



Integrative process for a sugarcane bagasse biorefinery to produce glucose, bio-oil and carbon microspheres

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ARTICLE INFO

Article history:

Received 20 October 2017

Received in revised form 6 December 2017

Accepted 10 January 2018

Keywords:

Solid acid catalyst

Ionic liquid

Sugarcane bagasse

Hydrothermal

Pyrolysis

Bio-oil

Carbon microspheres

ABSTRACT

Selective fractionation of cellulose from sugarcane bagasse (SCB) for the production of fermentable sugars was studied. The SCB was pretreated by hydrothermal pretreatment using five different types of solid acid catalysts (SACs): (1) self-synthesis $\text{SiO}_2\text{-OSO}_3\text{H}$, (2) $\text{SO}_4^{2-}/\text{TiO}_2/\text{Fe}_3\text{O}_4/\text{WO}_3$, (3) sulfonated bentonite and commercial SAC, (4) Amberlyst[®] 15, and (5) Dowex[®] 50WX8. Fractionation of SCB was conducted in a combination with choline acetate (ChOAc) ionic liquid (IL) at moderate temperature. After enzyme hydrolysis, the highest total reducing sugar (TRS) yield of 78.1% was achieved when pretreated with $\text{SiO}_2\text{-OSO}_3\text{H}$. For the black liquor from fractionation, the highest amount of lignin could be precipitated from ChOAc IL through treatment with $\text{SO}_4^{2-}/\text{TiO}_2/\text{Fe}_3\text{O}_4/\text{WO}_3$. Pyrolysis of dry precipitated lignin was performed at 400 and 500 °C under a nitrogen atmosphere, and phenolic-rich oil was produced. Carbon microspheres appeared in the solid phase from the pyrolysis. It was postulated that lignin was first degraded to phenol with an aromatic structure, while other sugar residues (C5 and C6) in the lignin molecule formed ring compounds. These compounds underwent condensation polymerization as a shell-like shape, and carbon rich microspheres were thus formed.

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1. Introduction

Sugarcane bagasse (SCB), a solid residue from the sugar industry, is one of the highest potential lignocellulosic residues from agro-industry in Thailand. SCB is a renewable resource with high feasibility for use as a precursor for biofuels and bio-based materials and chemicals. In the biotechnological conversion of lignocellulosic biomass to sugars, the pretreatment and the hydrolysis steps are the bottleneck and the energy intensive process that result in high operating costs. Recently, many researchers have developed green processing for effective and cost-competitive pretreatment and hydrolysis of lignocellulose using heterogeneous catalysts that could be simply recycled (Lanzafame et al., 2012), as well as ionic liquids (Olivier-Bourbigou et al., 2010), biphasic solvents (Sakdaronnarong et al., 2016) or deep eutectic solvents (Tang et al., 2017), which are currently emerging as a new class of promising solvents for lignocellulose fractionation and the valorization of lignin.

To eliminate hemicellulose from lignocellulose feedstock, hydrolysis by weak acid catalyst is needed. Among solid acid catalysts used for cellulose hydrolysis, metal oxides have been identified as the most active heterogeneous catalysts, having high conversion and enhanced product selectivity for cellulose hydrolysis. In recent years, the synthesis of various types of solid acids, such as sulfonated or acid-activated carbon-based catalysts (Hu et al., 2015; Pang et al., 2014; Namchot et al., 2014), zeolites (Lanzafame et al., 2012), metal oxide catalysts (Sakdaronnarong et al., 2016, 2017), and magnetic nanoparticle-based catalysts (Xiong et al., 2014), have been studied to achieve higher catalytic activity for cellulose pretreatment and hydrolysis. Among the aforementioned catalysts, acid activated carbon support and mixed metal oxides were promising catalysts, providing large surface areas, improved acidity, high sugar yield and selectivity. To develop a green process for lignocellulose fractionation, biocompatible and environmentally friendly solvents are attractive. A number of studies utilized imidazolium-based ionic liquids (ILs) for lignocellulose pretreatment and fractionation. ILs dissolve crystalline cellulose under mild conditions and offer several advantages for lignocellulose fractionation because of their thermal stability, non-volatility, and recyclability (Olivier-Bourbigou et al., 2010). Moreover, cellulose

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dissolved in ILs undergoes greater enzymatic hydrolysis because of its decreased crystallinity (Swatloski et al., 2002). However, imidazolium ILs are non-biodegradable and are toxic to the environment. Recently, deep eutectic solvents and choline chloride based ILs are emerging as solvents to replace imidazolium-based ILs (Sakdaronnarong et al., 2017; Hou et al., 2012; Liu et al., 2012). These types of solvents have been observed to be easily available, cost-effective, biodegradable, non-toxic or biocompatible, and benign for the environment, especially for the aquatic eco-system (Gadilohar and Shankarling, 2017; Lopes et al., 2013). Using biocompatible and non-toxic ILs for lignocellulosic hydrolysis, residual ILs in cellulose-rich material have an insignificant effect on cellulase activity to produce glucose and subsequently fuel grade ethanol; therefore, a washing process is not necessary.

Lignin is a branched and crosslinked phenolic polymer and thus contributes in a major way to the biochemical and thermochemical recalcitrance of lignocellulose. Consequently, effective fractionation could enhance the efficiency of lignocellulose utilization. Conventionally, lignin residue in black liquor during lignocellulose fractionation is used as a low value fuel in pulping boilers. Thermochemical conversion via pyrolysis is the most suitable valorization method for energy and resource recovery (Ojha et al., 2017). Phenolic bio-oil and biochar or carbon are produced as high-grade chemicals and fuel from lignin pyrolysis (Li et al., 2014). Another route to the valorization of lignin is synthesizing carbon material. Among the carbon materials synthesized, carbon microspheres are attractive because of the variety of applications, such as lubricant, polymer additives, precursor for synthesis of diamond films (Romero-Anaya et al., 2014), anode material for Li batteries (Hu and Shu, 2015), adsorbent, superacid catalyst functionalized by H_2SO_4 (Sakdaronnarong et al., 2016; Namchot et al., 2014; Zainol et al., 2015; Suriyachai et al., 2017) and super-capacitor (Stavropoulos et al., 2015; Zhao et al., 2015). Different materials can be used for the preparation of carbon microspheres by thermal treatment, such as xylose and fructose (Ryu et al., 2010), glucose and xylose (Titirici et al., 2008), cellulose (Sevilla and Fuertes, 2009), polyethylene, and polystyrene (plastic waste) (Romero-Anaya et al., 2014; Gong et al., 2014). Carbon microspheres (CMS) can be prepared by different methods. It has been reported that hydrothermal synthesis is the simplest way to obtain carbon spheres characterized by regular spherical form, and most likely, it also represents the most suitable route for the scale-up of the synthesis process (Frusteri

et al., 2016). For carbon materials produced from lignin, a research work investigated the synthesis of carbon cryogel from lignin and furfural under condensation reaction and subsequent carbonization; it was used as a catalyst for biodiesel production (Zainol et al., 2015). Different drying methods of carbon gel, such as supercritical, freeze and evaporative, would produce aerogels, cryogels, and xerogels, respectively. Additionally, lignocellulosic materials have been objects of considerable interest as precursors for the preparation of spherical activated carbons because of their low cost and because of environmental concerns (Romero-Anaya et al., 2014).

In this work, an integrative near-zero-waste process for simultaneous sugarcane bagasse conversion to glucose, bio-oil and carbon microspheres is proposed. First, a lignocellulose fractionation process with high selectivity for cellulose separation using solid acid catalysts (SACs) in a combination with choline based IL was investigated. Lignin separated from sugarcane bagasse underwent pyrolysis to produce bio-oil and carbon microspheres, from which ratios of carbon to oxygen and carbon to hydrogen in the carbon material were investigated.

2. Materials and methods

Sugarcane bagasse (SCB) containing 35.6% cellulose, 18.6% hemicellulose, 28.4% lignin and 12.4% extractives was supplied from Kornburi Sugar Mill, Nakorn Ratchasima, Thailand. All chemicals were analytical grade and purchased from Sigma–Aldrich, Merck, and Alfa Aesar.

An overall schematic diagram of the experiment including sugarcane bagasse hydrothermal fractionation, catalyst synthesis, cellulose hydrolysis, lignin separation and pyrolysis is illustrated in Fig. 1.

2.1. Catalyst synthesis

In the present work, three types of solid acid catalysts were synthesized: sulfonated silicon dioxide-based catalyst (SiO_2-OSO_3H), heterojunction metal oxide catalyst ($SO_4^{2-}/TiO_2/Fe_3O_4/WO_3$) (Sakdaronnarong et al., 2016) and sulfonated bentonite catalysts.

Sulfonated silicon dioxide-based catalyst (SiO_2-OSO_3H) was sequentially synthesized using SiO_2 as a support. For the synthesis of the self-assembled SiO_2 support, 0.5 g Pluronic F127 was dissolved in 5 mL ethanol and stirred for 4 h. Later, 6 mmol tetraethyl

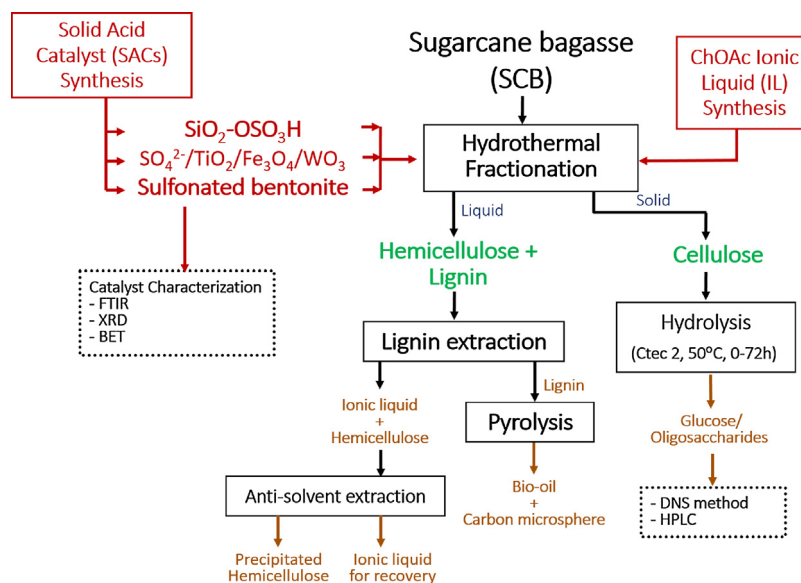


Fig. 1. Overall schematic diagram of experiments on SCB biorefinery.

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