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Chemical Engineering Research and Design

journal homepage: www.elsevier.com/locate/cherd

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Synthesis, adsorption and regeneration of nanoporous silica aerogel and silica aerogel-activated carbon composites

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A B S T R A C T

Usage of aerogels as an adsorbent has become more widespread because of its specifications such as high porosity and specific surface. Nanometer silica aerogel and silica aerogel-activated carbon composites were synthesized using a water glass precursor by ambient pressure drying method. Then, the adsorption capacity of synthesized adsorbents was studied in terms of benzene and ethyl benzene adsorption by chromatography method for continuous and batch testing. Results showed that silica aerogel and silica aerogel-activated carbon composites had high tendency for benzene and ethyl benzene adsorption. Silica aerogel showed maximum adsorption capacity of 2.3 g g^{-1} and 0.7 g g^{-1} in static adsorption of benzene and ethyl benzene respectively. Also, in dynamic adsorption of benzene and ethyl benzene, silica aerogel had maximum equilibrium adsorption capacity of 0.954 g g^{-1} and 0.219 g g^{-1} respectively. Minimum equilibrium adsorption capacity in benzene and ethyl benzene static adsorption was related to activated carbon with 0.7 g g^{-1} and silica aerogel-2 wt% activated carbon with 0.25 g g^{-1} respectively. After adsorption process, silica aerogel and silica aerogel-0.5 wt% activated carbon composite were regenerated by solvent extraction-thermal treatment method and, after at least 15 adsorption/desorption cycles, their adsorption capacity became fixed.

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Keywords: Adsorption; Equilibrium adsorption capacity; Regeneration; Silica aerogel; Pore size distribution; BET surface area

1. Introduction

Volatile Organic Compounds (VOC) are the most common air pollutants emitted from chemical, petrochemical, motor vehicle exhaust, industrial paints, allied industries and use of organic solvents (Štandeker et al., 2009; Song et al., 2005; Carrasco et al., 2009). Contamination caused by volatile organic compounds in the environment is mainly the result of the historic disposal practices of industrial wastes containing these solvents, most of which have been considered dangerous materials since the early 1970s, when the first environmental laws were enacted (Health Effects Fact Sheet, 2000). Atmospheric emissions of these volatile organic compounds cause serious environmental problems and financial losses. They are toxic and carcinogenic to human health (Štandeker

et al., 2009; Song et al., 2005) and cause serious environmental problems such as the destruction of the ozone layer (Song et al., 2005), global warming (Dou et al., 2011), and formation of tropospheric ozone and other oxidants that cause photochemical smog (Carrasco et al., 2009; Atkinson, 2000). Therefore, controlling VOC emissions according to increasingly stringent environmental regulations is necessary. There are many separation processes to address this problem [B] and prevent VOC effluence such as condensation (Štandeker et al., 2009; Anita and Wilfreid, 1999), catalytic oxidation (Carrasco et al., 2009; Ruddy and Carroll, 1993; Patkar and Laznow, 1992), absorption (Song et al., 2005) membrane-based recovery process (Semenova, 2004; Khan and Ghoshal, 2000), and adsorption (Dou et al., 2011; Crittenden and Thomas, 1998). One of the most widely used methods for controlling VOC emissions and

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Received 10 March 2014; Received in revised form 21 August 2014; Accepted 3 September 2014

Available online 16 September 2014

<http://dx.doi.org/10.1016/j.cherd.2014.09.003>

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the removal of them in gaseous streams is adsorption, for which adsorbents with high specific surface areas and the possibility to be used in many cycles are needed (Štandeker et al., 2009; Song et al., 2005; Abril et al., 2010). Small pores, such as micropores and mesopores, result in large specific surface area responsible for adsorption. Pore size, pore distribution and surface area, as well as pore surface chemistry, are the major factors in the adsorption process (Yang, 2003). Silica aerogels are extremely porous (Bhagat et al., 2008; Alnaief and Smirnova, 2010), nanostructured (Bangsi et al., 2008), and mesoporous (Ciolek, 2006) and have high specific surface areas (Gurav et al., 2009a; Rao et al., 2006) that are easily recovered. They are stable even after many adsorption/desorption cycles with no loss of efficiency, they are easily recovered and also exhibit capacities which enormously exceed those of commonly used adsorbents such as activated carbon and silica gel (Štandeker et al., 2009; Song et al., 2005). In 2009, Štandeker et al. showed that silica aerogels were excellent adsorbents for the adsorption of BTEX¹ vapors from polluted gas stream in comparison to activated carbon and silica gel. Despite their good potential applications, the usage of aerogels as an adsorbent has been restricted due to their weak and fragile mechanical strength. One of the appropriate methods to overcome and strengthen the mechanical fragility of aerogels is the preparation of silica aerogel composites (Rao et al., 2006; Kim et al., 2008). In 2003, Coleman et al. found that silica aerogel-granulated activated carbon was superior in terms of removing uranium from a stock solution compared to granulated activated carbon alone for batch testing. In 2011, Dou et al. showed that silica aerogel-activated carbon composite has high affinity towards aromatic molecules and fast adsorption kinetics. Excellent performance of dynamic adsorption and desorption observed on silica aerogel-activated carbon composite is related to its higher adsorption capacity (Dou et al., 2011). Nanostructured silica aerogels are synthesized by a two-step sol-gel route and drying of wet gel to remove the trapped solvent from the pores of the gel (Bhagat et al., 2006; Gurav et al., 2009b). Aerogels, based on water glass precursor, are hydrophilic and become wet with atmospheric moisture or water, but with appropriate chemical modification the surface of the aerogel can be converted to hydrophobic so that the water molecules will be repelled (Štandeker et al., 2009). The main reason for hydrophilicity of silica aerogels is the presence of Si–OH groups in aerogel structure as they promote the adsorption of water. By replacing Si–OH groups with Si–CH₃ groups, hydrophobic silica aerogels are obtained, because the adsorption of water is prevented. Preparation of silica aerogels using water-glass precursor followed by an ambient pressure drying (APD) is the cheapest and safest method for aerogel production (Gurav et al., 2009b; Shi et al., 2006). In order to make the use of silica aerogels economically feasible on a large scale, the exhausted adsorbents must be regenerated and reused (Abril et al., 2010; Garcia-oton et al., 2005). Silica aerogels are easily regenerated as a result of having an open pore structure and being mesoporous (Štandeker et al., 2007). Regeneration of the silica aerogel can be performed using different methods such as thermal, extractive, chemical, electrochemical and biologic ones. Among these methods, thermal regeneration and solvent extraction are considered two of the most effective (Abril et al., 2010). The production of hydrophobic silica aerogels has become an important and

intensive area of research, like many of the scientific and technological applications of the aerogels (Rao and Kulkarni, 2002). Therefore, in this study, silica aerogel and its composites with activated carbon were synthesized using water-glass precursor by a two-step sol-gel process followed by ambient pressure drying. Performance of synthesized adsorbents was studied under static and dynamic conditions for benzene and ethyl benzene adsorption in fixed beds. So, for the first time, exhausted adsorbents were regenerated by solvent extraction-thermal treatment combined method.

2. Experimental procedure

2.1. Synthesis of silica aerogel

The chemicals used for the synthesis of silica aerogel were water-glass (Na₂SiO₃, 1.35 g ml⁻¹, Merck Co., Germany), trimethylchlorosilane (TMCS) (C₃H₇ClSi, 0.86 g ml⁻¹, Merck Co., Germany), isopropyl alcohol (IPA), ammonium hydroxide solution (1.0 M), and n-hexane. The ion exchange resin, Amberlite IR 120 H⁺ (Merck Co., Germany), was used for replacing the Na⁺ ions present in the water-glass with the H⁺ ions. Hydrogels were prepared using water-glass by a two-step sol-gel process. The water-glass solution was first diluted with the deionized water (water-glass: deionized water (volume ratio) = 1:4) so as to obtain the desired weight percentage of silica in the starting material. In the next step, the ion exchange was carried out by mixing the diluted water-glass solution with the ion exchange resin in 1:1 volume proportion. The mixture was stirred for 5 min and, consequently, pH of the ion exchanged solution changed from 13 to 2, clearly showing the removal of unwanted Na⁺ ions from the water-glass solution and resulting in the formation of silicic acid (SA). Then, 1.0 M NaOH solution was added to the silica sol to raise its pH to 4 for gelation. The obtained silica sols were stirred for 1 min and then transferred to a plastic beaker with 30 mm diameter and 60 mm height, where the sols aged into hydrogels within about 180 min at 60 °C. The next step was to immerse the hydrogels in isopropyl alcohol and normal hexane and then subsequently age them for 18 h at 60 °C in order to extrude pore water (exchanged by n-hexane) and also strengthen the networks of the gels. After solvent exchanging and aging, the wet gels were immersed in TMCS/n-hexane solutions for 12 h at 60 °C in order to adequately modify the surface and discharge the pore water, mainly containing H₂O/n-hexane solutions. Volumetric ratio of n-hexane/TMCS was fixed at 5, because less shrinkage was observed at this ratio. Finally, the modified gels were dried at room temperature for 24 h and then at 60, 80, 120, and 180 °C for 2 h, respectively, in the oven to reduce shrinkage during drying. In the synthesis of silica aerogel-activated carbon composite, powdery activated carbon (Iran, 200 mesh, 1.6 g cm⁻³, 300 m² g⁻¹) was immersed in silica sol after the ion exchange step.

2.2. Characterizing aerogels

Apparent density of the aerogel was measured by mass to volume ratio ($\rho = mv^{-1}$) of the aerogel, in which mass was measured by the microbalance with the accuracy of 10⁻⁵ g and volume of the aerogel was measured by filling aerogel in the measuring cylinder of the known volume. The microstructure and morphology of silica aerogels were observed by scanning electron microscopy (SEM: JSM-6700F). Specific surface area and pore size distribution of aerogels were determined

¹ Benzene, Toluene, Ethylbenzene and Xylenes.

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