



# Synthesis of a bioadsorbent from jute cellulose, and application for aqueous Cd (II) removal

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## ARTICLE INFO

### Chemical compounds studied in this article:

Jute fibers pretreated by continuous screw-extrusion steam explosion (JSE)  
JSE-g-polyacrylamide (JAM)  
Hydrolyzed JAM under alkaline conditions (J-g-P(AM-co-AANa))  
Acrylamide (AM)  
Potassium persulfate (KPS)  
Sodium hydroxide (NaOH)  
Cadmium nitrate tetrahydrate  
Dilute hydrochloric acid (HCl)

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## ABSTRACT

A low-cost, high-adsorption-capacity, eco-friendly bioadsorbent for removing Cd<sup>2+</sup> from aqueous solution is reported. J-g-P(AM-co-AANa) was prepared by hydrolysis of the grafted copolymer, which was synthesized by free radical polymerization of acrylamide (AM) with jute fibers (JSE) pretreated by continuous screw-extrusion steam explosion. Fourier transform infrared and solid-state <sup>13</sup>C nuclear magnetic resonance spectroscopies, confirmed that amino and carboxylate groups were introduced into J-g-P(AM-co-AANa). X-ray diffraction showed that the crystallinity of J-g-P(AM-co-AANa) was significantly lower than that of JSE. The surface morphology of bioadsorbent was investigated by scanning electron microscopy (SEM). The adsorption capacity of Cd<sup>2+</sup> on J-g-P(AM-co-AANa) was evaluated for different solution pH values, contact times, and initial Cd<sup>2+</sup> concentrations. The adsorption kinetics followed the pseudo-second-order kinetic model, and the rate controlling step was chemisorption. The adsorption isotherm was well fitted by the Freundlich model, and the adsorption process was multilayer adsorption. The maximum adsorption capacity was 344.8 mg/g, which indicated that the bioadsorbent was effective for removing Cd<sup>2+</sup> from aqueous solution.

## 1. Introduction

Heavy metals in water are mainly derived from mining, industrial discharges, and particulates in the atmosphere. Cadmium is a common heavy metal, and long-term exposure is linked to bone and kidney damage (David, Colin, & Thomas, 2008; Nayan et al., 2017; Rahmi & Julinawati 2017). Conventional methods for removing heavy metal ions from aqueous solution include reduction, precipitation, ion exchange, filtration, electrochemical treatment, membrane technology, and coagulation (Deng et al., 2017; Liu et al., 2017; Özcan et al., 2005; Sanna, Eveliina, & Mika, 2013). These methods are subject to various problems, such as high chemical reagent consumptions and time requirements, low cost-effectiveness and efficiencies, and secondary pollution. In contrast, adsorption is a simple, inexpensive, environmentally friendly, effective, and practical method for removing heavy metal ions (Abdusalam et al., 2017; He et al., 2017; Łukasz et al., 2017; Preeti &

Anjali, 2017; Zhao et al., 2013). Activated carbon has been the most widely applied adsorbent, but its application is restricted by high operating costs and difficulties with regeneration.

Cellulose is the most abundant and renewable natural polymer. Cellulose-based adsorbents have attracted much attention for removing heavy metal ions from aqueous solution owing to their low cost, good performance, and low secondary pollution (Liu, Xie et al., 2016; Noriyuki et al., 2013; Sapana & Ghanshyam, 2014; Sanna, Eveliina, Lena et al., 2014; Sanna, Eveliina, Terhi et al., 2014; Sun et al., 2014; Tamutsiwa & Chieu, 2014; Wafa & Sami, 2015; Zhu et al., 2016). The preparation of cellulose-based adsorbents has two major issues, as follows. 1) Cellulose is a semicrystalline polymer and is insoluble in water and common solvents owing to strong hydrogen bonding between cellulose chains. To increase the accessibility of the reaction, raw cellulose must be pretreated with large amounts of acid and alkali before preparing cellulose-based adsorbents, which is not environmentally

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friendly. 2) The preparation of cellulose-based adsorbents requires many organic solvents, which is costly, and presents post-processing difficulties and environmental concerns. To maximize the removal of heavy metals from aqueous solution, the metal should strongly bind on the adsorbent surface. In summary, some form of pretreatment is often necessary for lignocellulosic materials.

Continuous screw-extrusion steam explosion (CSESE) pretreatment is a continuous high-efficiency pretreatment that destroys the cellulose fiber structure, dramatically decreases the fiber size and thus increases the specific surface area, and partially strips off the middle lamella which contains a high lignin content (Liang et al., 2016; Ma et al., 2016). CSESE pretreatment is an effective way to render the hydroxyl groups of cellulose macromolecules more accessible for the introduction of reaction reagents. Functional groups such as  $-\text{COO}^-$ ,  $-\text{NH}_2$ , and  $-\text{OH}$  in the polymer reportedly play an important role in the adsorption of heavy metal ions on cellulose-based adsorbents and also affect the adsorption capacity (Liu, Li et al., 2016). For example, acrylamide (AM) can be graft copolymerized with cellulose and then subsequently hydrolyzed in aqueous sodium hydroxide. The resulting bioadsorbent J-g-P(AM-co-AA<sub>Na</sub>) has  $-\text{COO}^-$ ,  $-\text{NH}_2$ , and  $-\text{OH}$  functional groups and exhibits a good adsorption capacity for heavy metal ions.

To the best of our knowledge, the use of modified jute fibers to adsorb heavy metal ions from water has not been reported. There are also no reports on the synthesis of modified jute fibers with AM and then subsequently hydrolyzed with aqueous sodium hydroxide. The grafting of jute fibers with AM has two advantages: 1) The introduction of a large number of amide groups on the modified jute fibers allows the adsorption of cadmium ions through coordination, thereby increasing the adsorption capacity; 2) the amide groups can be partly hydrolyzed to  $\text{COO}^-$  in alkaline conditions (NaOH), which can then form electrostatic adsorption with cadmium ions, thereby increasing the adsorption capacity. NaOH was used because it is cheap and easy to obtain. Methods that have been reported for the pretreatment of jute fibers include alkali treatment (Dheeraj, Anupama, & Mandeep, 2017; Donghwan et al., 2011; Lin et al., 2014; Shanmugam & Thiruchitrambalam, 2013; Umit et al., 2016), bleach treatment (Haydar, Ruhul, & Mubarak, 2012), and enzymatic treatment (Ali & Mohammad, 2014; Avtar et al., 2016). However, there is no report of the CSESE pretreatment of jute fibers.

In the current study, jute fibers were pretreated by CSESE, chemically modified with AM, and then hydrolyzed with aqueous sodium hydroxide. This introduced carboxylate ( $-\text{COO}^-$ ) and amino ( $-\text{NH}_2$ ) groups into the pretreated jute fibers (JSE). The morphology, structure, and crystallization of JSE before and after modification were studied by Fourier transform infrared (FTIR) and solid-state  $^{13}\text{C}$  nuclear magnetic resonance ( $^{13}\text{C}$ NMR) spectroscopies, and X-ray diffraction (XRD). The adsorption capacity of  $\text{Cd}^{2+}$  by modified JSE (J-g-P(AM-co-AA<sub>Na</sub>)) was evaluated for different solution pH values, contact times, and initial  $\text{Cd}^{2+}$  concentrations. Different kinetic and isotherm models were evaluated to examine the mechanism of adsorption.

## 2. Materials and methods

### 2.1. Materials

Jute fibers were obtained from Nanjing Xinhe Textile Co., Ltd. Prior to use, jute fibers were pretreated using a laboratory-designed CSESE apparatus. The purpose of the pretreatment is to reduce the diameter of the jute fibers, increase the specific surface area, and increase the accessibility of the reaction. The pretreated jute fibers are referred to as JSE. Analytical grade AM, potassium persulfate (KPS), sodium hydroxide (NaOH), and cadmium nitrate tetrahydrate were purchased from Tianjin Kermel Chemical Reagent Co., Ltd. and Nanjing Chemical Reagent Co., Ltd. All chemicals were used as received without further purification.

### 2.2. Experimental procedure

#### 2.2.1. Synthesis of JSE-g-polyacrylamide (JAM)

We have previously found it difficult to graft copolymerize jute fibers with AM, so jute fibers were pretreated by CSESE prior to graft copolymerization. The synthesis of JAM was carried out according to Ye and Yang (2015). An appropriate amount of JSE was immersed in 100 mL of deionized water for 24 h. The swelled JSE was then transferred to a 250 mL three-necked flask equipped with a magnetic stirrer and reflux condenser. The reaction system was charged with nitrogen for 15 min. Then, KPS as an initiator (0.01–0.035 mol/L) was added to the three-necked flask. After 10 min, a certain amount of AM was slowly added to the reaction system, and the mixture was stirred in a water bath for the desired time (2–12 h) at the desired temperature (27–75 °C) to complete the polymerization. The reaction mixture was then cooled in an ice-water bath for 30 min. The obtained products were then washed with deionized water to remove remaining monomer, initiator, and resulting homopolymers, and finally dried at 60 °C in an electric oven. The product is referred to JSE-g-polyacrylamide (JAM). Dried products were ground into powder before further application.

The grafting percentage (GP) and grafting efficiency (GE) (Mangesh & Javed, 2012) were calculated by the following equations:

$$\text{GP} = (W_2 - W_0)/W_0 \times 100\% \quad (1)$$

$$\text{GE} = (W_2 - W_0)/W_1 \times 100\% \quad (2)$$

where  $W_0$ ,  $W_1$ , and  $W_2$  are the weights of JSE, monomer (AM), and JAM after the homopolymers were removed, respectively.

#### 2.2.2. Hydrolysis of JAM

Hydrolysis of JAM was carried out according to Liu, Xie et al. (2016) with a simple modification. Specifically, dried JAM powder was added into 500 mL of 1 M or 2 M NaOH aqueous solution, which was then stirred with a magnetic stirrer at 50 °C for 12 h. The sample was then dialyzed in flowing tap water and then dried at 60 °C in an electric oven. The resulting sample is referred to as J-g-P(AM-co-AA<sub>Na</sub>). The dried J-g-P(AM-co-AA<sub>Na</sub>) was ground into powder, for the adsorption of  $\text{Cd}^{2+}$  in aqueous solution. The synthesis and hydrolysis of JAM are shown in Scheme 1. As shown in Scheme 1, compared with JAM, new carboxylate groups were introduced in the molecular structure of JAM after it was hydrolyzed. The degree of hydrolysis of JAM in alkaline solution was based on its nitrogen content, which was determined by elemental analysis.

### 2.3. Characterization

#### 2.3.1. FTIR spectroscopy

JSE, JAM, and J-g-P(AM-co-AA<sub>Na</sub>) were dried in an electro thermal oven at 60 °C. The samples were then ground with dried potassium bromide in an agate mortar. The resulting powder was pressed into discs which were then dried in an infrared box. FTIR spectra were collected from the dry discs samples using a Nicolet Nexus 670 infrared spectrophotometer.

#### 2.3.2. Solid-state $^{13}\text{C}$ NMR spectroscopy

JSE, JAM, and J-g-P(AM-co-AA<sub>Na</sub>) were dried at 60 °C, and ground into powder. Solid-state  $^{13}\text{C}$  NMR spectra were recorded using a Bruker NMR spectrometer (NMR, Avance III 400 M, Bruker Co., Germany) at 400 MHz.  $^{13}\text{C}$  NMR spectra were referenced against tetramethyl silane.

#### 2.3.3. Xrd

XRD patterns of JSE, JAM, and J-g-P(AM-co-AA<sub>Na</sub>) were recorded using a Bruker D8 Advance (Bruker, Germany) diffractometer (40 kV, 40 mA), by the refraction method using nickel-filtered  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.54 \text{ \AA}$ ). Scans were performed from 4 to 60° in increments of 0.04°, at a scanning rate of 0.2 s per step. Prior to testing, samples were

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