

Wheat straw cellulose dissolution and isolation by *tetra-n*-butylammonium hydroxide

Chao Zhong, Chunming Wang, Fan Huang, Honghua Jia*, Ping Wei

College of Biotechnology and Pharmaceutical Engineering, Nanjing University of Technology, Nanjing 211316, PR China

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ABSTRACT

In this article, a novel and high efficient solvent, *tetra-n*-Butylammonium Hydroxide (TBAH), was used for dissolution and isolation of straw cellulose from wheat straw. The composition analysis with gas chromatography (GC) and the spectroscopic characterization analysis conducted by X-Ray diffraction (XRD)/Fourier transform infrared spectroscopy (FTIR) indicated that straw cellulose can be directly dissolved and isolated by TBAH without derivatization. The investigation on the properties of straw cellulose isolation was based on the results of single factor experiments and orthogonal experiments, with optimum conditions for straw cellulose isolation being obtained as follow: reaction temperature 60 °C, reaction time 40 min, concentration of TBAH 50% and ratio of TBAH to straw 10:1 (m/m). In addition, as a solvent for cellulose, TBAH could be recycled several times with high activity being retained.

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1. Introduction

Cellulose is one of the most abundant natural polymers (Pinkert, Marsh, Pang, & Staiger, 2009). Cellulose and its derivatives are widely used in the chemical, pharmaceutical, papermaking industry and other areas (Klemm, Heublein, Fink, & Bohn, 2005). Partly because of the increased awareness of environment problems (Mohanty, Misra, & Drzal, 2002), recently there has been an increasing interest in more efficient utilization of agro-industrial residues for the production of various biodegradable and environment friendly cellulose materials (Pandey, Soccol, Nigam, & Soccol, 2000).

Agro-industrial residues primarily consist of cellulose, hemicelluloses and lignin (Zhang, Du, Qian, & Chen, 2010). In order to make full use of the residues, these major components need to be isolated. The cellulose isolated may be used as a raw material for papermaking, fuel alcohol and other industrial applications. With present technologies, efficient methods for isolating cellulose from agro-industrial residues are based on the dissolution of cellulose.

However, it's extremely difficult to dissolve cellulose in most traditional solvents because of the numerous intermolecular and intramolecular hydrogen bonds existing in cellulose (Lin, Zhan, Liu, Fu, & Lucia, 2009). Recently, there have been extensive

efforts to find effective solvents for cellulose, with several solvents being reported, such as N-methylmorpholine N-oxide monohydrate (NMMO) (Roder & Morgenstern, 1999), LiCl/N, N-dimethylacetamide (DMAC) (McCormick, Callais, & Hutchinson, 1985), some molten salt hydrates like LiClO₄·3H₂O (Fischer, Leipner, Thummler, Brendler, & Peters, 2003) and LiSCN·2H₂O (Nehls, Lukanoff, Philipp, & Zschunke, 1983), and some aqueous solution of metal complex like cuprammonium hydroxide (Saalwachter et al., 2000). However, there remain some disadvantages including toxicity, thermal instability, hazardous vapors and high cost (Liu et al., 2007; Zhang, Wu, Zhang, & He, 2005). The other solvents reported recently are neither toxic nor too expensive, among which concentrated phosphoric acid (H₃PO₄) plays the major role (Conte, Maccotta, De Pasquale, Bubici, & Alonzo, 2009; Zhang, Cui, Lynd, & Kuang, 2006). However, cellulose dissolution with phosphoric acid usually results in the degradation of cellulose in lower temperature (Boerstoel, Maatman, Picken, Remmers, & Westerink, 2001; Butera, De Pasquale, Maccotta, Alonzo, & Conte, 2011; Saeman, 1945), which is not appropriate for regenerating cellulose.

Recently, ionic liquids (ILs), such as 1-ethyl-3-methylimidazolium acetate (EMIMAc), 1-butyl-3-methylimidazolium chloride (BMIMCl), 1-allyl-3-methylimidazolium chloride (AMIMCl), have been used as solvents for cellulose (Remsing, Swatloski, Rogers, & Moyna, 2006; Song, Zhang, Niu, & Wang, 2010; Zhu et al., 2006), but there are still some limitations for the application of ionic

* Corresponding author. Tel.: +86 25 58139368; fax: +86 25 58139368.

E-mail address: hhjia@njut.edu.cn (H. Jia).

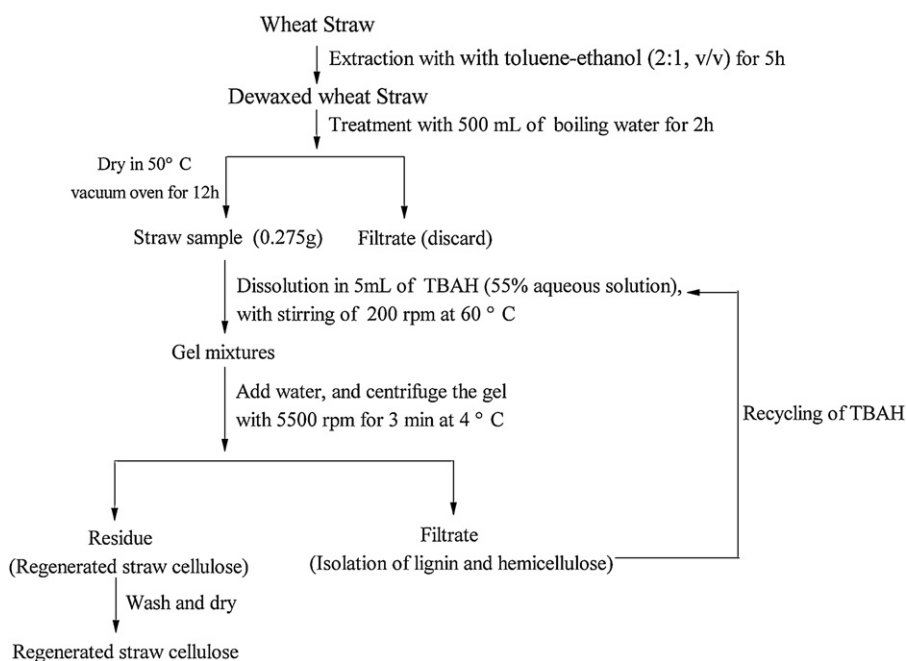


Fig. 1. Scheme for isolation of straw cellulose from wheat straw.

liquids, such as the relatively high viscosity, which slows down the dissolution of cellulose. Besides, the solubility of cellulose may decrease dramatically if a certain amount of water is added (Mazza, Catana, Vaca-Garcia, & Cecutti, 2009). Consequently, it's difficult to use polar ionic liquid as solvent to treat agro-industrial residues without pre-treatment. Furthermore, degradation of cellulose will also occur during the dissolution in ILs (Zhang et al., 2010), which is not appropriate for cellulose regeneration.

To overcome these disadvantages, hydroxide anion is considered as an ideal substitution (Cai, Liu, & Zhang, 2006; Liu & Zhang, 2009; Zhang et al., 2001). As the solvent for cellulose, sodium hydroxides (NaOH) aqueous solution with or without urea/thiourea added have already been reported (Cai & Zhang, 2006; Isogai & Atalla, 1998; Zhang, Ruan, & Gao, 2002). However, the applications of sodium hydroxide are strictly controlled for the conditions required to dissolve cellulose, which prevents it to be an ideal solvent for cellulose (Egal, Budtova, & Navard, 2007). In this article, we chose *tetra-n*-Butylammonium Hydroxide (TBAH) as the solvent for cellulose and isolated cellulose from wheat straw by dissolving it in TBAH. The feasibility of isolating straw cellulose from wheat straw was investigated. In addition, the recyclability of TBAH was also discussed. All the works in this article indicated that TBAH a useful solvent for dissolving and isolating cellulose from agro-industrial residues.

2. Materials and methods

2.1. Materials

Agricultural wheat straw, collected from Siyang, Jiangsu Province, was supplied by the biogas pilot plant of Nanjing University of Technology. It was dried in sunlight and then cut into small pieces with length of 1 cm. *Tetra-n*-Butylammonium Hydroxide (55% aqueous solution), acetic anhydride and potassium bromide were purchased from the Alfa Company. Microcrystalline cellulose, diethyltriamine pentaacetic acid, anthrone, sulfuric acid, hydrogen peroxide (30% aqueous solution) and sodium hydroxide were provided by the Aladdin Reagent Company.

2.2. Dissolution of microcrystalline cellulose in TBAH

The aqueous solution containing 55% TBAH by weight was used as the solvent for microcrystalline cellulose. Measured 5 mL of solvent into triangular flask, and then 0.55 g of microcrystalline cellulose was immediately added and mixed with the solvent. After stirring for 2 min, the microcrystalline cellulose could be completely dissolved in solvent at room temperature, with the resultant solution turning into a transparent gel. After that, 50 mL of water was added into the gel mixtures, with some white substances being precipitated. The white precipitates were obtained by centrifuging the gel system at a speed of 5500 rpm for 3 min at 4°C, followed by being filtered, washed with 40% ethanol/water, and dried in a vacuum oven for 12 h.

2.3. Isolation of wheat straw cellulose

Based on the results illustrated above, we chose TBAH (55% aqueous solution) as the solvent for isolating cellulose from wheat straw, with a scheme for isolation of cellulose from wheat straw being described below and shown in Fig. 1.

The wheat straw was first dewaxed in Soxhlet apparatus with toluene-ethanol (2:1, v/v) for 5 h (Sun, Lawther, & Banks, 1995), followed by being immersed in 500 mL of boiling water for 2 h, and then dried at 50°C in vacuum oven for 12 h. The dried wheat straw (0.275 g) was added into triangular flask and mixed with 5 mL of solvent completely. The mixtures were heated at 60°C and stirred with speed of 200 rpm until the straw samples completely dissolved (actually 15–20 min was enough), and converted into mixture of gel particles. After the gel was cooled to room temperature, 50 mL of water was added with stirring to stop the reaction, with some substances precipitating. The gel mixtures were then centrifuged at a speed of 5500 rpm to obtaining the precipitates (regenerated straw cellulose), which was filtered afterwards, washed with 40% ethanol/water, and dried at 50°C in a vacuum oven for 12 h.

2.4. Chemical identification of regenerated straw cellulose

Cellulose content of regenerated straw cellulose was determined by using the anthrone method (Frederick & Leslie, 1949)

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