



Short communication

Adsorption behaviors of sugars and sulfuric acid on activated porous carbon



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ABSTRACT

To investigate the suitability of activated porous carbon as the adsorbent for separation of sugars and sulfuric acid, the relevant adsorption equilibrium data were obtained by using a staircase frontal analysis method in the range of sulfuric acid concentration (0–13.61 g/L), glucose concentration (0–5.86 g/L), and xylose concentration (0–34.96 g/L). It was found from the resultant adsorption data that sugars had considerably higher adsorption affinities to activated porous carbon than sulfuric acid, which indicates that activated porous carbon has the potential to be utilized as an economical adsorbent for separation of sugars and sulfuric acid. It was also found that the resultant adsorption data could be well predicted by the Langmuir isotherm model.

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Introduction

One of the noteworthy developments in the field of renewable chemical industries was to utilize biomass such as corn stover for producing bio-ethanol and lactic acid, which have the potentials to be exploited as an alternative source of energy and a starting material of the biodegradable plastic polylactide respectively [1–7]. An important intermediate step in such production of biomass-derived chemicals is to hydrolyze the primary polysaccharide components of biomass into sugars (glucose and xylose) by using sulfuric acid as hydrolysis catalyst [1,5–9]. To ensure high fermentability of the hydrolyzed sugars into the aforementioned products (bio-ethanol and lactic acid), it is essential to separate sulfuric acid from sugars before fermentation.

To address the necessity for such separation, several ion-exclusion resins from major resin-production companies have been utilized as an effective adsorbent [1,5–9]. In addition, poly-4-vinyl pyridine (PVP) resin from Reilly Industries Inc. was also found to be useful for such a separation task [6,7]. All of these polymeric adsorbents are known to have an advantage in the aspect of regeneration and re-use [8]. However, all of them are relatively on

the expensive side, which are approximately in the range of \$118–\$2260 per kilogram on the basis of the retail price in Korea. This has sometimes caused the necessity for exploring another adsorbent that can be much more advantageous in terms of price competitiveness while maintaining its suitability for the aforementioned separation task. In regard to this issue, it is worth paying attention to a recent technical report [10], which mentioned the feasibility of using activated porous carbon as the adsorbent for separation of sulfuric acid and sugars.

It is well known that activated porous carbon possesses highly developed internal surface area and porous structure, and hence a large capacity for adsorbing chemicals from gases and liquids [11,12]. Furthermore, its retail price in Korea is approximately in the range of \$8–\$9 per kilogram, which is much cheaper than other polymeric adsorbents (\$118–\$2260 per kilogram) [10–12]. Due to such a well-established structure and such a low cost, activated porous carbon has attracted a lot of attention as a versatile and economical adsorbent in many industries.

Therefore, it is quite worth considering the development of an adsorption separation process based on activated porous carbon that is aimed at separating sulfuric acid and sugars. One of the essential prerequisites for such a process development is to investigate the adsorption behaviors of sulfuric acid and sugars on activated porous carbon and then to obtain the relevant adsorption equilibrium data. However, such work has not been performed so far in the literature.

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The goal of this study is to accomplish the aforementioned prerequisites that can serve as essential information in the stage of designing the adsorption process based on activated porous carbon for separation of sulfuric acid and sugars. For this purpose, the single-component adsorption equilibria of sulfuric acid and sugars on activated porous carbon will be measured on the basis of a staircase frontal analysis (SFA) method [13–15], which has been recognized to be highly accurate in acquiring adsorption equilibrium data. For such work, a model solution based on reagent-grade sulfuric acid and sugars will be used instead of a real hydrolyzate, and a maximum liquid-phase concentration for each compound will be set to be the same as in the actual corn-stover hydrolyzate, which was reported to contain 13.61 g/L of sulfuric acid, 5.86 g/L of glucose, and 34.96 g/L of xylose [6]. Although other concentration ranges of sulfuric acid and sugars in biomass hydrolyzate can be found in several previous publications [16,17], only the concentration range in the reference [6] will be taken into account in this work. In addition, other byproducts from the acid hydrolysis of corn-stover hydrolyzate, which include degradation byproducts (furfural and hydroxymethylfurfural) and several other sugar components (cellobiose, mannose, galactose, and arabinose) [6], will not be covered in this study because of the following two reasons: (1) such by-products were contained in the hydrolyzate with small amounts and (2) the separation between sulfuric acid and sugars has been a main subject in most of the related literature publications [5–9].

The relevant adsorption experiments for implementation of the aforementioned SFA method will be carried out at the temperature of 65 °C, which is the same as in the previously reported adsorption processes based on other adsorbents (ion-exclusion and PVP resins) for separation of sulfuric acid and sugars [1,5–7]. Furthermore, we will suggest an appropriate adsorption model for predicting the measured equilibrium data over the investigated range of liquid-phase concentration, which is set to cover the feed concentration of the actual biomass hydrolyzate [6]. Finally, the relevant parameters of the suggested adsorption model will be determined.

Experimental

Materials

Glucose and xylose were purchased from Sigma-Aldrich Co. (St. Louis, MO), and 1 N sulfuric acid aqueous solution was supplied from Yakuri Pure Chemicals Ltd. (Osaka, Japan). Distilled deionized water (DDW) was obtained from a Milli-Q system by Millipore (Bedford, MA) and used as an eluent in the SFA experiments performed. Activated porous carbon (CPG[®] LF 12 × 40) was purchased from Calgon Carbon (Pittsburgh, PA), and sieved into particles with diameter from 425 to 710 μm before its use as an adsorbent. This adsorbent was packed into a jacketed glass column, which was purchased from Ace Glass Inc. (Louisville, KY). The column has a diameter of 1.1 cm and a packing length of 30.5 cm. The intra-particle porosity of the adsorbent was 0.725, which was taken from the literature [18]. The inter-particle porosity of the packed column was determined in the following manner. First, the packed column was pre-treated such that the solid phase (adsorbent) could have no adsorption affinity to H₂SO₄, which was implemented by saturating the column with a sufficiently high concentration of H₂SO₄ solution to keep the solid-phase concentration far higher than its saturation capacity. Under such condition, a pulse test with H₂SO₄ was carried out and its retention time was measured. Based on this result, the total porosity of the packed column was estimated. The estimated total porosity was then combined with the aforementioned intra-particle porosity in order

to calculate the inter-particle porosity. The resulting value of the inter-particle porosity was 0.362.

Apparatus

The experiments associated with the implementation of the SFA method were conducted with the HPLC system, which was manufactured by Young Lin Co. (Anyang, South Korea). This system consisted of two HPLC pumps (Young Lin SP-930D), a refractive index detector (Young Lin 750F), an HPLC mixer from Analytical Scientific Instruments Co. (El Sobrante, CA), an HST-250WL circulator from Hanbaek Co. (Bucheon, South Korea), and a BW-20G water bath from Jeio Tech Co. (Daejeon, South Korea). The experimental data from this HPLC system were collected and analyzed with the help of automatic data acquisition software (Young Lin Autochro-3000), which operated in the Windows environment.

Method

The acquisition of the adsorption equilibrium data of interest were conducted in accordance with the staircase frontal analysis (SFA) method [13–15]. Before explaining the experimental procedure relevant to the SFA method, its principle is summarized first as follows. This method usually begins by the feeding of a solution with known concentration into a column packed with adsorbent. Such a feed-loading lasts until the equilibrium liquid-phase concentration (C) becomes identical with the concentration of the feed solution (C_{feed}). Under this condition, the equilibrium solid-phase concentration for a given feed concentration can easily be estimated through a material balance over the packed column. This procedure is then repeated by increasing stepwise the feed concentration, which is continued until the final feed concentration reaches the highest liquid-phase concentration investigated (i.e., the upper bound for the liquid-phase concentration). This can eventually allow us to determine a set of solid-phase concentrations (q_i) in equilibrium with a given range of liquid-phase concentrations (C_i). The mathematical expression relevant to such procedure, which results from the material balance for each step (i.e., each period between the loadings of the $(i-1)$ th feed concentration and the i th feed concentration), can be found elsewhere [13,14,19].

To realize the aforementioned SFA principle experimentally, the two pumps in the HPLC system were utilized simultaneously. One pump delivered DDW and the other pump the stock solution that was prepared by dissolving one of the considered components into DDW. The concentration of each component in the stock solution was set at its real concentration in the actual biomass hydrolyzate [6]. The streams from the two pumps were mixed before being fed into the column packed with activated porous carbon. Such a mixing process was facilitated by the HPLC mixer, which enabled the two streams to attain the state of perfect mixing before being fed into the packed column. The total flow rate for the mixed stream that was fed into the column was kept constant at 2 mL/min. The concentration of the solution fed into the column (i.e., the feed concentration) was controlled by adjusting the ratio of the two streams. The ratio was changed only after a concentration plateau was fully developed at the column outlet. The column effluent was monitored using the refractive index detector.

In order to maintain temperature at 65 °C during the experiments, water at a fixed temperature of 65 °C was continuously circulated through the jacket enclosing the packed column, which was carried out by the HST-250WL circulator. In addition, both reservoirs containing DDW and the stock solution respectively were immersed in the BW-20G water bath, which was also maintained at 65 °C.

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