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Preparation of modified superhydrophobic sponge and its application in xylene leakage recovery

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ABSTRACT

In recent years, a considerable increase in demand and transportation of organic chemicals has caused frequent spill accidents, usually posing a severe threat to the aquatic ecological system. In order to recycle and remove chemicals from water effectively, modified melamine sponge with a superhydrophobic surface was prepared by a facile and inexpensive method. Polyvinyl chloride and modified SiO_2 nanoparticles were applied to fabricate water-repellent coatings on the surface of melamine sponge. The results showed that the water contact angle of the modified sponge was improved to 150°. The sorption capacity for xylene reaches 41.45 g/g, which can still achieve 93% after 500 cycles of absorption and desorption. And the xylene – water separation efficiency can reach 99%. The results indicate that the modified sponge has a great application perspective in xylene leakage recovery and water purification.

Keywords: Superhydrophobic surface; Modification; Xylene recovery; Adsorption; Separation

1. Introduction

Benzene series such as xylene, as widely used chemical products, play a vital part in the development of industrialization and economy. However, frequent leakage accidents were happened due to transportation accidents. A typical example is the 2002 Japanese coast xylene leakage, which may result in long-term effects on marine life, fishing industry, and so on. For the pollutant water caused by these leakage accidents that can bring a severe impact on the marine ecosystem, environment, and public health [1–3], many technologies have been developed to eliminate pollution and alleviate the energy sources loss caused by these accidents [4,5]. The conventional methods for oil-spill cleanup are mechanical recovery (adsorbents [6], skimmers [7]), chemical methods (dispersants [8]), and bioremediation [9]. Among them, adsorption recovery is considered to be an effective way for its low cost and excellent efficiency [10]. Thus, it's imperative to develop ideal adsorbents with high adsorption capacity and excellent recyclability.

In recent decades, superhydrophobic materials with surface water contact angle (WCA) greater than 150° are the most attractive candidate for xylene adsorption and xylene – water separation [11]. Generally, the substrate used for fabricating a superhydrophobic surface can be divided into two types. One type is mesh material such as copper [12], stainless steel [13], glass [14], and so on. The other type is porous material like polyurethane sponge [15] and polyurethane foam [16]. Among them, sponge material with a three-dimensional network structure and sufficient storage space has attracted considerable attention [17,18]. As an

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inexpensive, commercially available, and porous material, sponge material is the most appropriate substrate for superhydrophobic modification. However, traditional methods used to fabricate superhydrophobic surfaces on sponge are either high cost or complicated. Whereas researches about superhydrophobic film or coating mainly focus on their preparation process. Thus, the reports about how to develop facile and low-cost methods to fabricate superhydrophobic coating on sponge are few up to now.

In this work, superhydrophobic silica-based polymer coating was developed to modify melamine sponge (NM-sponge) by a facile method. More specifically, inexpensive engineering material polyvinyl chloride (PVC) was adopted. And modified SiO_2 nanoparticles were also used to enhance the hydrophobic property. The modified NM-sponge was applied as an adsorbent for xylene adsorption and xylene – water separation.

2. Experimental

2.1. Materials

Melamine sponge (density 0.013 g/cm^3, porosity 97.5%). Polyvinyl chloride (PVC, *K*-value 59-55), hydrophobic SiO_2 particles (n-SiO_2, Hydrophobic-115), and 3-aminopropyltriethoxysilane (KH 550) were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd., (Shanghai, China). Ethanol, ethyl acetate, Sudan III, and tetrahydrofuran (THF) were analytically pure and purchased from Sinopharm Chemical Reagent Co., Ltd., (China). Meta-xylene, ortho-xylene, paraxylene, benzene, styrene, and ethylbenzene (Sinopharm Chemical Reagent Corporation, China, analytical grade) was used as the adsorbate. Gasoline and diesel oil were purchased from Sinopec Gas Station. Deionized water was prepared by Exo-Pure Water Machine Exceed-Ba, and the water conductivity was 1.34 μs/cm.

2.2. Sample preparation

NM-sponge was cut into cubes with a size of 2 cm^3 × 2 cm^3 × 2 cm^3. The cubes were first washed repeatedly by pure ethanol in an ultrasonic cleaning machine (E100H, Elma, German), and they were thoroughly washed with deionized water. After washing, the cubes were dried in an oven for at least 24 h.

Preparation of SiO_2-NM (Fig. 1): 0.1 g n-SiO_2 was dissolved in 20 mL ethyl acetate by ultrasonically dispersing at room temperature. The dried cubes were impregnated with this aqueous solution for at least 12 h. KH550 (1 wt.%) dissolved in ethyl acetate was then slowly added into the mixture. After 12 h, the samples were taken out to centrifuge to obtain SiO_2-NM samples. The samples were then dried at 60°C for at least 6 h.

Preparation of PVC/SiO_2-NM: 0.1 g PVC was dissolved in 10 mL THF, then 0.05 g n-SiO_2 was added into the solution. After being stirred vigorously for 30 min, PVC (1 wt.%) dissolved in KH550 was then added into the mixture. The mixture solution was then dispersed by an ultrasonic dispersion instrument. Then the cubes were immersed in the mixture solution for 12 h. After that, the samples were then centrifuged and dried with the same process.

2.3. Characterization

The morphologies of the sponge samples were characterized by a field emission scanning electron microscopy (SEM, Phenom G5 Pro, Holland). The accelerating voltage was set to 10 kV. The sponge was cut into a certain thickness and fixed on the sample table. The surface wettability of the samples was measured by a contact angle system (OCA20, Dataphysics, Germany), and operated with a water drop volume of 5 μL. The measurements of the samples were carried out on a horizontal surface. And the final values were acquired using the average of five measurements at different positions. Infrared analysis of the sponge samples was performed on a NEXUS FTIR spectrometer. Instron 5967-E2 was applied to characterize the mechanical properties of the sponge samples.

2.4. Adsorption performance

Sponge samples were cut into cubes with a size of 2 cm^3 × 2 cm^3× 2 cm^3. The initial mass M_1 was recorded before oil/xylene absorption at room temperature. The sponge samples were then immersed in organic solvents or oil. After adsorption saturation, the samples were removed out from the solution, held for 5s before weight measurement (M_2). The adsorption capacity Q can be calculated by the following formula:

$$Q(g/g) = \frac{M_2 - M_1}{M_1} \tag{1}$$

where M_2 (g) and M_1 (g) were the mass of the saturated and initial sponge samples, respectively.

The cycling stability of the sponge samples is an important factor for adsorption performance. After adsorption, the organic solvents adsorbed in the sponge samples were discharged by a simple mechanical squeezing method, then the samples were weighed and reused for subsequent sorption tests. And each saturation adsorption and desorption was taken as one cycle. The adsorption capacities after several cycles were recorded to evaluate the recyclability of the sponge samples under different conditions.

2.5. Xylene – water separation performance

To examine the removal performance of sponge samples for xylene on the water surface, 2 ml xylene was dropped on the top of deionized water. And for convenience Sudan III was applied to dye xylene. The sponge samples were placed on the surface of the mixture solution, and the separation process was observed and recorded.

To verify the separation performance of xylene and water, a gravity-driven experiment was performed. The sponge sample was fixed in the front of a syringe, which was mounted at 45°. And the xylene/water mixture solution (with a volume ratio of 1:1) was poured in from the end of the syringe. The water sample filtered through the sponge sample was collected, and xylene concentrations of which were determined by gas chromatography (Model 7890B, Agilent, USA). To further evaluate the performance of the sponge sample in xylene/water separation, the separation efficiency was calculated using the oil rejection coefficient ($R(\%)$) according to:

$$R(\%) = \frac{C_2 - C_1}{C_2} \times 100\% \tag{2}$$

where C_2 was the xylene concentration of the original xylene/water mixture and C_1 was the xylene concentration of the collected water after separation.

To study the separation performance of the sponge samples for dissolved xylene, the xylene solution (150 mg/L) were prepared. After the sponge samples were immersed in the mixture, the solution was stirred continuously at room temperature. The concentrations of the solution before and after adsorption were determined by gas chromatography.

3. Results and discussion

The microscopic morphology photos of raw commercial sponge samples and modified sponge samples are shown in Fig. 2. It can be seen that the commercial sponge has a hierarchical porous structure with a smooth surface (Fig. 2a). After being modified, the skeleton and pore structure of the sponge retained its original morphology. However, the surfaces of the sponge become rougher after modification (Figs. 2b and c), which is beneficial to form a superhydrophobic surface [19,20]. As shown in Fig. 2b modified SiO_2 nanoparticles located far from each other on the surface of SiO_2-NM, and the gap between nanoparticles may be occupied with air bubbles [21]. Thus, this surface structure may prevent drop penetration and movement, which indicates that continuous superhydrophobic film may not be obtained [22]. From Fig. 2c, it can be observed that the surface of PVC/SiO_2-NM is relatively flat, compact, and homogeneous, implying excellent hydrophobicity [23,24].

Wettability is a critical property for a superhydrophobic surface, and the WCA is the key criterion for wettability [25–27]. Fig. 3 shows the WCA of sponge samples before and after modification. It can be seen that the commercial sponge is hydrophilic, the WCA of which is almost zero. After modification, the WCA value of SiO_2-NM is 145.8°. And the contact angle of PVC/SiO_2-NM is 150°, showing the superhydrophobic characteristic [28]. It can be inferred that the superhydrophobic property of the sponge samples was improved after modification by PVC and n-SiO_2. This may because the roughness and the low-free-energy surface of sponge samples were fabricated by the PVC/SiO_2 compound, which provides the capacity of water-repelling [29].

IR spectra of the sponge samples are depicted in Fig. 4. Characteristic peaks of melamine sponge can be seen from Fig. 4a, such as the stretching vibration peak of N–H at 3,354 cm^{-1}, the absorption peak of C=N at 1,487 cm^{-1}, and the bending vibration peak of thiotriazinone at 812 and 1,341 cm^{-1} [30,31]. The peak exists at 1,547 cm^{-1} can be attributed to O–H stretching vibration. The peak at 1,126 and 1,033 cm^{-1} can be assigned to C–O–C group vibration, which indicates the existence of the ester group. In Fig. 4b, the less pronounced peaks can be observed at 812, 1,341 and 1,487 cm^{-1}, implying the PVC film had been coated on the surface of the sponge samples. The characteristic peak of O–Si–O can be seen at 1,082 cm^{-1}, indicating hydrophobic SiO_2 particles were coated on the surface of the sponge samples [32]. In addition, A weak peak at 1,547 cm^{-1} indicates that less hydrophilic oxygen-containing groups existed on the surface of PVC/SiO_2-NM.

In addition to hydrophobic properties, the mechanical property is also one of the most significant factors that affect adsorption performance. The main parameters are listed in Table 1. From the value of maximum load and tensile elongation, it can be inferred that the mechanical property of commercial sponge has been improved effectively after modification.

Adsorption capacity and reusability are also important factors for adsorbents applied in xylene – water separation [33]. The saturated adsorption capacities of PVC/SiO_2-NM for various organic chemicals are shown in Fig. 5a. It can be observed that PVC/SiO_2-NM exhibits a good adsorption for various organic solvents, such as meta-xylene (41.45 g/g), ortho-xylene (44.55 g/g), paraxylene (36.03 g/g), benzene

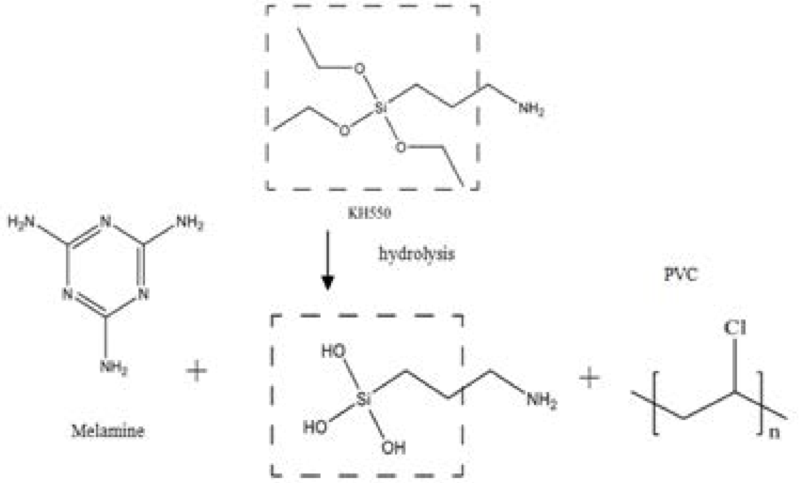

Fig. 1. Schematic illustration of the preparation process of PVC/SiO_2-NM.

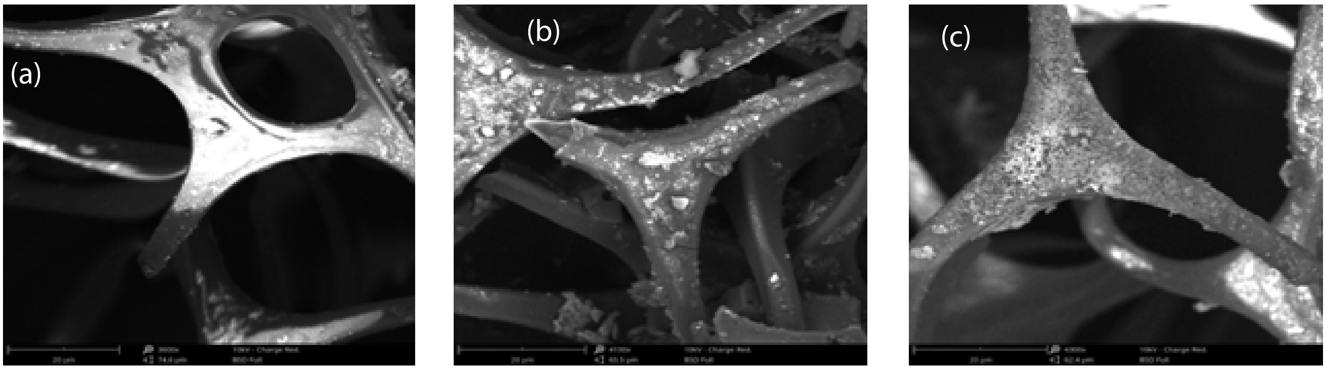

Fig. 2. SEM images of the sponge samples (a) commercial sponge, (b) SiO_2-NM, and (c) PVC/SiO_2-NM.

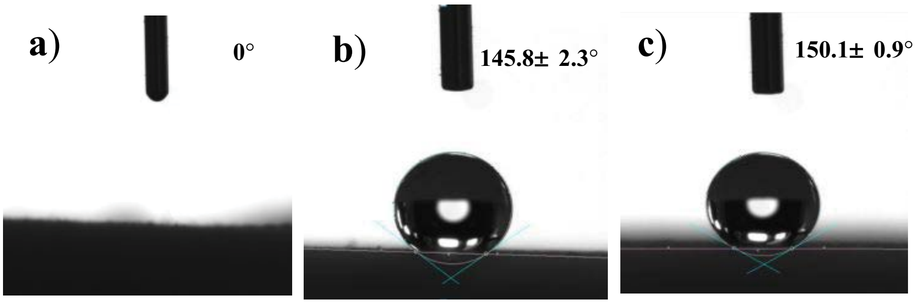

Fig. 3. Water contact angles of sponge samples (a) commercial sponge, (b) SiO_2-NM, and (c) PVC/SiO_2-NM.

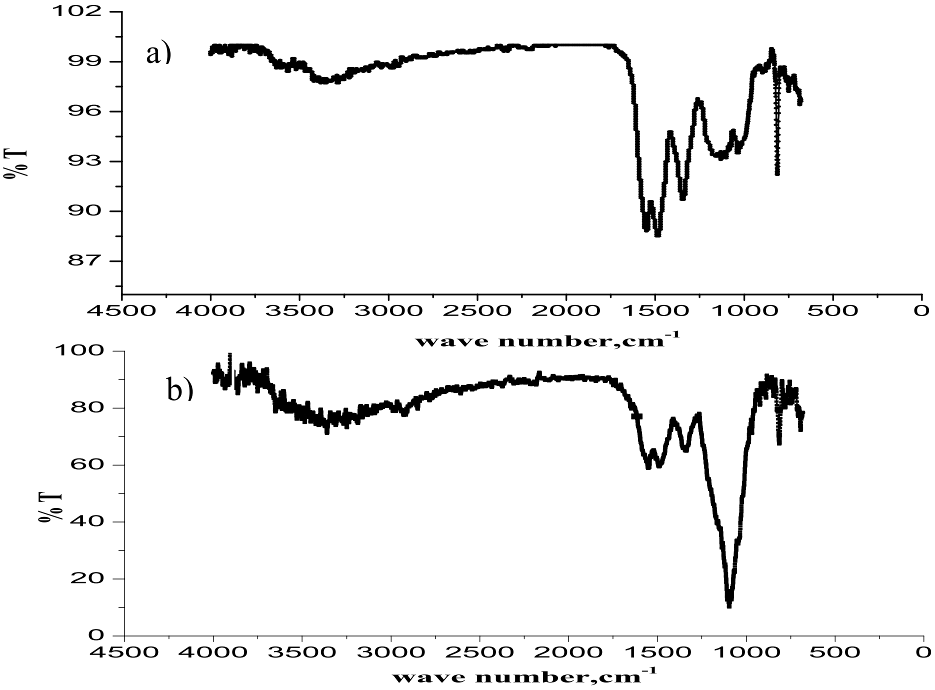

Fig. 4. Infrared spectra of (a) commercial sponge and (b) PVC/SiO_2-NM.

(46.33 g/g), styrene (37.27 g/g), ethylbenzene (34 g/g), gasoline (33.27 g/g), diesel oil (31.54 g/g). This may due to the fact that PVC/SiO_2-NM has high porosity (97.5%) and low density (13 kg/m^3). The recyclability of PVC/SiO_2-NM was tested for the removal of meta-xylene, benzene, and diesel oil. The durability of PVC/SiO_2-NM under harsh conditions was also investigated. As shown in Fig. 5b, the xylene adsorption capacity of PVC/SiO_2-NM remained 93% of its

Table 1
Mechanical properties of the sponge samples

Samples	Commercial sponge	PVC/SiO_2-NM
Width (mm)	20.00	20.00
Thickness (mm)	20.80	20.80
Maximum load (N)	55.80	57.21
Pressure (N mm^{-2})	0.128	0.136
Tensile elongation (%)	114.09	118.02

initial adsorption capacity even after 500 cycles, which may be attributed to the strong mechanical strength, high porosity, and high elasticity of PVC/SiO_2-NM [34].

As shown in Fig. 6a, PVC/SiO_2-NM can float above water under surface tension, due to its hydrophobic nature, whereas the untreated sponge sank beneath the water surface after being placed on water. This may because PVC/SiO_2-NM exhibited a mirror-like, water-repelling surface when immersed into water, and water can not penetrate into PVC/SiO_2-NM. After the external force was released, the sponge instantaneously floated onto the surface of the water. Thus, organic pollutants (such as xylene) floating on the water can be absorbed quickly by PVC/SiO_2-NM, for the superhydrophobic property and the capillary effect [35].

The xylene – water separation performance of PVC/SiO_2-NM was also examined. As exhibited in Fig. 6c, 5 mL of xylene (dyed by Sudan III) was poured onto the surface of 10 ml water to prepare xylene – water interface. And once a piece of PVC/SiO_2-NM touched the xylene layer, it completely adsorbed the xylene within 10 s, resulting in a transparent water region. After staying for another 5 s on the surface of the water, PVC/SiO_2-NM was removed out, and the water still remains clear without contamination. This indicates that PVC/SiO_2-NM can be applied as a promising adsorbent for xylene leakage recovery.

To verify the separation performance of xylene and water, a piece of sponge sample was fitted in the front of a syringe as shown in Fig. 6d. With sponge used as a filter, the gravity-driven separation apparatus was designed and build up. The experiments were conducted in Figs. 6e and f. 10 mL mixture solution of xylene/water (v/v = 1:1) was poured into the syringe. Fig. 6e shows that xylene (dyed by Sudan III) quickly passed through PVC/SiO_2-NM and flowed down by gravity, while the water layer was retained in the syringe. And the removal rate can achieve 99.99%. However, both the xylene layer and water layer were not repelled and flowed through the unmodified commercial sponge shown by Fig. 6f.

To investigate the separation capacity of PVC/SiO_2-NM for dissolved xylene, 150 mg/L xylene solution was prepared in sealed containers. PVC/SiO_2-NM and unmodified commercial sponge were both applied for dissolved xylene adsorption. The adsorption curves are shown in Fig. 7. It can be observed that the concentration of xylene in solution reduced to 138.15 mg/L for evaporation. The concentration of xylene in solution was 91.22 mg/L after adsorption by commercial sponge for 24 h, and the removal rate was 39.19%. While PVC/SiO_2-NM had the best removal

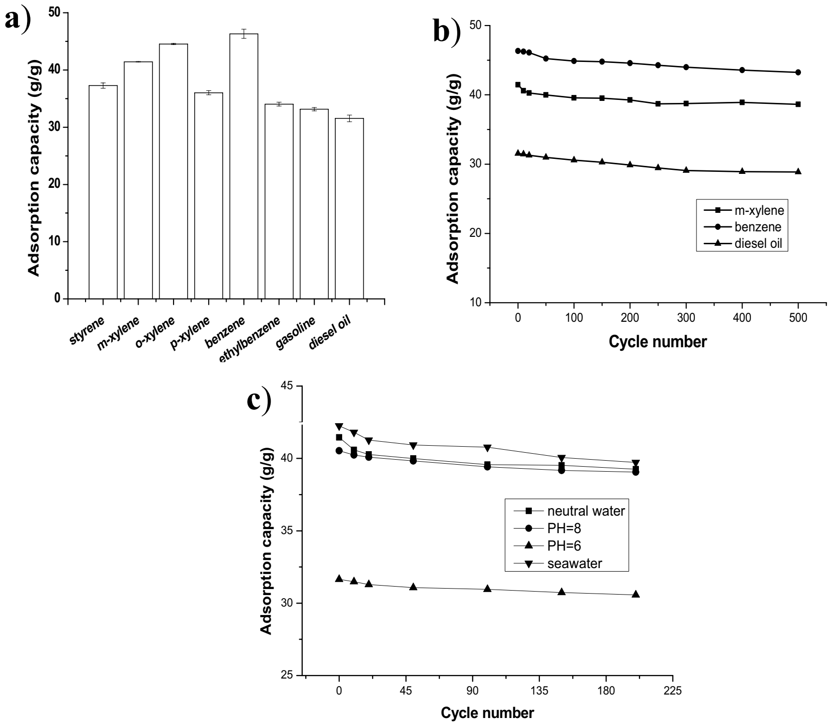

Fig. 5. (a) Sorption capacities of PVC/SiO_2-NM for various organic solvents, (b) cycling stability of PVC/SiO_2-NM, and (c) durability of PVC/SiO_2-NM under harsh conditions

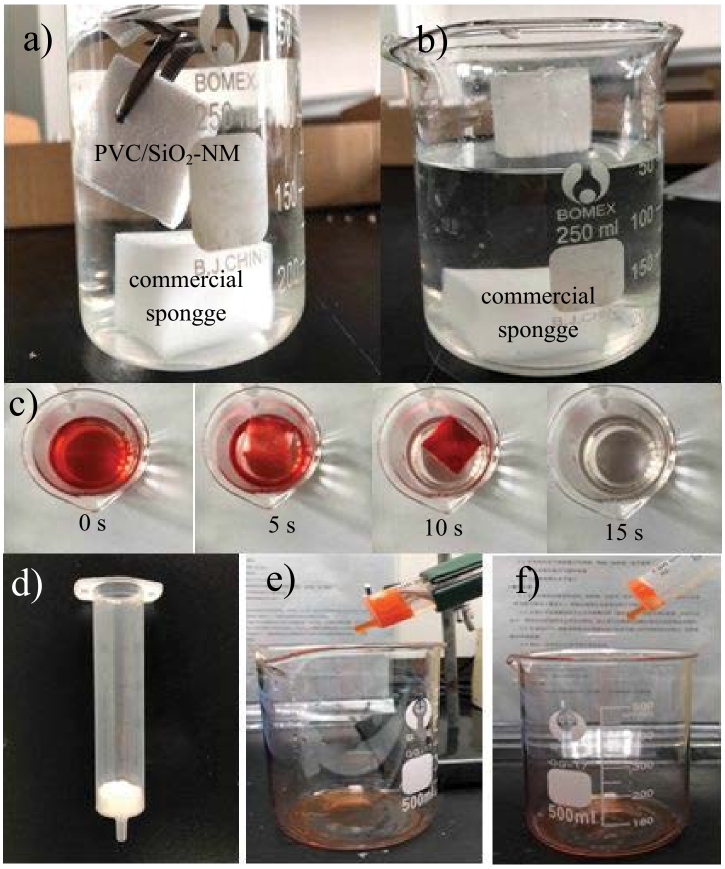

Fig. 6. Photographs of (a) PVC/SiO_2-NM under force in the water, (b) floating PVC/SiO_2-NM free of force, (c) removal process of xylene by using PVC/SiO_2-NM, (d) syringe with PVC/SiO_2-NM fixed in the front, (e) gravity-driven separation apparatus with PVC/SiO_2-NM, and (f) gravity-driven separation apparatus with a commercial sponge.

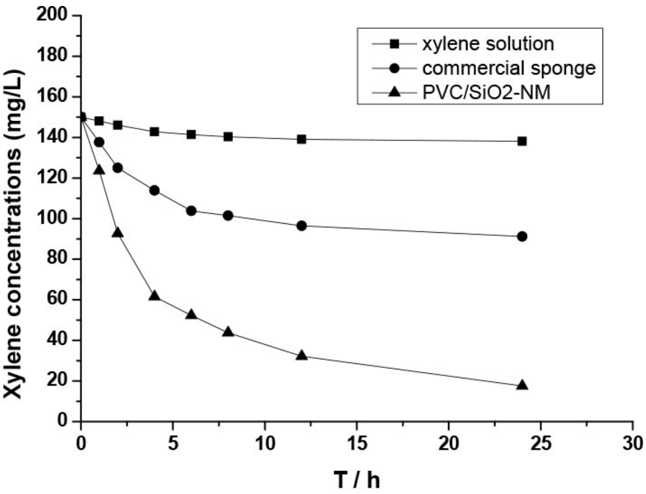

Fig. 7. Adsorption curves for toluene over different absorbents.

efficiency for xylene. After adsorption for 24 h, the xylene concentration decreased to 17.52 mg/L, and the removal rate achieved 88.32%. This implies the excellent dissolved xylene removal ability of PVC/SiO_2-NM. Thus, PVC/SiO_2-NM can not only be applied in the rapid separation of xylene – water mixture, but also for xylene adsorption.

The specific adsorption of PVC/SiO_2-NM for organic solvents may be related to its surface wettability and spontaneous infiltration process. Spontaneous infiltration process is a process in which a porous medium spontaneously inhales a certain wetting liquid driven by capillary force [36]. The sponge has a porous structure, and each pore channel can be approximated as a capillary tube. After superhydrophobic modification, the sponge is more easily wetted by oil substances, such as gasoline, xylene, benzene, and so on. Thus oil substances are more likely to enter the capillary tube and replace the non-wetting fluid (water molecules) [37,38]. In addition, the roughness of the modified sponge is increased, so that the capillary action is enhanced. Therefore, PVC/SiO_2-NM can adsorb oil substances selectively without adsorbing water. It can be inferred that PVC/SiO_2-NM has great application potential in the field of xylene/water separation and water purification.

4. Conclusion

A novel super-hydrophobic PVC/SiO_2-NM sponge was successfully fabricated by a facile, inexpensive method. The PVC/SiO_2-NM sponge reveals excellent super-hydrophobic property with a WCA above 150°. During modification, PVC not only provides attachment sites for SiO_2 nanoparticles but also polymerized onto the skeleton of melamine sponge. The modified PVC/SiO_2-NM sponge exhibited outstanding xylene adsorption capacity (41.45 g/g), superhydrophobic stability, excellent reusability (the adsorption capacity can achieve 93% of its initial capacity after 500 cycles), and high selective adsorption performance (the xylene – water separation efficiency can reach 99%). Due to its property of high selective adsorption and excellent reusability, PVC/SiO_2-NM sponge can be applied as a potential candidate for xylene leakage recovery.

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