



Characterization of oxalic acid pretreatment on lignocellulosic biomass using oxalic acid recovered by electro dialysis



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HIGHLIGHTS

- ▶ Most of the oxalic acid was recovered by electro dialysis treatment.
- ▶ Some of the fermentation inhibitors were removed by electro dialysis treatment.
- ▶ The ethanol production and ethanol yield significantly increased by electro dialysis treatment.
- ▶ The performance of oxalic acid pretreatment using recovered oxalic acid was stable.

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ABSTRACT

The properties of pretreated biomass and hydrolysate obtained by oxalic acid pretreatment using oxalic acid recovered through electro dialysis (ED) were investigated. Most of the oxalic acid was recovered and some of the fermentation inhibitors were removed by ED. For the original hydrolysate, the ethanol production was very low and fermentable sugars were not completely consumed by *Pichia stipitis* during fermentation. Ethanol yield was less than 0.12 g/g in all stage. For the ED-treated hydrolysate, ethanol production was increased by up to two times in all stages compared to the original hydrolysate. The highest ethanol production was 19.38 g/l after 72 h which correspond to the ethanol yield of 0.33 g/g. Enzymatic conversion of the cellulose to glucose for all the pretreated biomass was in the range of 76.03 and 77.63%. The hydrolysis rate on each pretreated biomass was not significantly changed when oxalic acid recovered by ED was used for pretreatment.

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1. Introduction

Biomass has received much attention as a resource for renewable energy. In particular, lignocellulosic biomass such as wood and agronomic residues are regarded as promising energy sources because they do not compete with food sources and can displace fossil fuels (Sun and Cheng, 2002; Soderstrom et al., 2003). Moreover, lignocellulosic biomass has the advantage of being renewable, environmentally friendly, and abundant in the natural world. Lignocellulosic biomass consists of cellulose, hemicelluloses, lignin, and some extractives. Cellulose has a high crystalline structure, and it is complexly connected to hemicellulose and lignin (Chew and Bhatia, 2008). Lignin composed of phenylpropanoid

units has remarkable resistance against chemical and microbial attack. Due to its recalcitrant properties, lignocellulosic biomass has difficulty hydrolyzing carbohydrate to fermentable sugar (Sarkar et al., 2012).

Therefore, pretreatment of lignocellulosic biomass is required to improve the bioconversion process (Chandra et al., 2007; Yang and Wyman, 2008). Various methods for pretreatment have been suggested including chemical, physical, physicochemical, and biological processes (Lloyd and Wyman, 2005; Pan et al., 2005; Ferraz et al., 2001). Especially, supercritical water and ionic liquid treatment have been of great interest because of their potential to effectively remove lignin and hemicelluloses and to decrease cellulose crystallinity (Weerachanchai et al., 2012). Among the pretreatment methods, dilute acid pretreatment with sulfuric acid has been commonly used to hydrolyze hemicellulose. However, it suffers several disadvantages, such as the production of inhibitory products affecting the downstream process, the corrosion of equipment, and the difficulty in the acid recovery after pretreatment (Yang and Wyman, 2008).

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Recently, oxalic acid has been proposed as an alternative to sulfuric acid for pretreatment because it has higher efficiency for hydrolysis than sulfuric acid (Kim et al., 2011; Lee et al., 2009; Mosier et al., 2001). Oxalic acid is less toxic to yeasts than other microbes and does not produce noxious odors during pretreatment (Scordia et al., 2011; Qin et al., 2012). However, oxalic acid is more expensive on a weight basis than sulfuric acid, thus consideration from an economical perspective is required for its commercial use. Recovery processes of used oxalic acid can overcome this problem. Organic acids including oxalic acid can be recovered using conventional techniques, such as ion-exchange and adsorption (Lee et al., 2002; Huang et al., 2007). Research on the recovery and reuse of oxalic acid in the pretreatment process has been rarely reported until now. In this study, oxalic acid pretreatment of a waste mushroom medium was carried out in an oxalic acid recycle process by electrodialysis (ED). We evaluated the recycle performance of oxalic acid by ethanol production and the structural properties of the pretreated biomass. This research could provide useful information on the commercial utilization of oxalic acid for ethanol production.

2. Methods

2.1. Biomass and pretreatment

Waste medium after cauliflower mushroom (*Sparassis crispa*) cultivation was used as biomass in this study. The medium for the cultivation of cauliflower mushroom consists of 80% Douglas fir sawdust, 10% wheat powder, 10% corn powder and 16 brix of oligosaccharide. The biomass was supplied from Jeonnam Forest Resources Research Institute (Naju, Jeonnam, South Korea). The

waste mushroom medium was milled and screened to the size of 40–60 mesh and stored at 4 °C with less than 10% moisture content.

The pretreatment was conducted in 500 ml cylindrical stainless steel reaction vessels. Each vessel was loaded with 50 g (dry weight basis) of material and sufficient oxalic acid/water mixture to give a total solid/liquid ratio of 1:4 (w/w). The pretreatment was performed at 130 °C for 25 min with oxalic acid of 0.064 g/g (dry weight of biomass), and then the temperature was rapidly increased to 170 °C. Reactor heater was turned off when the temperature reached at 170 °C. After pretreatment, each vessel was placed in a water bath until the temperature reached the room value. Subsequently, the liquid fraction (hydrolysate) was separated from the pretreated biomass by vacuum filtration.

2.2. Electrodialysis(ED) to recover oxalic acid in hydrolysate

In order to recover oxalic acid, the ED experiments were performed using a hydrolysate obtained by oxalic acid pretreatment. A ten cell pair stack was assembled in a CJ-S3 electrodialysis stack having a total membrane effective area of 550 cm² (Changjo Techno, Korea). In the stack, NEOSEPTA[®] CMX and AMX (ASTOM Corp., Japan) were used as commercial cation and anion exchange membrane, respectively. In this study, three stages of oxalic acid recovery treatments were followed by ED. Fig. 1 shows the schematic illustration for the recovery and reuse of oxalic acid in the pretreatment process using ED. The hydrolysate in the first stage was obtained from the pretreatment of biomass using oxalic acid, and was then treated with ED to recover the oxalic acid. The recovered oxalic acid was used for pretreatment in the second stage with new

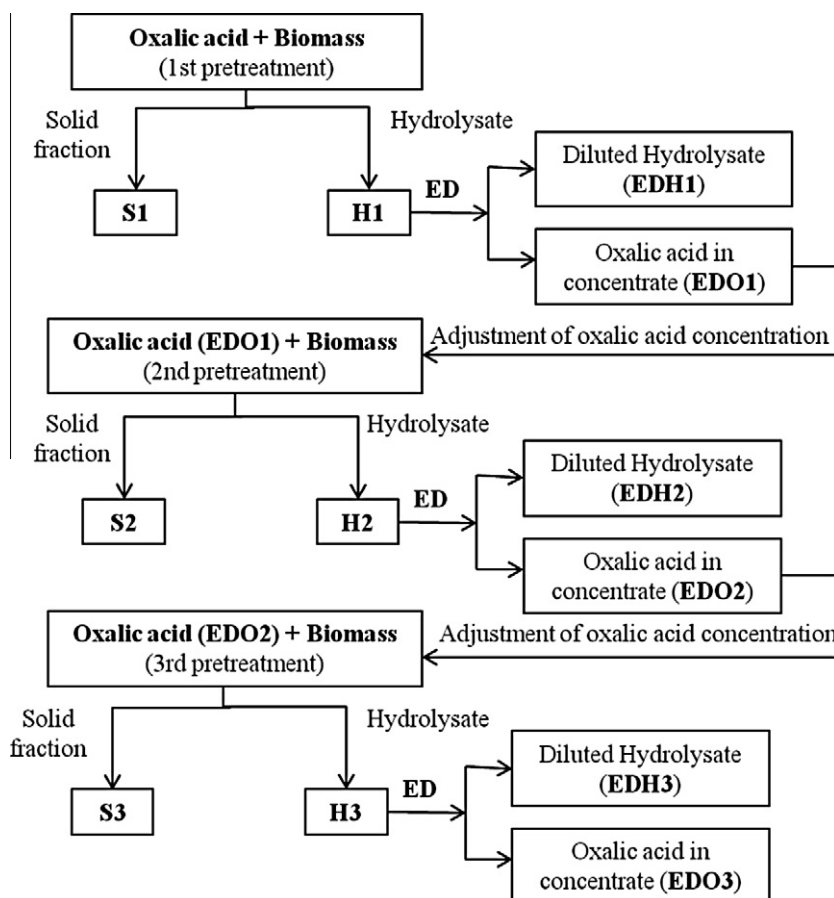


Fig. 1. Scheme of oxalic acid pretreatment on waste mushroom medium using oxalic acid recovered by electrodialysis (ED).

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