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Dowex 1X4 and Dowex 1X8 as substitute of Diaion MA03SS in simulated moving bed chromatographic separation of sulfuric acid and sugars in concentrated sulfuric acid hydrolysates of bamboo

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ABSTRACT

To find commercially available ion exchange resins comparable to a strong-base anion exchange resin Diaion MA03SS, which is specialized for the separation of sulfuric acid and sugars in concentrated sulfuric acid hydrolysates of non-food biomass by means of a simulated moving bed (SMB) chromatography, the screening test of 6 kinds of commercially available resins containing quaternary ammonium groups was carried out by a batch column-mode chromatographic separation of glucose and sulfuric acid at 50 °C. Based on results of the screening test, Dowex 1X4 and Dowex 1X8 were selected as replacements of Diaion MA03SS. The following SMB study using Diaion MA03SS, Dowex 1X4 and Dowex 1X8 at 50 °C clarified that Dowex 1X8 showed performances almost the same as those of Diaion MA03SS in the separations of sulfuric acid and sugars (mainly glucose and xylose) in hydrolysates of bamboo with 27 wt% sulfuric acid under the averaged flow rate of feed loading from 0.136 to 0.273 L/(h L-resin). For Dowex 1X8, recoveries of sulfuric acid were 90.5–93.4% and recoveries of glucose and xylose were 94.9–99.7% and 82.8–88.3%, respectively. Dowex 1X4 showed slightly higher recoveries for three solutes; namely sulfuric acid recoveries were 95.0–95.5% and glucose and xylose recoveries were 100–101% and 86.1–91.7%, respectively. However, the highest averaged flow rate of feed loading for Dowex 1X4 was 0.205 L/(h L-resin) because of the higher column pressure loss owing to its high shrinking in 30 wt% sulfuric acid. Thus, it became clear that Dowex 1X8 exhibit excellent performances comparable to or higher than those of Diaion MA03SS in the SMB chromatographic separation of sulfuric acid and sugars in concentrated sulfuric acid hydrolysate of bamboo.

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1. Introduction

In previous papers [1,2], we reported a process designed to utilize bamboo as a raw material for bioethanol production; this process consisted of the hydrolysis of bamboo with concentrated sulfuric acid, the separation of sulfuric acid and sugars in resulting sulfuric acid hydrolysates of bamboo by means of a Divided Improved Simulated Moving Bed (D-ISMB) system and the continuous ethanol fermentation; these works clarified that the D-ISMB system with 6 columns packed with a strong-base anion exchange resin Diaion MA03SS is promising to recover sulfuric acid at the concentration as high as 187 g/L (16.8 wt%). Diaion MA03SS was specialized for the separation of sulfuric acid and sugars in concentrated sulfuric acid hydrolysates of non-food biomass by means of the D-ISMB chromatography. However, it went out of the production because it was produced on a trial base.

To expand the bioethanol production scale, the D-ISMB system must be enlarged. Then, we had to find out alternative commercial strong-base anion exchange resins with the performances comparative to those of Diaion MA03SS in the separation of sulfuric acid and sugars based on the D-ISMB system. In this context, Heinonen and Sainio comprehensively reviewed chromatographic fractionation of lignocellulosic hydrolysates [3]. In this review, they summarizes uses of ion exchange resins in the chromatographic separation of sugars and various impurities in lignocellulosic hydrolysates including the separation of sulfuric acid and sugars; here, strong-acid cation exchange resins having sulfonic acid groups were much more widely used but not so much use of anion exchange resins including weak-base ones [3]; only several papers were reported in the chromatographic separation of target sugars and impurities [1,2,4–8]. In their review, in addition, there was

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no mention about use of amphoteric ion exchange resins containing both quaternary ammonium and carboxylate groups in this field, although the amphoteric resins have been used in the chromatographic separation of nonelectrolytes and electrolytes since early 1950' [9–11]. Therefore we are also interested in their use in the separation of sulfuric acids and sugars.

In this work, we firstly conducted screening test of resins by evaluations of pulse elution profiles of solutes using a synthetic sample solution of sulfuric acid and glucose and a column filled with each resin given in Table 1 (five kinds of strong-base anion exchange resins including Diaion MA03SS and two amphoteric resins). By referring results given in Table 2, it turned out that Dowex 1X4 and Dowex 1X8 exhibited chromatographic performances comparable to those of Diaion MA03SS. Therefore, secondly, we tested performances of Diaion MA03SS, Dowex 1X4, and Dowex 1X8 in continuous SMB separation of sulfuric acid and monosaccharides (glucose and xylose) using concentrated sulfuric acid hydrolysates of bamboo as feed and a D-ISMB chromatographic system. Dowex 1X4 and Dowex 1X8 also gave almost the same performances as those of Diaion MA03SS as anticipated by screening test based on evaluation of pulse elution profiles.

2. Methods

2.1. Screening test by a batch column method

2.1.1. Materials

Table 1 lists disclosed properties of commercially available ion exchange resins having quaternary ammonium groups fixed to polystyrene matrices crosslinked with divinylbenzene (DVB). Diaion MA03SS, Dowex 1Xn (n = 2, 4, 8) and Amberjet 4002 are strong-base anion exchange resins. Among them, Diaion MA03SS is specialized for the SMB separation of sulfuric acid and sugars but its detailed properties were not disclosed. As amphoteric resin, Diaion AMP03 and Retardion 11A8 were used. The ion exchange groups of Diaion AMP03 in electrically neutral form are $-N^{+}(CH_3)(CH_2COOH)(CH_2COO^{-})$; then ammonium to acetic acid molar ratio is equal to 0.5. Retardion 11A8 is a snake cage resin [9], which is a Dowex 1 resin containing polyacrylate anions as polymeric counter ions; its quaternary ammonium and carboxylate contents are 1.1 and 1.2 meq/mL, respectively. Diaion MA03SS was purchased from Mitsubishi Chemical Co. Ltd., Japan. Dowex 1Xn (n = 2, 4 and 8) were from Wako Pure Chemical Industries Co. Ltd., Japan. Amberjet 4002 and Retardion 11A8 were purchased form Organo Co. Ltd., Japan and Muromachi Technos Co. Ltd., Japan, respectively. The amphoteric resin Diaion AMP03 was kindly provided by Mitsubishi Chemical Co. Ltd.; Diaion AMP03 is an improved resin of Diaion AMP02. Sulfuric acid (47 wt%) of guaranteed grade was obtained from Wako Pure Chemicals Co. Ltd., Japan. β-D-glucose was obtained from Tokyo Kasei Co. Ltd., Japan. CHN analysis of ion exchange resins was conducted by Engineering Research Equipment Center, Faculty of Engineering, Kumamoto University; here, a sample of each resin after conditioning was air dried and then dried on P_2O_5 in vacuum desiccator until its weight became constant.

2.1.2. Batch column-mode chromatography (pulse elution profiles test using a synthetic solution containing sulfuric acid and glucose)

The experimental setup consisted of a peristaltic pump, a switch cock for feed and eluent, a glass chromatographic column (1.5 cm inner diameter) with water heating jacket, a water circulating thermostated bath and a fraction collector. All resins received were conditioned in column-mode by successively feeding water, 1 M NaOH, water, 1 M HCl and water just prior to use. Then, each conditioned resin was packed into the chromatographic column. Before the separation experiment, 3.73 M (29.9 wt%) sulfuric acid (25 mL) was fed to the column to change ionic form of the resin into sulfate ion form. Then the column was washed with water until the washing became acid-free and the resin bed volume was adjusted to be 50 mL (the resin bed height 28.3 cm). Then, the temperature of the column was kept at 50 °C by circulating water from the circulating thermostated water bath. After column temperature attained at 50 °C, a sample solution containing both 3.32 M H_2SO_4 (326 g/L, 27.1 wt%) and 150 g/L glucose (15 mL, 0.30 bed volumes) was loaded on to the column and water as eluent was immediately supplied to the column. Composition of the sample solution and the loaded sample volume were determined by referring to the previous work [1]. In this experiment, the flow rate of the sample solution and water was fixed at an hourly flow rate of 2 bed volumes. Column effluent was collected on to a fraction collector and a volume of each fraction was 5 mL (0.10 bed volumes). Concentration of sulfuric acid was determined by acid-base titration and that of glucose by the Somogyi-Nelson method [12,13]. Dowex 1X4 and Dowex 1X8, which gave chromatographic performances comparable to those of Diaion MA03SS, were selected by referring to the results given in Table 2.

2.2. Separation of sugars and sulfuric acid in the saccharified liquid using a D-ISMB system

2.2.1. Materials

Three kinds of strong-base anion exchange resins, Diaion MA03SS, Dowex 1X4 and Dowex 1X8, in sulfate form were used. Each resin received in chloride ion form (ca. 2.5 L) was converted into sulfate ion form with 30% Na_2SO_4 solution (5 L) prior to use. In packing each resin into columns, the resin slurry was poured into empty columns quickly. After the excess mobile phase was removed, the bed was compressed to the bed height of 50 cm.

Resin shrinking measurement was carried out according to a reported method [14]. A sample of each dry resin (10.0 g) was put into glass containers with 50 mL of 30 wt% sulfuric acid or distilled water, and was equilibrated for 24 h. The resin shrinking was

Table 1
Properties of gel type commercially available ion exchange resins tested in this work.

Name	Type	Ionic form as received	Nitrogen content (mmol/g)[b]	Degree of crosslinking	Particle size (μm)	Total capacity (meq/mL)	Moisture regain (%)
Diaion MA03SS	Strong-base	Chloride	3.4	–	100–200	–	–
Dowex 1X2	Strong-base	Chloride	3.1	2	110–250	0.6	70–80
Dowex 1X4	Strong-base	Chloride	3.6	4	110–250	1.0	55–63
Dowex 1X8	Strong-base	Chloride	3.0	8	110–250	1.2	40–48
Amber JET 4002	Strong-base	Chloride	3.7	–	500–650	1.3	49–55
Diaion AMP03	Amphoteric[a]	–	3.5	–	260	–	–
Retardion 11A8	Amphoteric[a]	–	2.5	–	150–500	–	43–48

[a] Ion exchange group: quaternary ammonium and carboxylate. For detail, refer to text.
[b] Measured in this work by CHN analysis of dried resins.

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