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Adsorption behavior of benzenesulfonic acid by novel weakly basic anion exchange resins

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ABSTRACT

Two novel weakly basic anion exchange resins (SZ-1 and SZ-2) were prepared via the reaction of macroporous chloromethylated polystyrene-divinylbenzene (Cl-PS-DVB) beads with dicyclohexylamine and piperidine, respectively. The physicochemical structures of the resulting resins were characterized using Fourier Transform Infrared Spectroscopy and pore size distribution analysis. The adsorption behavior of SZ-1 and SZ-2 for benzenesulfonic acid (BA) was evaluated, and the common commercial weakly basic anion exchanger D301 was also employed for comparison purpose. Adsorption isotherms and influence of solution pH, temperature and coexisting competitive inorganic salts (Na₂SO₄ and NaCl) on adsorption behavior were investigated and the optimum desorption agent was obtained. Adsorption isotherms of BA were found to be well represented by the Langmuir model. Thermodynamic parameters involving ΔH , ΔG and ΔS were also calculated and the results indicate that adsorption is an exothermic and spontaneous process. Enhanced selectivity of BA sorption over sulfate on the two novel resins was observed by comparison with the commercial anion exchanger D301. The fact that the tested resins loaded with BA can be efficiently regenerated by NaCl solution indicates the reversible sorption process. From a mechanistic viewpoint, this observation clearly suggests that electrostatic interaction is the predominant adsorption mechanism. Furthermore, results of column tests show that SZ-1 possesses a better adsorption property than D301, which reinforces the feasibility of SZ-1 for potential industrial application.

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Introduction

In the past several decades, water safety has been of increasing concern all over the world and water pollution through organic compounds has drawn great attention. Aromatic sulfonic acids (ASAs), which are widely used as intermediates in the manufacturing industry like tanning agents, dyestuffs and pharmacy, are of special interest due to the good solubility in water and negative impacts to water resources once discharged with industrial wastewater (Pan et al., 2005; Zhu et al., 2012). Usually, wastewaters containing ASAs are disposed by biodegradation, advanced oxidation, extraction and adsorption processes (Igwo-Ezikp et al., 2010; Wang et al., 2013). However, biodegradation needs specific cultured bacteria because of the poor biodegradability of ASAs. Advanced oxidation process may have better removal efficiency, but the cost of oxidant, catalyst and reactor is very high (Mauro et al., 2009). Extraction can remove ASAs or its intermediates from wastewaters, but it may cause a secondary pollution by solvent loss in wastewaters (Li et al., 2007; Sun et al., 2011). Adsorption is one of the most effective processes for removal and recovery of organic pollutants from contaminated waters. However, due to their high

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water solubility and strongly polar nature, ASAs cannot be removed effectively by traditional adsorbents such as activated carbon and polymeric adsorbents except for the polymeric anion exchange resins (Xu et al., 2012; Yang et al., 2007a, 2007b).

Although strongly basic resin shows high selectivity for ASAs because of strong electrostatic interaction, its regeneration process is a tough challenge. Otherwise, commercial available weakly basic resin possesses relatively better regeneration performance, but its obvious disadvantage is relatively low selectivity for the removal of ASAs from wastewaters in the presence of inorganic salts which can strongly compete for sorption sites on resin with ASAs (Pan et al., 2008a).

In view of the above-described drawback of commercial anion exchanger, the main target of this study is to synthesize novel weakly basic anion exchange resins for ASAs removal from industrial wastewaters with high level of inorganic salts. Benzenesulfonic acid (BA) was chosen as a representative of ASAs while Na₂SO₄ and NaCl were used as the competitive inorganic salts due to their widespread occurrence in wastewaters. Batch experiments were carried out to test the adsorption performance of the resulting resins, especially to evaluate selectivity of sorbents for BA in existence of inorganic salt. After adsorption, the regeneration efficiency of exhausted resin was evaluated by three agents including ethanol, 5% NaCl water solution and 5% NaCl ethanol-water (50/50 mass ratio) solution. To evaluate the adsorption performance of weakly basic anion exchange resins for practical application, the column experiments were carried out using the simulating BA wastewater with competing Na₂SO₄.

1. Materials and methods

1.1. Materials

Macroporous chloromethylated polystyrene-divinylbenzene (Cl-PS-DVB) beads (cross-linking density 6%, chlorine content 18%) and commercial weakly basic anion exchanger D301 (cross-linking density 6%) were purchased from Zhengguang Co., Ltd. (Zhejiang, China). BA was purchased from Aladdin Industrial Corporation (Shanghai, China). Dicyclohexylamine and piperidine were purchased from Sinopharm Chemical Reagent Co., Ltd. (Beijing, China) and Kelong Chemical Reagent Co., Ltd. (Bijing, China), respectively. The other chemical reagents such as NaCl, Na₂SO₄, HCl, H₂SO₄, NaOH and N,N-dimethylformamide (DMF) were purchased from Nanjing Reagent Co., Ltd. (Jiangsu, China). All the chemicals were of analytical grade and used without further purification.

1.2. Resin synthesis and characterization

Forty grams of Cl-PS-DVB beads was swelled in 80 mL of DMF at room temperature under mechanical stirring. Afterwards, 80 g of dicyclohexylamine was added slowly. The mixture was then stirred for 12 hr at 353 K. Finally, the weakly basic anion exchanger SZ-1 was obtained through filtration of residual dicyclohexylamine. SZ-2 resin was synthesized with similar procedure except for the piperidine dosage adjusted to 38 g.

All the resins were washed by 4% hydrochloric acid and pure water flushing to neutral pH prior to further use. Finally, the resins were extracted with ethanol for 8 hr in a Soxlet apparatus, and then vacuum desiccated at 323 K for 8 hr.

Infrared spectra of Cl-PS-DVB beads and both the newly synthesized resins were taken from a Nicolet 5700 Fourier Transform infrared spectroscopy (FT-IR) (Madison, WI, USA) with a pellet of powdered potassium bromide and adsorbent. BET surface area and pore size distribution of the resins were determined by a Micrometics ASAP-2010C automatic surface area analysis instrument (Micromeritics Instrument, Norcros, USA) with nitrogen as adsorbate following the BET method.

1.3. Static adsorption

Dry resins (0.100 g) were introduced into a series of 150 mL glass flasks containing 100 mL solution, and the concentrations of BA varied from 100 to 1000 mg/L. Sulfuric acid or sodium hydroxide was used to adjust the solution pH and sodium sulfate or sodium chloride was introduced into the flask before adsorption when necessary. After that, flasks were completely sealed and put into a THZ-82 shaker with thermostat (Changzhou Guohua Scientific Instruments, Jiangsu, China) at certain temperature for 24 hr and 120 r/min to ensure the adsorption process to reach to the equilibrium. Concentrations of BA were determined through UV1800 spectrophotometer (Shanghai Jinghua Scientific Instruments, China) at 263 nm wavelength. The adsorption capacity Q_e (mmol/g) was calculated using Eq. (1):

$$Q_e = V(C_0 - C_e) / WM \tag{1}$$

where, V (L) is the volume of solution; W (g) is the weight of dry resin; C_0 (mmol/L) and C_e (mmol/L) are the initial and equilibrium BA concentrations respectively; and *M* is the molecular weight of BA.

1.4. Desorption experiments

One hundred milliliters of BA solution with an initial concentration of 1000 mg/L was introduced into a series of 150 mL flasks and shaken with 0.100 g dry resin for 24 hr. After adsorption, the resin beads were transferred to another flask after filtration. One hundred milliliters of ethanol, 5% NaCl water solution or 5% NaCl ethanol–water (50/50, mass ratio) solution was separately added into each flask. The flasks were then put into the rotary shaker under 120 r/min for 24 hr at 303 K, afterwards, the amounts of BA eluted were measured.

1.5. Column sorption experiments

Column experiments were carried out with a glass column (18 mm in diameter). A 10 mL portion of resin (wet volume) was packed into individual columns for further study. A synthetic solution containing 2000 mg/L BA and 1% Na₂SO₄ was employed as the feeding solution and pumped down-flow through the column at 298 K. The hydrodynamic conditions, *i.e.*, a superficial liquid velocity (SLV) of 0.08 m/hr and an empty bed contact time (EBCT) of 30 min were identical for the column adsorption runs.

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