



## Preparation of lignocellulose aerogels from cotton stalks in the ionic liquid-based co-solvent system

Hassan Mussana<sup>a,b</sup>, Xue Yang<sup>a,b</sup>, Mengistu Tessima<sup>a,b</sup>, Fuyi Han<sup>a,b</sup>, Nousheen Iqbal<sup>c</sup>, Lifang Liu<sup>a,b,\*</sup>

<sup>a</sup> College of Textiles, Donghua University, Shanghai, 201620, PR China

<sup>b</sup> Key Laboratory of Textile Science and Technology, Ministry of Education, College of Textiles, Donghua University, Shanghai, 201620, China

<sup>c</sup> State Key Laboratory for Modification of Chemical Fibers and Polymer Material, College of Materials Science and Engineering, Donghua University, Shanghai, 201620, China

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### ABSTRACT

Ionic liquid-based lignocellulose aerogel systems have attracted significant focus in advanced material research due to their intrinsic environment-friendly properties. Use of co-solvent is deemed useful in improving these systems efficiency without affecting reaction dynamics. Herein, a green synthesis strategy is reported to produce highly porous, physically cross-linked lignocellulose aerogel by dissolving lignocellulose biomass obtained from the cotton stalk in 1-allyl-3-methylimidazolium chloride/dimethyl sulfoxide (AmimCl/DMSO) co-solvent system. Samples with different concentrations were treated under both cyclic conventional freezing-thawing conditions (−20 °C to 20 °C) and liquid nitrogen freezing-thawing conditions (−196 °C to 20 °C) followed by solvent exchange and freeze drying. Scanning electron microscopy (SEM) and Brunauer-Emmett-Teller (BET) analysis of these lignocellulose samples showed that conventional freezing-thawing treated samples exhibit hierarchical 3D open porous “web-like” structure with a low specific surface area and larger dominant pore diameter. On the other hand, use of liquid nitrogen freezing-thawing treatment resulted in the creation of “film-like” porous structure with a higher specific surface area and relatively smaller dominant pore diameter. Quantitative analysis through Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and thermo-gravimetric analysis (TGA) verified that cyclic freezing-thawing treatment has potential to fix all major components of lignocellulose in a matrix. The proposed strategy is not only environmentally benign but also the cost-effective making an ionic liquid-based system more commercially viable.

### 1. Introduction

The hazardous emission of carbon dioxide along with diminishing petroleum resources has compelled researchers to explore more eco-friendly alternatives for the protection of this planet (Ragauskas et al., 2006). Lignocelluloses are natural biopolymers that have immensely attracted the attention of many researchers as a green alternative. These biopolymers offer great advantages for instance renewability, biocompatibility and the low cost, which make them potential raw materials for functionalized applications like biofuel (Huber et al., 2006), biomedical materials (Cai, Sharma et al., 2014), and food packing materials (Innerlohinger et al., 2006). Lignocelluloses are existent mainly in the form of polysaccharides holocellulose and certain biopolymers such as lignin.

Most abundant lignocellulose polymer is cellulose which is a polydisperse  $\beta$  (1–4) linked D glucose repeating units bonded together with

hydrogen bonds to form supra-molecular structures (Finkenstadt and Millane, 1998). Extraction of cellulose has been a challenging task due to their tightly packed molecular structure and strong intramolecular and intermolecular hydrogen bonding. There are quite few solvents that have been employed to dissolve cellulose such as *N*-Methylmorpholine *N*-oxide (NMMO) (Heinze and Liebert, 2001), lithium chloride/*N,N*-dimethylacetamide (LiCl/DMAc) (Nishino et al., 2004), calcium thiocyanate (Sandra et al., 2008), sodium hydroxide (NaOH) solution (Gavillon and Budtova, 2007), alkali hydroxide-urea solution (Cai, Kimura et al., 2008), lithium chloride/dimethyl sulfoxide (LiCl/DMSO) solution (Wang et al., 2009). All these techniques inherited their own problems such as toxicity, instability, high cost or inability to dissolve high molecular weight cellulose.

Agricultural waste is an important source of lignocellulose biomass. In the past, several attempts have been made to extract cellulose from agricultural waste such as corn husk (Yang, Wang et al., 2016), cotton

\* Corresponding author at: College of Textiles, Donghua University, 2999 Renmin Rd. (North), Songjiang District, Shanghai, 201620, PR China.  
E-mail address: [lifangliu@dhu.edu.cn](mailto:lifangliu@dhu.edu.cn) (L. Liu).

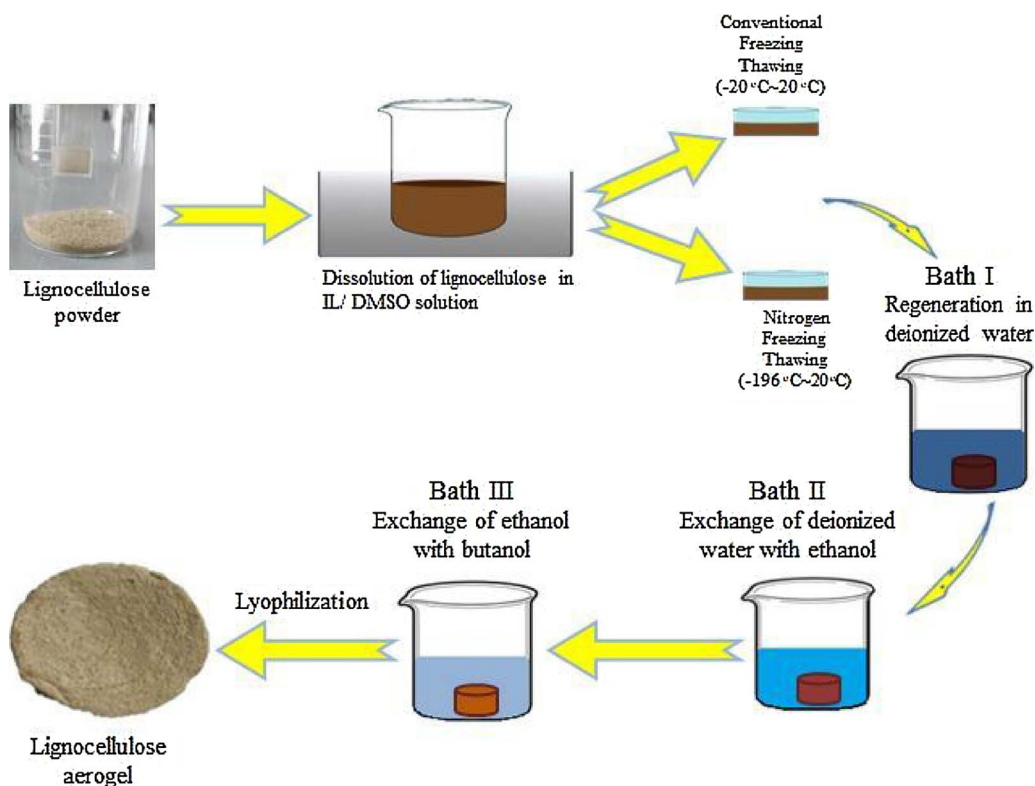


Fig 1. Schematic illustration of the preparation method of lignocellulose aerogel samples.

stalk (Shamskar, Heidari et al., 2016), and rice straw (Reddy and Yang, 2006). For instance, in the cotton plant, lignocellulose biomass content is three to five times more than cotton fibers content (Silanikove and Levanon, 1986). Previously, a major portion of this lignocellulose biomass was discarded for the reason of the non-availability of techniques (Reddy and Yang, 2009). Recently, various studies reported that by using so-called “green solvents” generally known as room temperature ionic liquids (RTIL) could dissolve major portion of the lignocellulose material (Hamada, Yoshida et al., 2013).

Ionic liquids are polar solvent comprised of anions and cations (Inoue et al., 2013). Extensive studies have been carried out to investigate characteristics of cellulose-based ionic liquid (IL) systems (Sescousse et al., 2010). Certain unique properties of these so-called room temperature ionic liquids such as low melting point, good thermal and chemical stability and very low vapor pressure made them highly useful for wide range of applications including dissolution of lignocellulose materials (Kilpeläinen et al., 2007). It has been reported that imidazolium-based ILs, especially ones with chloride anions are excellent solvents for different lignocellulose biopolymers (Vitz et al., 2009).

With all these advantages and potentials of ionic liquid-based lignocellulose systems, there are also some drawbacks that limit their large-scale industrialization. Two major limitations of ionic liquid-based lignocellulose system are the high cost of ionic liquid and high viscosity of the solution (Gardas and Coutinho, 2008). Some previous studies revealed that the choice of co-solvent could significantly affect the outcome of the dissolution process. Use of a co-solvent not only significantly reduce the cost of solvent but also improve the solvation power of ionic liquid by lowering the viscosity of the solution (Xu et al., 2013). Particularly use of aprotic solvents like dimethyl sulfoxide (DMSO) and dimethyl formamide (DMF) are very effective in reducing the viscosity of IL-based solutions (Zhao et al., 2013). While protic solvents like water (above certain concentration) and methanol considerably increase the viscosity of the solution that makes it difficult for ionic liquids to dissolve biopolymers (Parthasarathi et al., 2015). As a result of the addition of dimethyl sulfoxide in 1-butyl-3-

methylimidazolium chloride (BmimCl), the viscosity is decreased. Moreover, it not only improves the solvation power of IL but also significantly decrease the time and the temperature of dissolution of cellulose in ionic liquid (Andanson et al., 2014).

In 1-allyl-3-methylimidazolium chloride/dimethyl sulfoxide (AmimCl/DMSO) based co-solvent system, different concentrations of co-solvents could be used but higher concentrations of 1-allyl-3-methylimidazolium chloride (AmimCl) would make solvent system expensive. The use of very low concentrations of the ionic liquid was not suitable since some previous studies showed that the proper dissolution of 1 anhydrous glucose unit (AGU) required 2.5–3 mol of the ionic liquid (Le et al., 2014).

In the present research work, novel “ionic liquid/dimethyl sulfoxide” based co-solvent system has been used to dissolve cotton stalk for the preparation of ultra-porous lignocellulose aerogels. This research reported the green synthesis strategy of ultra-porous lignocellulose aerogels. Furthermore, for the optimization of the reaction conditions and lowering the cost of co-solvent system, an equal mass concentration of both components of co-solvent system has been chosen. It also highlights the effect of different freezing temperature on morphology and physical properties of lignocellulose aerogels.

## 2. Experimental section

### 2.1. Materials

Cotton stalks obtained from the local forms in Shanghai were used as a raw material. After thorough washing, shaving, drying, and milling cotton stalk biomass into powder, it was subsequently sieved with 60 mesh sieve. It was vacuum dried at 105 °C for 12 h before further use. 1-allyl-3-methylimidazolium chloride (AmimCl) was procured from Shanghai Boyle Chemical Co., Ltd., China. Absolute ethanol and dimethyl sulfoxide (DMSO) were acquired from Shanghai Lingfeng Chemical Reagent Co., Ltd., China. *tert*-butanol was purchased from Tianjin Fuchen Chemical Reagents Factory, China. Deionized water was of laboratory grade. All materials were used without any further

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