Research Article

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A novel superabsorbent material based on soybean straw: Synthesis and characterization

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Abstract: A novel superabsorbent copolymer (SAP) was developed by grafting acrylic acid onto the pretreated soybean straw (PSBS) using gamma-ray irradiation. The structure of soybean straw, treated soybean straw, and SAP were characterized through Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TG), and scanning electron microscopy (SEM). The effect of irradiation dose, the dosage of cross-link agent, and the monomer ratio of the graft copolymer on the water absorbency of the SAP was investigated. The highest water absorbency of 1,489 g/g in distilled water and 56 g/g in 0.9 wt% NaCl solution was obtained, when the ratio of acrylic acid (AA) to PSBS was 6:1. This irradiation technique is superior to traditional methods with advantages such as less heavy pollution, low-energy consumption, and high-water retention performance. The outcome is deemed to open up new pathways to synthesize environmentally safe superabsorbents with applications in the food, pharmaceutical, and medical industries.

Keywords: irradiation, soybean straw, graft, water absorption

1 Introduction

Superabsorbent polymers (SAPs) are three-dimensional cross-linked resins made of hydrophilic networks [1–3]. They absorb and retain large quantities of water even at elevated pressure and temperature conditions [4–6]. They have the required potential to be used as sorbents in a wide variety of applications including personal care products, infant diapers, and feminine hygienic products [7,8] to name a few. In addition, they have received considerable attention in agriculture, forestry, and horticulture as water-holding agents to address drought resistance and release fertilizers [9,10].

The basic materials for synthesizing SAPs are acrylamide-based products that predominantly originate from the petroleum processing byproducts [4,11–13]. Consequently, thus developed SAPs possess longer bio-breakdown periods of 4 years or more [14]. Such prolonged degradability is hazardous to the environment [15–17]. In this regard, the utilization of nontoxic and low-cost natural materials will be advantageous in the design and development of value-added and environmentally friendly SAPs. Substitutes from renewable biomass resources stand out as one of the viable alternatives and SAPs generated from biomass could be biodegradable [14,18,19].

Agriculture byproducts such as crop straws are abundant. They are a rich source of cellulose, hemicellulose, and lignin [20–22]. However, crop straws have not been properly explored in scientific and industrial applications. Instead, they are being treated as crop residues and left in the field for improving the soil quality apart from using a portion for animal feed. The cellulose of crop straws is one of the most widely distributed and abundant natural macro-molecules in nature. It has a high degree of polymerization, stable structure, and stable mechanical properties. It is a renewable resource with low price, biocompatibility, and good degradability [23]. Isolation and marketing of cellulose fraction from these biomasses could be useful to address biodegradability as well as to develop functional materials. Graft copolymerization on crop residues has not been explored to the best of our knowledge. Herein, the extract from soybean straw has been taken as the model system to synthesize a novel superabsorbent system by grafting acrylic acid. The structural, morphological, and functional properties of the composite are reported.

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2 Materials and methods

2.1 Materials

Acrylic acid (AA, analytical grade, without further purification) and N,N'-methylene-bisacrylamide (MBA, chemically pure) were from Shanghai Chemical Reagent Corporation (Shanghai, China). Soybean straw (SBS) was obtained from suburban Anhui Chizhou (Chizhou, China). All other commercially available solvents and reagents were analytical grade and were used as received. The normal saline was prepared by dissolving 0.90 g of sodium chloride (NaCl) in 99.10 mL of distilled water. The artificial urine was prepared by dissolving 1.94 g of urea, 0.11 g of magnesium sulfate heptahydrate (MgSO₄·7H₂O), 0.08 g of NaCl, and 0.06 g of calcium chloride (CaCl₂) in 97.81 mL of distilled water. Unless otherwise specified, double distilled water (with electrical conductivity of 6.40 µS/cm) was used throughout the experiment.

2.2 Pretreatment of soy dreg

The pretreated soybean straw (PSBS) was milled through an 80-mesh sieve and was treated with acetone and vacuum-dried for 24 h at 60°C. Later, 3 g of SBS was dispersed into 97 g of NaOH/urea solution with stirring for 5 min to obtain 3 wt% solution, which was stored under refrigeration (−15°C) for 12 h [24]. The frozen solid was thawed and stirred extensively at room temperature to obtain a transparent solution. It was then precipitated and washed with 70% EtOH. Subsequently, it was dehydrated using 95% anhydrous ethanol and dried at 60°C for 48 h. Finally, the regenerated SBS was ground and marked as PSBS.

2.3 Preparation of composites

Scheme 1 highlights the AA-g-PSBS preparation. Briefly, to a 100 mL solution (6% NaOH), 0.175 mol NaOH and 3 g of soybean straw (SBS) were added [25]; the $m_{AA}$ were 21, 27, and 33 g. Beyond the 6% NaOH solution, a certain amount of NaOH was needed in order to make the AA neutralization rate between 70 and 90%. Later, MBA was added to a 100 mL plastic beaker with mechanical stirring for 0.5 h. Dispersions were irradiated with $^{60}$Co gamma-rays at room temperature at the State Forestry Radiation Center (Hefei, China) facility. The obtained hydrogel was cut into small pieces and soaked in 75% ethanol for 5 h two times to remove nongrafted polyacrylic acid and other chemicals. The precipitates were collected and suspended in anhydrous ethanol for 2 h two times to remove water and then dried overnight at 60°C, for further analysis.

2.4 Water absorption

The water absorbency capacity of the acrylic acid-grafted PSBS (AA-g-PSBS) was measured at ambient temperature. The sample was dipped in distilled water and saline solution to reach the swelling equilibrium. The residual water was removed by filtering with a 120-mesh screen. The weight of the swollen polymer was determined until water ceased to drop, and the water absorbency ($Q$) of AA-g-PSBS was calculated using the following equation:

$$Q = \frac{m_2 - m_1}{m_1},$$

where $m_1$ is the weight of the dry sample and $m_2$ is the weight of the swelling sample (g). $Q$ is expressed as gram of water per gram of sample (g/g).

![Scheme 1: Preparation of AA-g-PSBS.](image-url)
2.5 Water-retaining property

In order to measure the water-retaining property of AA-g-PSBS, uniform particle sizes ranging from 100 to 200 mesh were used. Around 0.5 g of the graft copolymer was placed in a 1,500 mL beaker, and 1,000 mL of deionized water was added with stirring. Later, a 0.154 mm nylon mesh filter was used to drain the excess water at different time intervals; the residue was collected and weighed. The water swelling resin of the residue was in the dryer oven at 60°C, measured at hourly intervals. The water-retaining property of SAP was calculated using the following equation:

\[ Q = \frac{m_3}{m_2}, \]

where \( m_2 \) is the weight of the swelling sample (g) and \( m_3 \) is the weight of the dried swollen sample (g).

2.6 Structural characterization

FTIR spectra were collected using the Thermo Nicolet, NEXUS, TM by pressing the composite in KBr pellets. The surface morphology was examined using the JSM-5600LV SEM from JEOL, Ltd. The sample was coated with a thin layer of gold film at an acceleration voltage of 20 kV. The thermal stability of samples was recorded on a Netzsch STA-449C thermogravimetric analyzer (TGA) in the temperature range 25–600°C at a heating rate of 10°C/min. Dry nitrogen purge at a flow rate of 40 mL/min was used. Powder X-ray diffraction (XRD) measurements were carried out using Haoyuan DX 2700 with Cu Kα radiation (\( \lambda = 1.5406 \) Å). The diffractometer was operated at 40 kV and 40 mA. The data were collected in the 5–50° range (2\( \theta \)) at a step size of 0.02°.

3 Results

3.1 Spectroscopic characterization

Figure 1a shows the IR spectra of soybean straw without decrystallization (Figure 1(a(b)) and AA-g-PSBS (acrylic acid-graft-soybean straw of decrystallization, Figure 1(a(a)). Soybean straw is primarily composed of cellulose, and the absorption bands at 3,444 and 613 cm\(^{-1}\) are attributed to \(-\text{OH}\) stretching. The bands at 1,644 and 1,455 cm\(^{-1}\) are due to skeletal C–C stretching in the aromatic rings, and the band at 1,046 cm\(^{-1}\) is due to C–O stretching. The spectra of AA-g-PSBS show relatively intense bands at 1,720, 1,654, 1,387, and 772 cm\(^{-1}\), which are due to \(-\text{COOH}\) stretching, asymmetric, and symmetric stretching of \(-\text{COO}\)– groups, the planar rocking vibration of C–H in the long carbon chain, and the C=O out-of-plane bending vibration from the grafted polyacrylic acid, respectively. The absorption peaks of SBS and AA-g-PSBS at 1,462 and 1,164 cm\(^{-1}\) due to \(-\text{OH}\) stretching are weaker and narrower than those from soybean straw of decrystallization, which clearly suggest the soybean straw of decrystallization participation in the graft copolymerization reaction with AA. The AA-g-PSBS bands are strengthened compared to those of soybean straw predominantly due to decrystallization. Overall, it appears that the decrystallization of soybean straw leads to the formation of PSBS, the

![Figure 1: (a) Infrared spectra of AA-g-PSBS and SBS. (b) XRD of AA-g-PSBS and SBS. (c) TGA of AA-g-PSBS and SBS.](attachment:image.png)
grafting extends to the interior, and in turn increases the graft efficiency.

3.2 X-ray analysis

Figure 1b shows the X-ray diffraction (XRD) patterns of soy bean straw (Figure 1b(a)) and AA-g-PSBS (Figure 1b(b)). The soy bean straw displays a narrow peak at around 22° of 2θ and matches that of the natural cellulose diffraction pattern. The AA-g-PSBS did not possess any measurable peaks suggesting that the initial SBS crystallinity rapidly decreases upon the treatment [26]. It is an established fact that the cellulose network is formed through a host of hydrogen bonding interactions but such a tight crystalline structure transforms to an amorphous state due to the NaOH/urea treatment. This amorphous structure also prevails in AA-g-PSBS.

3.3 Thermogravimetric (TG) analysis

As shown in Figure 1c, the TG patterns of SBS and AA-g-PSBS appear to be the same. However, temperature-induced seminal features are more pronounced in the first-order differential curves [27,28]. The weight loss of SBS follows a three-step behavior as the temperature increases from 25 to 600°C. The 7% initial weight loss in the range 25–180°C is merely due to the loss of absorbed and bound water. The sample decomposition occurs during 180–310°C with an accompanying 50% weight loss. The breakage of C–O–C glycosidic bonds in the cellulose chain might be occurring during this stage. Finally, above 310°C, loss of volatile compounds at a very slow rate takes place leading to complete decomposition.

The melting behavior of AA-g-PSBS is significantly different from that of SBS. The first decomposition stage occurs from 25 to 190°C, mainly due to evaporation of the absorbed moisture (approximately 5% weight loss). The second stage occurs from 190 to 357°C, wherein a 35% weight loss is observed predominantly due to thermal decomposition. The third stage involving the decomposition of carboxy groups of polyacrylic acid takes place from 357 to 464°C, resulting in about 45% weight loss. Above 464°C, the fourth stage with an 8% weight loss could be attributed to the depletion of volatile compounds. Overall, AA-g-PSBS exhibits a relatively higher thermal stability compared to SBS.

3.4 Morphological features

The micrographs of SBS (Figure 2a) and AA-g-PSBS (Figure 2b) are depicted in Figure 2. The crop straw has an impacted multilayered structure; however, SBS displays a tight, smooth surface. After the NaOH/urea decrystallization treatment, the morphology becomes loose and coarse with an undulant surface structure [29,30]. This contributes to the penetration of reagents into the molecular chain, which in turn accelerates the chemical modification. After polymerization, more porous structures appear on the surface and will indeed aid in absorbing more liquid material.

Figure 2: SEM, (a) PSBS, and (b) PSBS-g-AA.
3.5 The effect of the ratio of AA/PSBS to water absorbency of AA-g-PSBS

As shown in Figure 3a, the water absorbency increases as the ratio of AA/PSBS increases. A maximum absorbance of 1,378 g/g in distilled water (quality of the absorbed distilled water, $Q_d$) and 52 g/g in the saline solution (quality of the absorbed saline water, $Q_s$) is achieved for AA/PSBS of 6:1. In general, with increasing AA, more branching and self-crosslinking occurs and results in more hydrophilic groups (e.g. $-\text{COO}^-$) grafted onto the SBS, predominantly the cellulose chains. The subsequently increased presence of cations in the polymeric network leads to a surge in the osmotic pressure between the interior of AA-g-PSBS and the external solution [25]. Such a scenario improves the absorption capacity of AA-g-PSBS. On the other hand, for the increased weight ratio of AA/PSBS, above 6:1, a decrease in the water absorbency has been noticed, which could be due to the formation of a closer polymeric network along with dissoluble homopolymer of polyacrylate than the graft copolymerization, which is not conducive to absorbance [12].

3.6 Effects of radiation dose rate on the water absorbency of AA-g-PSBS

As shown in Figure 3b, the water absorption of both $Q_d$ and $Q_s$ of AA-g-PSBS increase initially with increasing absorbed dose rate, up to 1.5 kGy/h, and then decreases with further increases in the dose rate. The irradiation dose rate is an important factor for radiation-induced graft copolymerization. The dose rate may determine the length of the grafted branches, and an increase in the dose rate enhances the formation of radicals in the reaction mixture of the monomer, PSBS, and water. All molecules are activated to induce a higher conversion for both homopolymer formation and the grafting reaction. If the reaction occurs at a high dose rate, the radical density is high. This causes greater recombination of monomer radicals to form homopolymers, fewer monomer radicals to initiate graft copolymerization, and maybe cause explosive boiling. If the dose rate is too high, however, the water absorption of $Q_d$ and $Q_s$ is decreased. The optimal dose ratio was therefore determined as 1.5 kGy/h.

Figure 3: Single-factor experiment for water absorption. (a) Rate of AA to PSBS, (b) irradiation dose rate, (c) irradiation dose, and (d) neutralization of AA.
3.7 Effects of radiation dose on the water absorbency of AA-g-PSBS

As shown in Figure 3c, the water absorbency increases with the absorbed radiation dose up to 4.5 kGy. This could be due to the enhanced formation of radicals on the cellulose backbone leading to a high degree of cross-linking. A subsequent increase in the dose reduces the absorbency as the addition of more radicals reinforces the termination step via bimolecular collision, which certainly results in the reduced chain length leading to crosslinking density and compact network structure [8]. In addition, higher irradiation might as well increase the amount of radiolysis products, i.e., OH and $e_{aq}$, which facilitate homopolymerization than the grafting reaction [31].

3.8 Effects of the neutralization degree of AA on the water absorbency of AA-g-PSBS

As shown in Figure 3D, the water absorbency is influenced by electrostatic interactions between functional groups and the ionic osmotic pressure. As the neutralization degree of AA increases, the hydrophilicity of the hydrophilic groups of the composite increases. In addition, the osmotic pressure increases between the interior and exterior networks when the SAP is immersed in an aqueous solution, which is really conducive to expanding the network. It manifests in maintaining a balance between the interior and exterior of the system. However, when the ratio of NaOH/PSBS is above 3:10, excess Na$^+$ ions shield the −COO$^-$ groups resulting in increased ionic strength, which concomitantly reduces the swelling ratio and water absorbency [8,11,32].

3.9 Optimization of the reaction parameters

The experiment was designed according to four factors and three-level orthogonal table, as shown in Table 1. (A) The ratio of acrylic acid volume/soybean straw mass, abbreviated to $V_{AA\text{-mSD}}$ (mL/g). (B) The neutralization degree of AA. (C) The quantity of radiation, abbreviated to dose (kGy). (D) Radiation dose rate, abbreviated to dose rate (kGy/h). For $A_2B_1C_2D_3$, however, the absorbency is 46 g/g in the 0.9 wt% NaCl solution. In contrast, the maximum absorbency of the NaCl aqueous solution is obtained for the $A_2B_2C_2D_2$ experimental combination, wherein the absorbency of the distilled water is found to be 1,396 g/g. The effect of the distilled water absorbency can be arranged in the order of $A > C > D > B$ (Table 1), and the absorbency of a 0.9 wt% NaCl aqueous solution can be arranged in the order $C > A > D > B$ (Table 1). Therefore, the optimum combination was set as $A_2B_2C_2D_3$.

Table 1: Orthogonal experiment results

| A: $V_{AA\text{-mSD}}$ (mL/g) | B: neutralization degree of AA | C: dose (kGy) | D: dose rate (kGy/h) | $Q_d$ in distilled water (g/g) | $Q$ in saline solution (g/g) |
|-----------------------------|-------------------------------|---------------|----------------------|-------------------------------|-------------------------------|
| 1 1(7:1)                    | 1(80)                         | 1(3.0)        | 1(1.0)               | 756 ± 11                      | 34 ± 3                       |
| 2 1                         | 2(70)                         | 2(4.5)        | 2(1.5)               | 809 ± 8                       | 45 ± 4                       |
| 3 1                         | 3(60)                         | 3(6.0)        | 3(2)                 | 977 ± 10                      | 48 ± 3                       |
| 4 2(9:1)                    | 1                             | 2             | 3                    | 1,498 ± 6                     | 59 ± 2                       |
| 5 2                         | 2                             | 3             | 1                    | 1,278 ± 12                    | 42 ± 4                       |
| 6 2                         | 3                             | 1             | 2                    | 1,086 ± 11                    | 41 ± 5                       |
| 7 3(11:1)                   | 1                             | 3             | 2                    | 1,015 ± 10                    | 37 ± 2                       |
| 8 3                         | 2                             | 1             | 3                    | 833 ± 6                       | 32 ± 3                       |
| 9 3                         | 3                             | 2             | 1                    | 1,156 ± 8                     | 41 ± 2                       |

$k_1^a = (\Sigma$ water absorbency in distilled water of single factor)/3, $k_2^b = \max k_2 - \min k_1$, $k_3^c = (\Sigma$ water absorbency in saline solution of single-factor)/3, $k_4^d = \max k_4 - \min k_2$. $Q = \max k_4 - \min k_2$. $Q = \text{the optimum conditions}$.
Using these conditions, AA-g-PSBS was prepared without heating or nitrogen protection. The absorbency of distilled water reaches 1,489 g/g, and that of the NaCl aqueous solution to 56 g/g.

### 3.10 Water-retaining property

Good water retention is one of the excellent properties of superabsorbent resin. The structure has a certain strength in the swelling state and can retain a large amount of water, even under certain external pressures. The water in the network is also not easy to lose. This property endows good properties to water required for superabsorbent resins. As shown in Figure 4 shows, with time, the moisture content maintained by the superabsorbent resin decreases continuously. As can be seen from the figure, AA-g-PSBS has a high moisture retention ability and can still retain 71% of water at 12 h. The water retention rate is still about 50% at 24 h. The cellulose intact crystalline region of acrylic acid-graft-soy bean straw without decrystallization (AA-g-SBS) might not have been damaged, and thus, monomer AA molecules might not be able to enter the cellulose structure and thus the resulting grafting would not be uniform. However, during prolonged time intervals, the external resin surface forms a layer of low-density shell resulting in water migration [13].

### 4 Discussion and conclusions

The commonly used materials for SAP are starch [6,18], cellulose [12,25,33,34], and chitosan [3,27]. Among them, cellulose is most studied, which is present in wood, plants, marine animals, algae, and microorganisms. It is composed of β-D-glucose units linked through 1,4 glycosidic bonds. Each unit has three hydroxyl groups and makes cellulose hydrophilic [24]. Its sheet-like molecular arrangement results in a robust hydrogen-bonding network arrangement, which in turn results in a highly crystalline three-dimensional network structure [35,36]. Consequently, cellulose is water insoluble and individual sugar units are not accessible for the acrylic monomers during grafting. Despite these difficulties, acrylic monomers such as acrylic acid [18] and acrylamide monomers could be grafted onto cellulose leading to a superabsorbent composite [37,38].

The NaOH/urea mixture could yield a homogeneous SBS solution. The static solution polymerization method adapted in our research indeed yields porous and superabsorbent AA-g-PSBS by 60Co irradiation of thus prepared SBS solution without any nitrogen protection and heating. The preparation of superabsorbent polymers using PSBS as a raw material helps to maintain the physical and chemical properties of a superabsorbent resin. The use of irradiation also aids to overcome the problem of chemical initiation and thus is conducive to reducing environmental pollution. We strongly believe that the outcome opens up new pathways in synthesizing environmentally safe superabsorbents with widespread food, pharmaceutical, and medical applications.

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