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Effect of template in MCM-41 on the adsorption of aniline from aqueous solution

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ABSTRACT

The effect of the surfactant template cetyltrimethylammonium bromide (CTAB) in MCM-41 on the adsorption of aniline was investigated. Various MCM-41 samples were prepared by controlling template removal using an extraction method. The samples were then used as adsorbents for the removal of aniline from aqueous solution. The results showed that the MCM-41 samples with the template partially removed (denoted as C-MCM-41) exhibited better adsorption performance than MCM-41 with the template completely removed (denoted as MCM-41). The reason for this difference may be that the C-MCM-41 samples had stronger hydrophobic properties and selectivity for aniline because of the presence of the template. The porosity and cationic sites generated by the template play an important role in the adsorption process. The optimal adsorbent with moderate template was achieved by changing the ratio of extractant; it has the potential for promising applications in the field of water pollution control.

1. Introduction

The control of environmental pollution is becoming increasingly important. Many international and national laws have been passed that require a reduction in the emission of volatile organic compounds (VOCs) (Martin et al., 2008). Volatile organic chemical pollutants in water threaten animal and plant life, including humans (Jones, 1999). Aniline is widely used in the manufacture of plastic, paint, pesticides, dyes and intermediates in the chemical synthesis industries (Gürtena et al., 2005). Wastewater containing aniline has caused many serious environmental problems because of its high toxicity and carcinogenic properties. A number of technologies are available for removing aniline, such as adsorption (Cai et al., 2005; Gürtena et al., 2005; Podkoscielny and Laszlo, 2007; Huang et al., 2008), degradation (Gheewala and Annachhatre, 1997; Song et al., 2007; Wang et al., 2007), incineration, and catalytic reaction. Adsorption is a widely used reliable chemical engineering method because of the flexibility of the system, low energy consumption and low operating costs. Many studies have been devoted to researching the adsorption mechanism and identifying suitable adsorbents (Unuabonaha et al., 2008). Activated carbon, one of the adsorption technologies available, has been extensively used in industrial processes (Ayranci and Hoda, 2005; Podkoscielny and Laszlo, 2007; Tseng et al., 2010). However, activated carbon presents several disadvantages, such as its limited adsorptive capacity, its hydrophilic nature, and problems associated with regeneration. Therefore, it is necessary to develop a novel and efficient material with considerable adsorption capacity, high adsorption rate and stable performance is needed.

The mesoporous molecular sieve MCM-41, a member of the M41S family, exhibits hexagonal arrays of uniform channels (1.5–10 nm in diameter), a high Brunauer–Emmett–Teller (BET) surface area (larger than 800 m^2/g) and large pore volume (larger than 0.9 cm^3/g). Therefore, the MCM-41 has potential applications in the fields of adsorption, catalysis, separation and environmental pollution control (Climent et al., 1996; Carvalho et al., 1999; Juang et al., 2006; Mehraban and Farzaneh, 2006; Qin et al., 2007; Puanngama and Unob, 2008). Most importantly, the surface chemistry and pore openings of MCM-41 can be tailored by post synthesis to meet various requirements (Zhao and Lu, 1998; Zhao et al., 1998; Lang et al., 2002; Horcajada et al., 2006).

Before and after modification, MCM-41 can be widely used in the field of adsorption. Eimer et al. studied the interactions of aniline adsorbed onto MCM-41 mesoporous material by thermal and Fourier transform infrared spectroscopic analysis (Eimer et al., 2003). Mangrulkar et al. reported that MCM-41 with surfactant template is an effective adsorbent for the removal of phenol and o-chlorophenol from aqueous solution (Mangrulkar et al., 2008). The reason for this may be that the existence of a cationic template within the framework of the MCM-41 materials causes changes in the surface chemistry and porosity of the adsorbent, which in turn affects the adsorption behavior of the adsorbent. In addition, Huang

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et al. investigated the effect of the cationic template on the adsorption of toluene and cumene on MCM-41 (Huang et al., 2007). The results showed that the samples with the template partially removed exhibited moderate adsorption capacity for toluene and cumene, and lower adsorption affinity for water. These studies clearly indicate that the surfactant template plays an important part in the adsorption process because of its strong hydrophobic properties and that it could improve the adsorption capacity of organic compounds.

In the present work, the primary MCM-41 was synthesized using a hydrothermal method and other MCM-41 samples were prepared by controlling the template removal by an extraction method. The optimal adsorbent with moderate template was achieved by varying the ratio of extractant. The adsorption of aniline from aqueous solution on various MCM-41 samples was investigated systematically using batch experiments. The effects of various parameters such as contact time, initial aniline concentration, temperature and pH on the removal of aniline were studied in detail. The adsorption isotherm was analyzed using the Langmuir and Freundlich adsorption model. Finally, the regeneration of adsorbent was also investigated.

2. Experimental

2.1. Materials

Sodium silicate (SiO_2; 27%) and n-cetyltrimethylammonium bromide (CTAB; 99%) were purchased from Tianjin Chemist Scientific Ltd, China. Aniline, ethanol (EtOH), hydrochloric acid (HCl), sodium hydroxide (NaOH) and activated carbon (coconut-made charcoal) were purchased from Tianjin Jiangtian Chemical Technology Ltd, China. All reagents used in this study were analytical reagent grade.

2.2. Synthesis of adsorbents

Primary MCM-41 samples were synthesized by a hydrothermal method as described previously (Beck et al., 1992). MCM-41 was crystallized from a gel composed of SiO_2: 0.64 Na_2O: 0.49 CTAB: 52H_2O in a Teflon autoclave at 393 K for 12 h under autogenous pressure. After cooling to room temperature, the solid product was recovered by filtration, washed thoroughly with deionized water, and dried in an oven overnight at 373 K. The as-synthesized sample was designated MCM-41-AS. The sample from which the template had been removed completely was designated MCM-41. The sample with cationic modification of MCM-41 was designated C-MCM-41 which was obtained through partial template removal by a solvent extraction method (Chen et al., 1995). The process was as follows: The MCM-41-AS sample was suspended and refluxed in 1 M HCl in EtOH at 348 K for 4 h, then filtered, washed with EtOH and dried at 373 K for 10 h under vacuum. Five different ratios of MCM-41-AS/extractant were used in the solvent extraction: (1) 1 g MCM-41-AS/250 ml extractant (designated C-MCM-41-1); (2) 2 g MCM-41-AS/250 ml extractant (designated C-MCM-41-2); (3) 3 g MCM-41-AS/250 ml extractant (designated C-MCM-41-3); (4) 4 g MCM-41-AS/250 ml extractant (designated C-MCM-41-4); and (5) 5 g MCM-41-AS/250 ml extractant (designated C-MCM-41-5) Scheme 1.

2.3. Characterization

Powder x-ray diffraction (XRD) was collected on a Bruker D8 focus diffractometer, with Cu Kα at 40 kV and 40 mA between 0.6° and 8° (2θ) with a step length of 1 s. Nitrogen adsorption–desorption isotherms of samples at 77 K were measured with a BEL-MINI adsorption analyzer. The surface area was calculated using a multipoint Brunauer–Emmett–Teller (BET) model. The total pore volume was estimated at a relative pressure of 0.99, assuming full surface saturation with nitrogen.

2.4. Batch adsorption experiments

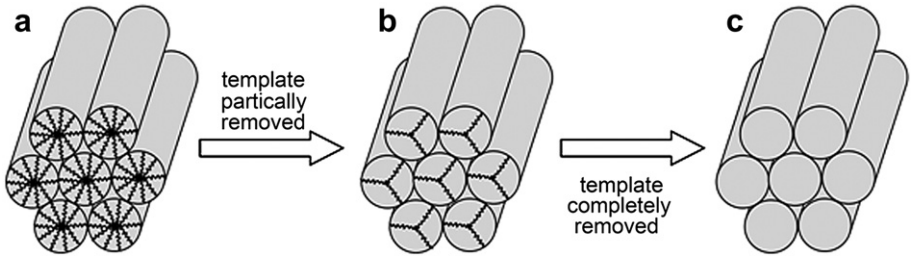
The aniline adsorption data from aqueous solution were obtained using the immersion method. The stock solution of aniline was prepared in distilled water. The experiments were performed in a temperature-controlled water bath shaker for a certain time at a mixing speed of 180 rpm. The effects of contact time, initial aniline concentration, temperature and pH on adsorption data were assessed by changing the adsorption conditions. The initial pH of the solution was adjusted with HCl or NaOH solution to reach a desirable value (1–13). Finally, the regeneration of adsorbent was also investigated. The regeneration method was that the adsorbent that had adsorbed aniline was washed in a large volume of water until there was no aniline in aqueous solution, and then dried in vacuum condition at 353 K for 10 h. At the end of the adsorption process, the adsorbent was filtered. Then, the residual concentration of aniline was determined using a UV-spectrophotometer (TU1900) at a wavelength of 280 nm. The percentage removal of aniline (%) was calculated according to the following equation:

$$R = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

The adsorption amount (Q_e) was calculated according to the following equation:

$$Q_e = \frac{V(C_0 - C_e)}{m} \times 100\% \quad (2)$$

where, Q_e (mg g^{-1}) represents the adsorption amount, V (L) is the volume of the aniline solution, C_0 (mg/L) and C_e (mg/L) are the initial and equilibrium liquid-phase concentration of aniline,



Scheme 1. Illustration of (a) MCM-41-AS, (b) MCM-41-C and (c) MCM-41.

respectively, and m (g) is the mass of the absorbent used in the experiments.

3. Results and discussion

3.1. Characterization of the adsorbents

The small-angle XRD patterns for the various MCM-41 samples are shown in Fig. S1 Hexagonally arranged channels, represented by (100), (110) and (200) reflection, were present in the samples. The sample MCM-41 had the highest reflection intensity. As the amount of template in the framework of MCM-41 increased, the reflection intensity has somewhat decreased. This may be attributed to little shape torts in the channels caused by the presence of partial template. However the framework of the sample was not changed and the porous channels remained in a hexagonal array. The sample MCM-41-AS had the lowest reflection intensity because the pore channel was filled with template.

The nitrogen adsorption/desorption isotherms of various MCM-41 samples and activated carbon are shown in Fig. S2. The porous channels of MCM-41-AS were filled with CTAB template. The nitrogen adsorption/desorption isotherm for MCM-41-AS was distinctly different from the other samples. The MCM-41 and C-MCM-41-n samples had a type IV isotherm with a capillary condensation step, which is characteristic of mesoporous materials according to the classification of the International Union of Pure and Applied Chemistry. The parameters calculated from the nitrogen adsorption/desorption data are listed in Table S1. We found that as the amount of template in MCM-41 increased, the BET surface area and total pore volume has somewhat decreased, but there was no significant change. The reason may be that the partial template CTAB in the framework of MCM-41 occupied some space.

3.2. Adsorption properties for aniline

3.2.1. The adsorption performance of different adsorbents

The adsorption performance of different adsorbents was studied and is shown in Fig. 1. To investigate systematically the adsorption performance of MCM-41, the adsorption of aniline on activated carbon was studied for comparison. The adsorption of aniline on MCM-41 and C-MCM-41 was significantly higher than that on MCM-41-AS and activated carbon. In the initial stage, the percentage removal of aniline increased with the increasing of the template content in MCM-41. The optimal adsorbent was achieved when the ratio of MCM-41-AS/extractant was 3 g/250 ml, and the percentage removal of aniline on C-MCM-41-3 was the highest. When the ratio of MCM-41-AS/extractant went beyond 3 g/250 ml, the percentage removal of aniline decreased. This may be caused by the BET surface and the CTAB template playing an important role in the process of aniline adsorption onto MCM-41. This can be explained as follows. The porous channels of MCM-41-AS were filled with template, and the BET surface area was very low; therefore, the adsorption performance was poor. The MCM-41 sample had the largest BET surface area and total pore volume; however, it had many −OH groups at the pore surface and many Si−O−Si bonds in its framework, which have some hydroscopic properties and limited selectivity to aniline. In the C-MCM-41-n samples, cationic sites generated by the template could simultaneously improve the hydrophobic property of the adsorbent and selectivity for aniline. The optimum adsorbent should have moderate porosity and template, the sample C-MCM-41-3 is a more favorable adsorbent for the removal of aniline from aqueous solution.

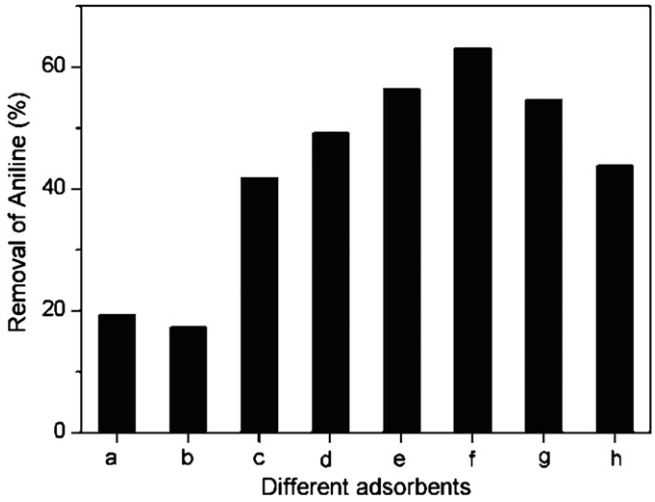


Fig. 1. Kinetic study of aniline on MCM-41-AS, MCM-41 and C-MCM-41-3 (adsorption conditions: initial aniline concentration = 50 mg/L, adsorbent dose = 4 g/L, pH = 7, and temperature = 298 K).

3.2.2. Aniline adsorption kinetics

The kinetics of aniline adsorption on MCM-41-AS, MCM-41 and C-MCM-41-3 samples were carried out when the initial aniline concentration was 50 mg/L (Fig. 2). As illustrated, the apparent adsorption equilibrium was usually established within 60 min. No significant change in the percentage removal of aniline was observed after 2 h. This is a much faster adsorption rate than that reported for other adsorbents (An et al., 2010). It was assumed that the adsorption process included two kinetic behaviors: a rapid initial stage and a slower second stage. The first stage was the instantaneous adsorption stage or external surface adsorption. The second stage was the gradual adsorption stage, and finally the aniline uptake reached equilibrium (Wu et al., 2009). Additionally, the percentage removal of aniline on C-MCM-41-3 at adsorption equilibrium was much higher than that of MCM-41-AS and MCM-41. This can be attributed to the porosity and cationic sites of adsorbent and is discussed in further detail below.

3.2.3. Effect of temperature on aniline adsorption

The adsorption isotherms of aniline onto C-MCM-41-3 at different temperatures (298, 308, 318 and 328 K) were studied to investigate the effect of temperature (Fig. 3). As shown, all the

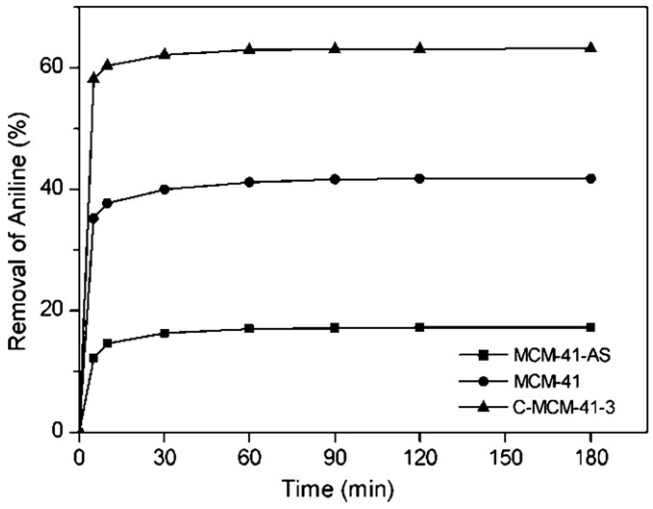


Fig. 2. The adsorption performance of (a) Activated carbon, (b) MCM-41-AS, (c) MCM-41, (d) C-MCM-41-1, (e) C-MCM-41-2, (f) C-MCM-41-3, (g) C-MCM-41-4 and (h) C-MCM-41-5 (adsorption conditions: initial aniline concentration = 50 mg/L, adsorbent dose = 4 g/L, temperature = 298 K, pH = 7, contact time = 2 h).

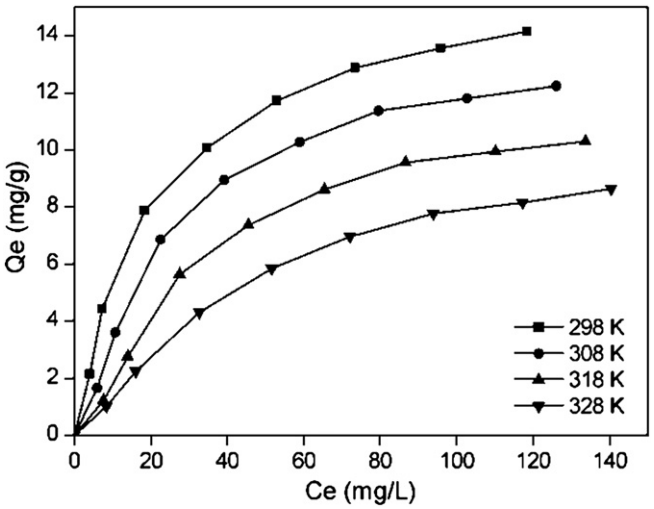


Fig. 3. Adsorption isotherm of aniline onto C-MCM-41-3 at different temperatures (adsorption conditions: adsorbent dose = 4 g/L, pH = 7, contact time = 2 h).

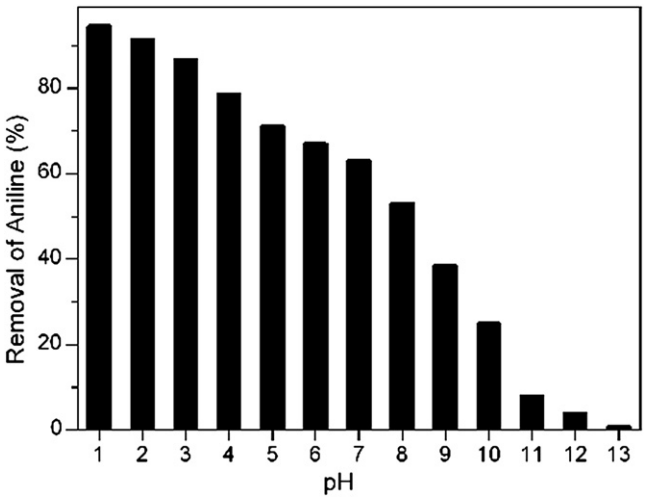


Fig. 4. Effect of pH on the adsorption of aniline onto C-MCM-41-3 (adsorption conditions: initial aniline concentration = 50 mg/L, temperature = 298 K, Adsorbent dose = 4 g/L, contact time = 2 h).

adsorption isotherms were nonlinear with curves concave to the abscissa.

To describe the adsorption isotherm in more detail, the Langmuir and Freundlich model equations were selected for use in this study. The Langmuir adsorption isotherm has been successfully applied to many pollutant adsorption processes from aqueous solution. It is commonly represented as:

$$Q_e = \frac{Q_0 K_L C_e}{1 + K_L C_e} \quad (3)$$

where, Q_e represents the equilibrium adsorption capacity of aniline on the adsorbent (mg/g), C_e is the equilibrium aniline concentration in solution (mg/L), Q_0 is the maximum monolayer capacity of adsorbent (mg/g) and K_L is the Langmuir adsorption constant (l/mg) related to the free energy of adsorption. The Freundlich isotherm is an empirical equation describing adsorption onto a heterogeneous surface. The common form is:

$$Q_e = K_F C_e^{\frac{1}{n}} \quad (4)$$

where, K_F [mg/g(L/mg)$^{1/n}$] and 1/n are the Freundlich constants corresponding to adsorption capacity and adsorption intensity, respectively. The Langmuir and Freundlich model parameters and linear regression correlations obtained from the fitting curves are given in Table S2. It can be seen that linear regression correlations using the Langmuir model were higher than those for the Freundlich isotherm for aniline, which suggested that the Langmuir model better describes the adsorption of aniline onto C-MCM-41-3. These results indicated that the adsorption of aniline onto C-MCM-41-3 was a typical monomolecular-layer adsorption. Moreover, the values for 1/n from the Freundlich model were all less than 1, which is indicative of high adsorption intensity between aniline and the adsorbent. The monolayer capacity Q_0 of aniline onto C-MCM-41-3 at 298 K (16.64 mg/g), 308 K (15.69 mg/g), 318 K (14.50 mg/g) and 328 K (13.68 mg/g), respectively, was calculated based on the Langmuir isotherm. As expected, the maximum adsorptive capacity of aniline onto C-MCM-41-3 decreased with the increase in temperature, indicating that the adsorption process was a exothermic process. According to the Van't Hoff equation:

$$\ln\frac{Q_e}{C_e} = -\frac{\Delta H}{RT} + C \quad (5)$$

When the adsorptive capacity was 8.0 mg/g, the curve of the ln (Q_e/C_e) vs. (1/T) was as shown in Fig. S3. The enthalpy change, ΔH, was −46.18 kJ mol^{-1}, which theoretically indicates that the adsorption of aniline onto C-MCM-41-3 is an exothermic process.

3.2.4. Effect of pH on aniline adsorption

In practical industrial processes, wastewater may be acidic or alkali and may contain many contaminants besides aniline. To investigate the effect of pH on the percentage removal of aniline, aqueous solutions were prepared with different pH values ranging from 1 to 13. As illustrated in Fig. 4, the pH value obviously had a considerable influence on the adsorption performance of C-MCM-41-3. The percentage removal of aniline decreased with an increase in pH value. At pH = 1, the percentage removal of aniline was the highest (94.52%), whereas at pH = 13, the percentage removal of aniline was only 0.71%. In the process of adsorption of aniline onto C-MCM-41-3, two kinds of hydrogen bonding can take place. First, the −OH of C-MCM-41-3 could form a hydrogen bond (O−H N hydrogen bond) with the N atom of aniline that acts as the acceptor. Second, the $-NH_2$ of aniline could form a hydrogen bond (N−H…O

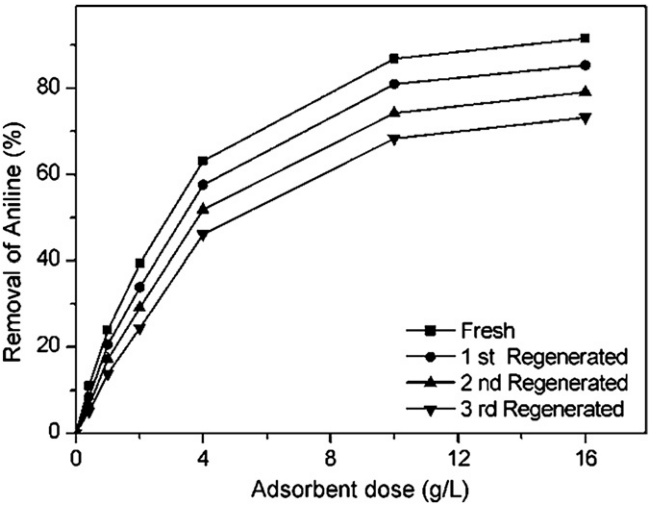


Fig. 5. Regenerated use of C-MCM-41-3 adsorbent for the removal of aniline (adsorption conditions: initial aniline concentration = 50 mg/L, temperature = 298 K, pH = 7, contact time = 2 h).

hydrogen bond) with the O atom in C-MCM-41-3 (Wu et al., 2009). At pH values below 7, there are many HCl molecules in the aqueous solution, aniline is a mild base that could interact with HCl and form $C_6H_5NH_3^+Cl^{--}$. Compared with $C_6H_5NH_2$, $C_6H_5NH_3^+$ is electron deficient and has three hydrogen atoms. Therefore, it could form stronger hydrogen bonds with C-MCM-41-3. A possible explanation for the sharp decrease in the percentage removal of aniline with an increase in pH may be that C-MCM-41-3 material is relatively stable with a high acid resistance, whereas it degrades readily in a basic solution that decreases its hydrophobic characteristics and destroys the structure of C-MCM-41-3 (Selvam et al., 2001). In addition, we characterized the C-MCM-41-3 sample that was used in acid and alkali aniline solution by XRD. The result showed that the structure of the C-MCM-41-3 sample in basic solution had collapsed and it had no hexagonally arranged channels.

3.2.5. Regeneration of the adsorbent

The cost of adsorbents is one of the most important factors in the adsorption industry. Therefore, the regeneration properties of C-MCM-41-3 were studied. The adsorption performance of regenerated C-MCM-41-3 was compared with fresh adsorbent (Fig. 5). After regeneration, the percentage removal of aniline had decreased slightly, and the decrease accelerated with the increasing of the regeneration time. The reason may be the decrease in template content and the deactivation of some adsorption sites. The adsorption performance of C-MCM-41-3 after the third regeneration was still higher than those of MCM-41 and activated carbon.

4. Conclusion

Various MCM-41 samples were synthesized by controlling template removal using an extraction method. The porosity and cationic sites generated by the template play an important role in the adsorption process. By changing the ratio of extractant, we prepared the optimal adsorbent, C-MCM-41-3, which showed the best adsorption performance for the removal of aniline. The XRD and BET characterization of the adsorbents indicated that C-MCM-41-3 had hexagonally arranged channels and was mesoporous molecular sieves. The adsorption performance of aniline onto C-MCM-41-3 was systematically investigated using batch adsorption experiments. The adsorption process is exothermic, and the adsorptive capacity has a tendency to decrease with temperature increase. The percentage removal of aniline was higher in acid solution and lower in alkali solution. Finally, the regenerative study showed that the adsorbent C-MCM-41-3 has excellent regeneration. The adsorbent C-MCM-41-3 has potential for promising applications in the field of water pollution control.

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Appendix. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jenvman.2011.07.006.

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