



Simultaneous concentration and detoxification of lignocellulosic hydrolyzates by vacuum membrane distillation coupled with adsorption



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HIGHLIGHTS

- A novel VMD-adsorption process is proposed to treat lignocellulosic hydrolyzates.
- The process achieves simultaneous concentration and detoxification of hydrolyzates.
- Fermentability of hydrolyzates is improved after treatment of the novel process.

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ABSTRACT

Low sugar concentration and the presence of various inhibitors are the major challenges associated with lignocellulosic hydrolyzates as a fermentation broth. Vacuum membrane distillation (VMD) process can be used to concentrate sugars and remove inhibitors (furans) efficiently, but it's not desirable for the removal of less volatile inhibitors such as acetic acid. In this study, a VMD-adsorption process was proposed to improve the removal of acetic acid, achieving simultaneous concentration and detoxification of lignocellulosic hydrolyzates by one step process. Results showed that sugars were concentrated with high rejections (>98%) and little sugar loss (<2%), with the significant reduction in nearly total furans (99.7%) and acetic acid (83.5%) under optimal operation conditions. Fermentation results showed the ethanol production of hydrolyzates concentrated and detoxified using the VMD-adsorption method were approximately 10-fold greater than from untreated hydrolyzates.

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1. Introduction

Recently, cellulosic ethanol has been considered as a potential alternative to partially replace fossil fuel-based resources (Brown, 2015). Cellulosic ethanol is a promising resource because it is sourced from lignocellulosic materials that are abundant and readily available. Although there have been significant developments in biotechnology, there are still many areas that require further development to improve the process efficiencies (Sims et al., 2010). For instance, there are two major challenges preventing the commercialization of cellulosic ethanol technology. First, the sugar concentration after hydrolysis is low for subsequent fermentation, and second, due to the complex composition of lignocellulosic materials, there is a high-content of by-products after thermochemical or dilute acid pretreatment (Ruan et al., 2015).

A low sugar concentration will result in higher energy consumption in the subsequent bioethanol purification process. This result is due to an extremely low ethanol concentration after fermentation. Furthermore, the by-products present, including aliphatic acids, furans and phenolic compounds, can severely inhibit the growth of fermenting microorganisms. This growth inhibition arises because the by-products affect the rate of sugar uptake by the microorganisms, which simultaneously decreases the rate of ethanol production (Mussatto and Roberto, 2004; Palmqvist and Hahn-Hägerdal, 2000). Two of the prototypical inhibitors are acetic acid and furfural. It has been reported that acetic acid is the most abundant toxic compound in hydrolyzates (Nabarlatz et al., 2004). The growth of microorganisms and the production of ethanol would be strongly affected by the presence of acetic acid at concentrations of 3 g/L or higher (Nabarlatz et al., 2004; Palmqvist et al., 1999). On the other hand, furfural is considered to be the most toxic inhibitor to microorganisms, even at low concentrations (Palmqvist et al., 1999). As reported, the furfural concentration at 0.5 and 1.0 g/L inhibited microbial growth by

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25% and 47%, respectively. Moreover, synergistic effects between the fermentation inhibitors and products, such as acetic acid, furfural and ethanol, can drastically hamper the fermentation reactions (Chandel et al., 2013; Nigam, 2001). Lignocellulosic hydrolyzates for fermentation must be concentrated and detoxified in order to obtain satisfactory yield and production of ethanol.

To concentrate lignocellulosic hydrolyzates, removing the solvent (mainly water), has been considered as the primary approach. Methods such as heating, vacuum evaporation and membrane separation, including nanofiltration and reverse osmosis, have been used to concentrate the hydrolyzates (Weng et al., 2010). To detoxify lignocellulosic hydrolyzates, a variety of approaches to detoxify the hydrolyzates have been attempted including biological, physical and chemical methods. Such treatment methods include microbiology (Almeida et al., 2007), overliming (Millati et al., 2002), extraction, vacuum evaporation (Palmqvist and Hahn-Hägerdal, 2000), adsorption (including activated carbon and ion-exchange) (Luo et al., 2002; Parajó et al., 1996; Weil et al., 2002; Zhang et al., 2011) and pervaporation (Sagehashi et al., 2007). However, most of these methods possess several disadvantages. They are time-consuming, require a high energy input, generate additional waste products or suffer from significant sugar loss (Parawira and Tekere, 2011). However, although there are a number of methods currently available to concentrate or detoxify lignocellulosic hydrolyzates, few methods can achieve simultaneous concentration and detoxification of lignocellulosic hydrolyzates. Among the above-mentioned methods, only evaporation can concentration the sugar solutions and simultaneously act as a detoxification method for the removal of particularly volatile compounds (Palmqvist and Hahn-Hägerdal, 2000). However, the removal of solvent by evaporation is energy-intensive and will result in considerable sugar loss due to the high operating temperatures required. Thus, there is a need to develop a novel and energy-efficient technology that can achieve simultaneous concentration and detoxification of lignocellulosic hydrolyzates.

Vacuum membrane distillation (VMD) has been developed as a novel membrane separation process in recent years (Du et al., 2014). This process is thermally driven, whereby an imposed temperature difference between both membrane sides results in a vapor pressure gradient across the membrane. This forces the volatile species present in the aqueous solution to pass through the membrane, while the non-volatile species to retain at the feed side. Similar to traditional distillation, the separation principle of VMD is based on the vapor–liquid equilibrium (VLE), which controls the performance of the VMD process. Thus, VMD can achieve not only the concentration of non-volatile components but also the removal of the volatile components from the aqueous solution. Moreover, VMD is an energy-efficient process, being carried out at low temperatures in the range of 50–80 °C and utilizing low-level or alternative energy sources such as waste hot steam and water, geothermal and solar energy (El-Bourawi et al., 2006). As reported previously, VMD has been applied in a wide range of systems, including the concentration of non-volatile components, such as in water desalination (Mericq et al., 2010) and fruit juice concentration (Meyer et al., 2004). VMD has also been the subject of several studies where the removal of volatile components, including alcohols, ketones and aromatic compounds from aqueous solutions, was explored (Banat and Simandl, 2000; Diban et al., 2009). As such, VMD is a promising, energy efficient and cost-effective approach that warrants further investigation for the simultaneous concentration and detoxification of lignocellulosic hydrolyzates. Previously, the sole VMD process was employed to treat lignocellulosic hydrolyzates, the effects of operating parameters used including the feed rate and temperature were investigated (Chen et al., 2013; Zhang et al., 2012). Results indicated that VMD was highly efficient in concentrating sugars and

removing furans from lignocellulosic hydrolyzates, but not desirable for the removal of less volatile components such as acetic acid. In order to achieve the effective concentration and detoxification of hydrolyzates simultaneously using VMD, the removal of acetic acid must be improved.

In this study, a VMD-adsorption coupled process is developed to improve the removal of acetic acid and further achieve simultaneous detoxification and concentration of the hydrolyzates. According to the separation principle of VMD, a significant acetic acid partial pressure gradient between both membrane sides is key to the successful removal of acetic acid from the hydrolyzates. Therefore, a selective adsorption of acetic acid at the permeate side of the VMD hollow fiber module is proposed to obtain a higher trans-membrane pressure of acetic acid (Fig. 1). When applied to remove acetic acid, this coupled process can be divided into four steps: (1) water and acetic acid molecules transfer from the hot solution bulk to the hot side of the membrane pores; (2) water and acetic acid vaporize on the hot side of the membrane; (3) water and acetic acid vapor diffuse from the hot side of the membrane surface to the cold side through the pores; (4) acetic acid vapor rapidly adsorbs by the adsorbents loaded at the permeate side, while the water vapor partially adsorbs. The VMD-adsorption coupled process is explored by using two adsorbents, activated carbon and a weakly basic resin (Amberlite IRA67), in terms of adsorption efficiency and the mechanism. The efficacy of the VMD-adsorption coupled process is then demonstrated by the fermentation of the concentrated and detoxified hydrolyzates.

2. Methods

2.1. Materials and model hydrolyzates

Analytical grade glucose, acetic acid and furfural were purchased from Sinopharm Chemical Reagent Co., Ltd. (China). Activated carbon made from coconut shells were obtained from Aladdin Company, and weakly basic gel-type resin Amberlite IRA67 were purchased from Shanghai Lang Chemical Co., Ltd.

Model hydrolyzates were prepared by dissolving a certain amount of glucose, acetic acid and furfural in deionized water, based on the real lignocellulosic hydrolyzates obtained from dilute sulfuric acid pretreated corn straw. The initial concentration of glucose was 40 g/L, and the concentration of acetic acid and furfural were 5 and 2 g/L, respectively.

2.2. Dilute acid pretreatment and subsequent enzymatic hydrolysis

Lignocellulosic hydrolyzate produced from corn straw was provided by the College of Life Science and Pharmacy, Nanjing University of Technology. First, the corn straw was milled for hydrolyzation, containing 37.2% cellulose, 17.0% Klason lignin, 22.5% hemicellulose and 4.6% ash. Then, the milled biomass was pretreated with 2% (w/v) H₂SO₄ before being enzymatically hydrolyzed at a solid to liquid ratio of 1:10. The solid and liquid phases were separated by filtration and the liquid phase containing the hydrolyzates were chemically characterized (Yan et al., 2009).

2.3. Experimental setup

The solar-heated VMD apparatus used here consisted of a flat-plate solar energy collector, a hollow fiber module and a permeation condenser (Zhang et al., 2012). Solar energy was explored as heat source for the VMD process, as it is the most abundant green energy with low operating and maintenance costs. In the previous work, the effects of operational conditions, such as feed temperature and velocity, on the VMD performance were

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