretreatment may activate soil salt ions and has a certain solubilizing effect, which contributes to the downward movement of salt ions in the upper soil layer with soil solution [2,46].

Meanwhile, despite zero exogenous Ca²⁺ supply, the addition of OAC-SR decreased the pH value of 20–40 cm topsoil from 8.04 to 7.56 and exchangeable sodium percentage (ESP) from 19.1% to 14.3% (Figure 11). This is because oxalate acts as a weak-acid chelator

that solubilises lattice Ca^{2+} , enabling in situ Ca^{2+} - Na^+ exchange [38]. A meta-analysis covering more than 1400 data sets indicated that biochar decreased ESP by 28.9% and enhanced soil cation exchange capacity (CEC) by 49%, outperforming gypsum-based materials, which averaged 29% ESP reduction [54].

The OAC-SR pretreatment reduced total salinity by 19% at the 0–20 cm depth while significantly increasing organic carbon and polysaccharide content within this soil layer (Figures 9, S6 and S7). The increased organic carbon further confirmed the turnover effects of accelerated decomposition of cotton straw in saline–alkali soils. In the Hetao irrigation district of China, a $30 \text{ t} \cdot \text{ha}^{-1}$ organic–inorganic composite amendment achieved 46%, 33%, and 27% reductions in Na^+ , Cl^- , and EC, respectively, but required twice the OAC-SR addition dosage and contained desulphurisation gypsum [55]. The increase in organic nutrients in soil helps to alleviate crop salt damage (Figure S5) [2]. It was reported that rice straw returning significantly reduced the soil exchangeable sodium percentage (ESP), saturated paste extract (ECE), sodium adsorption ratio (SAR), pH, Na^+/K^+ and $\text{Na}^+/\text{Ca}^{2+}$ ratios, and increased cation exchange capacity (CEC) compared with CK [56]. Similarly, Rice straw returning significantly increased the availability of soil total N, alkali-hydrolysable N, $\text{NH}_4\text{-N}$, $\text{NO}_3\text{-N}$, available P, available K, soil organic matter (SOM), and enzyme activities of highly saline–alkali paddy soils. These advantages contributed to the increase in yield [56]. Although we did not obtain the cotton yield in the field experiments, the cotton growth (e.g., seedling emergence, plant height, dry weight) demonstrated a favorable yield-increasing effect for OAC-SR pretreatment (Figure S5). The oxalic acid can be detected during the decomposition of rice straw and is responsible for P solubilization from phosphate and udaipur rock phosphate [35]. Further, the long-term effects of oxalic acid pretreatment on cotton straw return will be focused on in future work.

In conclusion, OAC-SR addition reconstructed the cotton straw return of saline soils through a mild-temperature, low-carbon, and low-risk route. The results provide a field-verified, “waste-to-value” strategy for mediating saline soils in arid regions.

4.3. OAC-SR Addition Alteration of Microbial Ecological Function: From Salt Suppression to Growth Promotion

During the decomposition of straw, the oxalic acid, as an organic acid and a plant root exudate, was produced and was responsible for P solubilization [33–35]. Meanwhile, it can activate and release salt ions in soil to promote their mobility [29,30]. The OAC-SR addition not only restructured the physicochemical template of saline–alkali soils but also acted as an active carrier that converted a salt-stressed community into a growth-promoting consortium [56]. It can also significantly increase the saturated hydraulic conductivity and total porosity [56]. These advantages contribute to the positive turnover of soil microbes [57]. The 16S/ITS sequencing revealed that a 1.0% NaCl soil containing OAC-SR materials increased bacterial Shannon index by 4%, whereas it decreased the fungal Shannon index by 33% (Tables S3 and S4). This indicated that bacteria, with higher functional redundancy, assume the dominant role in maintaining nutrient cycling under hyper-osmotic stress patterns, which is consistent with global saline-soil surveys [58]. Although soil enzyme activity was not tested in our results, it has been identified that straw decomposition directly affected the soil chemical properties and indirectly affected soil enzyme activity [57].

The first driver of this shift is the carboxylated interface. These negatively charged sites preferentially adsorb extracellular proteins of gram-positive phosphate-solubilisers (*Bacillus*, *Pseudomonas*) via Ca^{2+} bridging, forming a “carboxyl- Ca^{2+} -bacterium” ternary complex [59]. As a result, the relative abundance of *Sphingomonadaceae* (a canonical P-solubiliser) was increased by 40% compared with untreated cotton straw addition, matching the $+0.8 \text{ log-unit increase per } \text{mmol} \cdot \text{g}^{-1}\text{—COOH}$ reported for citric acid-functionalized

biochar [60]. Second, the OAC-SR continuously leaked low-molecular-weight organic acids (LMWOAs). These C₂–C₄ molecules act as “microbial fast food” that rapidly stimulates r-strategists such as *Gammaproteobacteria*. LMWOAs increased soil respiration and boosted Chao1 abundance by 7–21%, corroborating previous metagenomic evidence from desert saline soils where LMWOA pulses raised the Shannon index 8% [61]. Third, the OAC-SR reprogrammed the fungal trophic structure. The saprotrophic genus *Psathyrellaceae*, capable of lignocellulose breakdown and humic acid production, was increased, whereas the root-pathogenic *Chaetomiaceae* declined (Figure 10h). A parallel field study in the Ningxia region of China found that every 1% increase in *Psathyrellaceae* abundance elevated soil IAA by 0.6 ng g^{−1} and wheat biomass by 5%. The cotton shoot dry weight responded with a 100% increment, suggesting that the “saprotroph–humus–IAA” axis was a major growth-promotion conduit [62]. Additionally, the drop in *Chaetomiaceae* lowered the risk of seedling damping-off, a frequent constraint in salt-affected cotton stands [63]. These positive effects deserve further in-depth study, especially the regulatory role of straw nutrient release on soil microbial community and structural functions under oxalic acid pretreatment.

At the network level, the OAC-SR increased average degree by 27% and reduced modularity by 15%, implying a more cohesive and resilient community (Tables S3 and S4). Such topological tightening is characteristic of healthy soils coping with osmotic stress, where enhanced cross-feeding and quorum sensing maintain metabolic continuity even when fungal hyphae are disrupted [64]. Notably, these ecological benefits were achieved without external mineral fertilizer or heat-intensive biochar, highlighting the sustainability of the oxalic acid pretreatment. Straw return can affect the structure of water-stable aggregates and the composition of soil microflora in saline–alkali soil. With the increase in the content of straw, the abundances of some genera (e.g., *Thermobacillus*, *Thermopolyspora*, and *Thermobispora*) were significantly increased, indicating that they display an important role in improving soil nutrient components and physicochemical properties by promoting saline–alkali soil activities and microbial communities [9,65]. Similarly, in our results, some plant-beneficial microbes (e.g., *Sphingomonadaceae* and *Gemmatimonadaceae*) were stimulated, with their relative abundance increasing by 26–40% and 27–63% respectively (Figure 10). It further indicated that the oxalic acid pretreatment can activate the microbial activity in the short term (50-day cultivation) through promoting the release of organic nutrients derived from straw decomposition, and facilitating the mobility of salt ions in soils (Figures 9, 10, S6 and S7). The above effects will ultimately further promote crop growth and increase yields [65,66].

It should not be overlooked that the calcium oxalate, as an insoluble substance, may be formed by the pretreatment of oxalic acid. The oxalic acid, as an acidic amendment, can neutralize soil alkalinity by introducing H⁺, which neutralizes OH[−] released by CO₃^{2−} and HCO₃[−] in the composite clay minerals, effectively lowering soil pH value and mitigating alkalization [2,67]. However, the overuse of acidic amendments may disrupt the acid–base balance and negatively affect microbial populations, leading to soil acidification [2]. Meanwhile, the positive side that pretreated cotton straw may regulate soil nutrient turnover through influencing interfacial microbial community deserves further in-depth investigation. Then, the long-term, deep-seated positive or negative effects of oxalic acid pretreatment require ongoing observation.

In summary, combined with pot and field experiments, the OAC-SR can reduce the salt content of the 0–20 cm soil layer and significantly increase the content of organic carbon and polysaccharide within this layer (Figures 9, S6 and S7). The remarkable effects on the cotton growth (e.g., seedling emergence, plant height, dry weight) can be attributed to the release of more beneficial organic nutrients from cotton decomposition (Figures S5–S7) and the improvement in soil microbial community activity (Figure 10 and Tables S3 and S4).

The OAC-SR addition to saline soils acted as a functional lever that tilts the microbial balance from salt-tolerant survival to plant-growth promotion: carboxyl groups recruit beneficial bacteria, low-molecular-weight organic acids (LMWOAs) fuel rapid metabolism, and fungal re-assembly channels nutrients into phytohormone production [61]. The comprehensive effect of OAC-SR offers a field-ready, low-energy strategy to kick-start biological fertility in saline soils while avoiding the microplastic or heavy-metal risks associated with synthetic amendments.

5. Conclusions

This study demonstrated the optimal pretreated conditions of oxalic acid for cotton straw: 60 °C pretreatment temperature, 0.2% acid dosage, and 1 d pretreatment time. The pretreated cotton straw displayed a carboxyl-rich cotton straw material with 24% higher surface area and 33% water retention within 5 h. The field trials demonstrated a significant increase in organic carbon and polysaccharide contents derived from decomposed cotton straw in the 0–20 cm soil layer. In the pot experiment, embedding OAC-SR material into diverse saline–alkali soils formed an “active barrier”, increasing soil moisture content from 16% to 43% in the 0–20 cm surface layer while reducing the Na⁺ content by approximately 30%. The addition of OAC-SR materials can enhance cotton emergence by 44–88% and increase biomass by doubling ($p < 0.01$), compared to that of untreated cotton straw. The 16S/ITS results showed an increase in the bacterial Shannon index by 4%, and plant-beneficial *Sphingomonadaceae* by 40% in salt soils with OAC-SR pretreatment. The feasible oxalic acid pretreatment reconstructs the cotton straw return of saline soils under a low-carbon and “waste-to-value” strategy to mediate saline soils in arid regions. Certainly, based on the fact of soil health and green development, in future studies, the long-term, deep-seated positive or negative effects of oxalic acid pretreatment require ongoing observation over multiple growing seasons. Optimal pretreatment dosages and durations can be further screened to adjust for the needs of diverse crops in different soils. To improve its scalability and practical applicability, oxalic acid pretreatment may be integrated into agricultural practices (e.g., soil basal fertilization or straw incorporation) without extra application costs. Meanwhile, the study on how pretreated cotton straw structure turnover regulates soil nutrient biotransformation through structural change of interfacial microbial community deserves further in-depth investigation in future work.

The most important finding in this study is as follows: oxalic acid pretreatment can destroy the structure of cotton straw to release more organic matter by biodegradation in the soils and enhance its adsorption capacity to reduce the risk of crop salt damage.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/agronomy15112657/s1>, Figure S1: Daily maximum and minimum temperature evolution chart of Turpan city, in Xinjiang, China, from April to October during the three-year period of 2022–2024; Figure S2: Effect of organic acid pretreatments of cotton straw on the adsorption of different ions; Figure S3: Na⁺ adsorption effects of organic acid pretreated cotton straw on six soils; Figure S4: Effect of oxalic acid pretreated cotton straw return on soil moisture in the upper (a) and lower (b) layers of potted plants; Figure S5: Effect of oxalic acid pretreatment of cotton straw on cotton growth; Figure S6: The effect of oxalic acid pretreatment of cotton straw on soil organic carbon content; Figure S7: The effect of oxalic acid pretreatment of cotton straw on soil polysaccharide content; Table S1: The information of soil salinity content from different soil sample; Table S2: Basic information of six soils in China; Table S3: Total number of bacterial species, dominant bacteria abundance index and diversity index; Table S4: Total number of fungal species, abundance index of dominant fungi and diversity index.

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