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ORIGINAL PAPER

Preparation and post-treatments of ordered mesoporous carbons (OMC) for resorcinol removal

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Abstract In this investigation, acrylic acid was used as a carbon precursor and SBA-15 as a mesoporous template to synthesize the ordered mesoporous carbons (OMC). Different ratios of SBA-15 to acrylic acid were evaluated to improve the OMCs adsorption capacity. It was found that the optimal ratio of SBA-15 to acrylic acid is 3:1. In the post-treatment study, four methods (NaOH, urea, NH_3H_2O, and $AlCl_3$) were explored to investigate their influences on resorcinol removal. Results showed the ammonium hydroxide-treated OMC had the highest adsorption capacity of 40.6 mg/g for resorcinol removal. The nano-structures of the OMC and post-treated OMCs were characterized and confirmed. FTIR analysis indicated that the surface functional groups were changed after post-treatments. XRD patterns and TEM images suggested that the ordered structure was well maintained during the post-treatment processes, although the erosion effect was observed.

Keywords Ordered mesoporous carbon · Preparation · Post-treatment · Adsorption · Resorcinol removal

Introduction

Recently, development of ordered mesoporous carbons (OMCs) has attracted much attention because they have been explored for industrial applications such as catalyst supports, electrochemical materials, and gas separation media (Guo, et al., 2013). OMCs show great potential for environmental improvement for their ability to remove inorganic and organic contaminants from liquid and gas phases. Historically, activated carbon with a large fraction of micropores (<2 nm) has been used for contaminant adsorption; the material's applications, however, can be limited by slow contaminant diffusion kinetics and the inaccessibility of the sorption sites. Ordered mesoporous carbons (OMCs), on the other hand, have high surface areas and controlled mesopores (Antonio 2004). This would result in much higher adsorption capacity and faster adsorption kinetics than regular activated carbon-based materials. They represent innovative and promising adsorption materials for use in water and wastewater treatment. More and more researchers are showing great interest in the OMC material as a replacement for granular activated carbon (GAC) used in water and waste water treatment systems.

In 1999, the first ordered mesoporous carbon was synthesized using mesoporous crystalline material 48 (MCM-48) as a template (Ryong et al. 1999). Since then, various synthesis methods of OMC have arisen. However, adsorption studies on the OMCs demonstrated that the adsorption capacity was not as good as expected (Yang and Zhao 2005). In addition, OMCs were still not selective for a specific contaminant. All these limitations have restricted the OMC's applications. To improve the performance of the adsorption, one important innovative method is to modify both the surface chemistry and pore structure through post-treatments, because both the surface chemistry and the pore structure of the synthetic materials play a cooperative role in their application (Vinke et al. 1994). The idea of OMC post-treatment primarily came from the modification of the activated carbon materials. These approaches, which introduce functional groups to the surface of the activated carbon, have been categorized into oxidation, reduction, and electrode methods (Shafeeyan et al. 2010).

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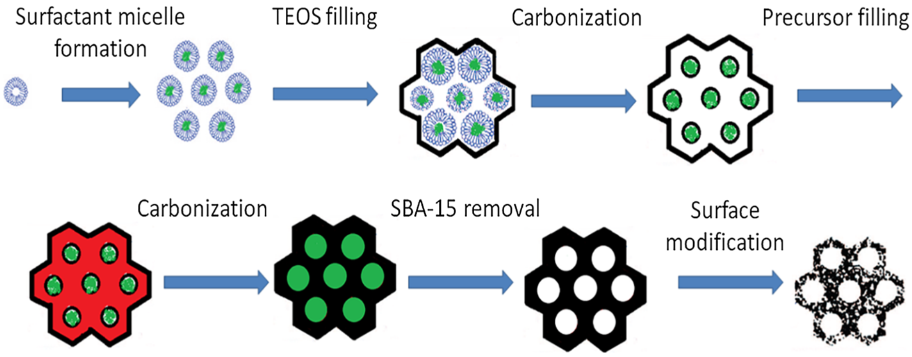
Fig. 1 Schematic illustration for the synthesis of OMC and surface modification



In this investigation, four different post-treatment methods were used to modify the ordered mesoporous carbons to improve their adsorption capacity. Figure 1 shows a schematic illustration for the synthesis of OMC and surface modification. The effects of template to carbon precursor ratio on adsorption were also evaluated during the initial stage of the OMCs' fabrication.

Materials and methods

Preparation of silica template

The silica templates (SBA-15) were synthesized under acid conditions using surfactant Pluronic P123 (BASF). The silica source was tetraethyl orthosilicate (TEOS, Aldrich). In an aqueous solution, 100 ml of concentrated hydrochloride acid (HCl, 37 %) was added into 525 ml of distilled water. Then, 20 g of Pluronic P123 was added into the mixture. The mixture was stirred for 1 h at room temperature. After Pluronic P123 was completely dissolved, 46.5 ml of tetraethyl orthosilicate (98 %) was added to the homogenous solution with vigorous stirring for 10 min. Then, the mixture was placed in a constant temperature water bath (Premiere Thermostatic water bath HH-4) for 4 h at 40 °C, followed by aging for 24 h at 90 °C. After aging, the solid product was washed with 80–90 °C hot distilled water and dried in an oven at 105 °C overnight. After drying, the product was calcined in a muffle furnace at 550 °C for 8 h (Guo et al. 2013). The white silica template SBA-15 was stored in a desiccator for the next preparation of OMC.

Preparation of ordered mesoporous carbons (OMCs)

The synthesis of OMC was accomplished by in situ polymerization of acrylic acid in the mesoporous structure of the silica template SBA-15 in an aqueous solution. Ninety (90) ml of acrylic acid (>99 %) was added into 180 ml of distilled water to form the acrylic acid aqueous solution. Then, different amounts of SBA-15(3 g [OMC1], 6 g [OMC2], 9 g [OMC3], and 12 g [OMC4]) were added into the acrylic acid aqueous solution. After stirring for 30 min, 0.06 g of 2.2-azobisisobutyronitrile (AIBN) was added as a free radical initiator, and the mixture was stirred for 15 min and then heated in the water bath (70 °C) for in situ polymerization with continuously stirring for about 30 min. The mixture of the polyacrylic acid (PAA) and the silica template was dried in an oven at 200 °C overnight. Then, the composite was heated under N_2 flow at a temperature ramp rate of 5 °C min^{-1} to 700 °C and held for 8 h for carbonization. The resulting carbon–silica composite was immersed into 50 ml 48 % HF (Aldrich) at room temperature with magnetic stirring for 24 h to remove the silica template. The OMC was then washed with distilled water (1.5 L) to remove the residual HF and dried in an oven at 90–100 °C overnight.

Post-treatments of the ordered mesoporous carbons

The four post-treatment methods are adopted from the modification of granular activated carbon materials (GACs) because they are classic methods for activated carbon (AC) material modifications.

1. Modification of the OMC with NaOH (OMC3-NaOH): The OMCs (0.10 g) were mixed with 10 ml of NaOH solution (0.1 %) for 3 h at 90 °C. Then, the mixture was filtered and washed until the water attained the same pH value as the distilled water employed. After that, the solid product was dried in an oven at 105 °C overnight.
2. Modification of the OMC with urea (OMC3-urea): The OMCs (0.10 g) were mixed with 10 ml of aqueous solution containing 2.0 g of urea ($(NH_2)_2$-CO > 99.5 %, Riedel) for 5 h at 90 °C. The mixture was filtered and pyrolyzed in a horizontal tubular furnace in a flow of dry nitrogen with a flow rate of 85 mL/min to a final temperature of 500 °C for 2 h. The sample was cooled below 50 °C before removing

from the furnace. The mixture was then washed with 1 L of distilled water and stirred at 80 °C for 1 h. The sample was filtered and oven-dried at 100 °C overnight.

3. Modification of the OMC with aluminum chloride (OMC3-$AlCl_3$): The OMCs (0.10 g) were mixed with a 10 ml aluminum chloride (1 mol/L) solution for 6 h at room temperature. After stirring, the solid product was filtered and dried in an oven (120 °C) overnight.
4. Modification of the OMC with ammonium hydroxide (OMC3-NH_3H_2O): OMCs (0.10 g) were immersed in 10 ml ammonium hydroxide (30 %) and placed in the incubator shaker (New Brunswick Scientific). The shaker was set at 60 °C with 110 rpm and allowed to shake for 3 h. Next, the mixture was filtered, and the solid product was then calcined in the protection of N_2 at 500 °C for 2.5 h.

Structure characterization

Small-angle X-ray diffraction (SAXRD) patterns were scanned with a Diano 2100E instrument using Cu Ka_{12} radiation ($k = 1.5418$, 45 V, 30 mA). The scanning rate was 0.03°/step and 15 s/step. Measurements were carried out to obtain resolved XRD patterns at 2θ angles from 0.5° to 3.5°. Transmission electron microscopy (TEM) was used to characterize the structure of the silica template and the carbon materials. The measurements were performed with an acceleration voltage of 100 kV by using a Hitachi 7600 Transmission Electron Microscopy. The BET specific surface areas were measured with a Micromeritics Flow-Sorb III surface area analyzer (Micromeritics). The surface functional groups and chemical bonds were detected by Fourier transform infrared spectroscopy (FTIR). The samples were scanned in a range of 4000–600 cm^{-1} using a 100 series Perkin–Elmer spectrometer. Thermogravimetric analysis (TGA) was conducted on a thermogravimetric analyzer SDT 2960 (Thermal Analysis Instruments, USA) in air with a flow rate of 100 mL/min. All the samples were heated from room temperature to 800 °C with rate of 20 °C/min.

Adsorption study and resorcinol measurement

Resorcinol, a typical total organic carbon (TOC) model compound, was selected to evaluate the adsorption behavior of the OMCs. Batch adsorption experiments were carried out in 250-mL conical flasks placed in an E24 incubator shaker (New Brunswick Scientific). The adsorption studies were conducted at 25 °C with a pH of 7.0. One sample of the same concentration solution (blank) without OMC/modified OMC was prepared and treated under the same conditions as the solutions containing adsorbent. This blank was used as a reference to establish the initial concentration of the solutions containing OMCs/modified OMCs. The mixture of OMC and resorcinol solution was placed in a shaker (200 rpm, room temperature) for 24 h. After the adsorption was completed, the conical flasks were removed and the solution was filtered using a 0.45-μm glass filter paper. The filtrate was analyzed for the final concentration of resorcinol. The amount of resorcinol adsorbed by OMCs/modified OMCs was determined by subtracting the final concentration from the initial concentration using the following formula:

$$q = \frac{(C_i - C_f)V}{M}$$

where q is the adsorption capacity (mg/g), C_i is the initial concentration of resorcinol in solution (mg/L), C_f is the final concentration of resorcinol in treated solution (mg/L), V is the volume of the solution taken (L), and M is the weight of the adsorbent OMCs (g).

The Cary 50 UV–visible spectrophotometer (Varian) was used to measure the resorcinol concentration because UV–visible spectrophotometry is a very sensitive method for phenol compound measurement. A standard method with wave length of 500 nm and temperature of 25 °C had settled for the test (Guo et al. 2013).

Results and discussion

Effect of SBA-15-to-carbon precursor (acrylic acid) ratio on structure and adsorption capacity of OMC

The amount of the silica template used could have a significant effect on the structures and adsorption capacity of OMC. In this investigation, different ratios of SBA-15 to carbon precursor [OMC1 (1:1), OMC2 (2:1), OMC3 (3:1), OMC4 (4:1)] were investigated.

Adsorption study

The adsorption capacities of four different OMCs with different ratios of SBA-15 to carbon precursor for resorcinol removal are shown in Fig. 2. The adsorption capacity of OMC3 made with SBA-15-to-carbon precursor ratio of 3:1 is 36.8 mg/g, that is, 10 % more than that of OMC2, 12 % more than that of OMC4, and 60 % more than that of the OMC1. It is noticed that the adsorption capacity increased by increasing the amount of silica template, and it reached the highest point when the ratio of silica template to carbon precursor reached 3:1. After that, the adsorption capacity dropped with the increase in the silica template.

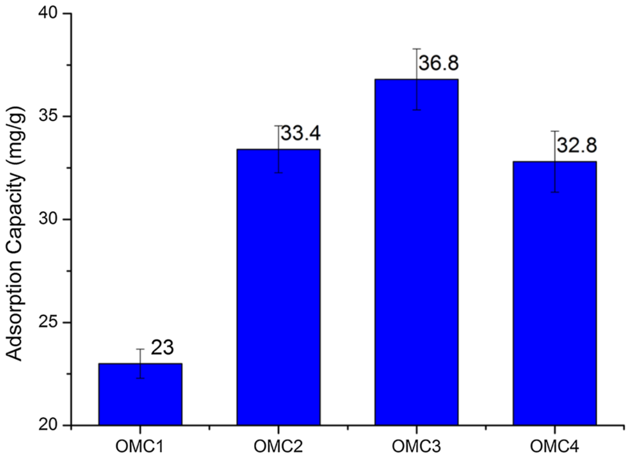


Fig. 2 Adsorption capacity of different OMCs

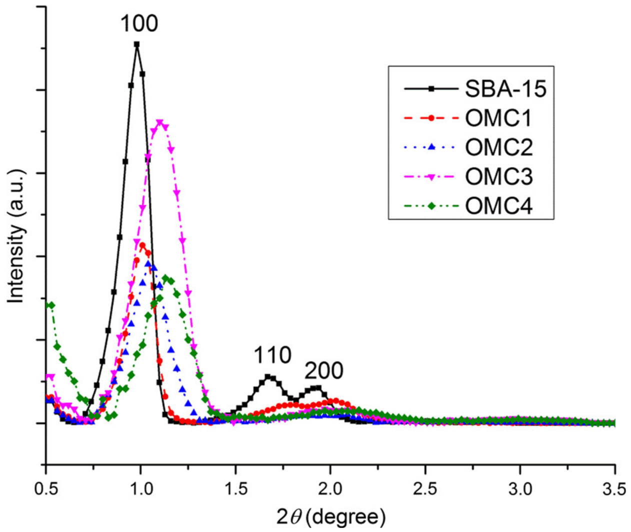


Fig. 3 XRD patterns of SBA-15 and ordered mesoporous carbon

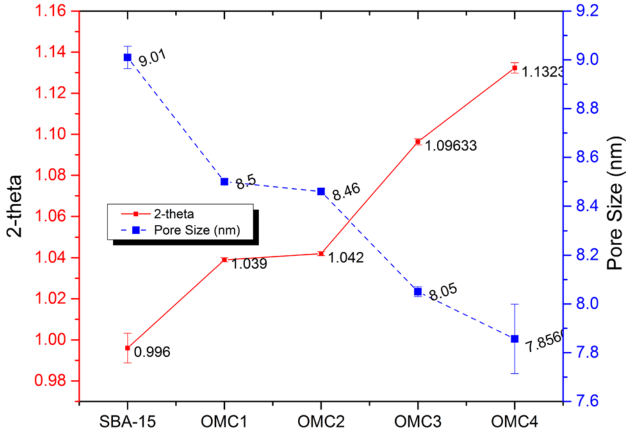


Fig. 4 Two-theta degrees of ordered mesoporous carbons

XRD analysis

The XRD patterns of silica template and ordered mesoporous carbons are presented in Fig. 3. It can be seen that the SBA-15 has three clear diffraction peaks indexed as crystal planes (100), (110), and (200), which indicates a well-ordered two-dimensional (2D) hexagonal structure. Similar results were reported by Skar et al. (2013). The OMCs have the similar structure replicating from the silica templates. Due to the defect of the polymerization and high-temperature damage from carbonization procedure, the intensities of the carbon materials were reduced at the peak of (100) diffraction.

It is obvious that with the increasing ratio of the silica template to carbon precursor, the peak of (100) diffraction has shifted to a larger degree. Based on Bragg's law (Kacher et al. 2009), this means a decrease in pore size. This may result from an inhomogeneous distribution caused by increase in silica template. OMC3 has the highest intensity. This indicated that it has the best ordered structure. Figure 4 illustrates the 2θ degree and pore size changes with the increasing amount of silica template. The pore sizes were calculated using the Jade 6 analysis. It can be seen that the pore sizes of OMCs are less than that of SBA-15. Zhang et al. (2005) reported the similar results.

Transmission electron microscopy

Figure 5 shows the TEM images of the silica template. Figure 5a shows the parallel array of SBA-15 template, and Fig. 5b shows the picture from the perpendicular direction. The hexagonal ordered structures of SBA-15 were similar to those described in the literature (Skar et al. 2013). Figure 6 shows the TEM images of the ordered mesoporous carbons. Even though the OMCs were made from different ratios of templates to carbon precursor, the TEM results showed that all the carbon materials have a highly ordered structure. Compared with the OMCs from other literature (Bazula et al. 2008), it was clear that the structure of ordered mesoporous carbon was faithfully duplicated from the structure of the silica template. The XRD and TEM images showed the silica template and all the OMCs have similar ordered mesopore structures.

BET surface area

The specific surface areas (SSA) of SBA-15 template and OMCs are listed in Table 1. Table 1 shows that OMC3 has the largest surface area. The general trend is OMC3 > OMC4 > OMC2 > OMC1. It is apparent that increase in the silica template to carbon precursor ratio can change the surface area of OMCs. The optimal ratio of silica templates to carbon precursor is 3:1. A similar phenomenon was

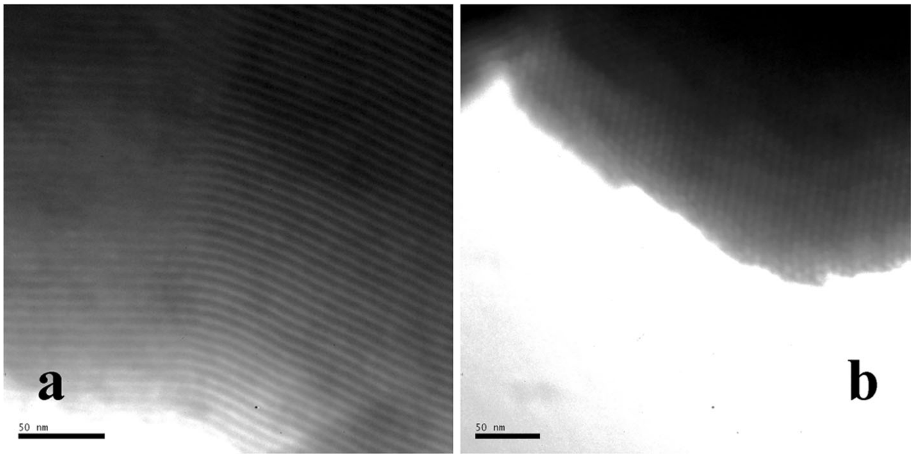


Fig. 5 TEM images of SBA-15: **a** channel direction, **b** perpendicular direction

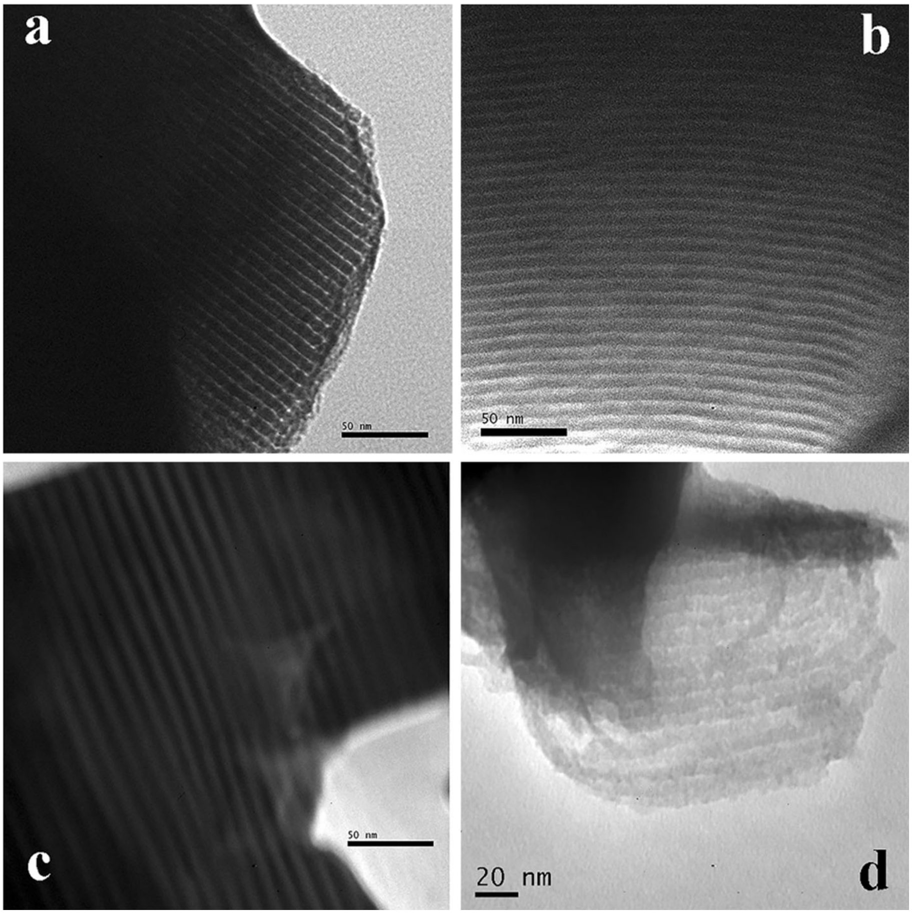


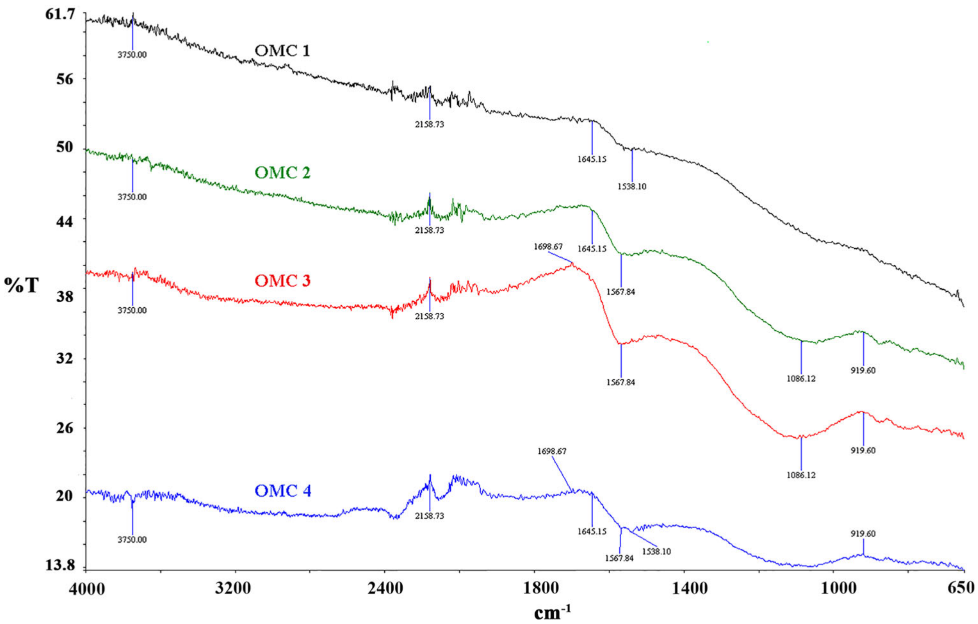
Fig. 6 TEM image of ordered mesoporous carbons: **a** OMC1, **b** OMC2, **c** OMC3, **d** OMC4

reported by Ramanathan et al. (2013) in a different carbon material preparation. Their results indicated the increasing ratio of silica templates to carbon precursor could increase the carbon material's specific surface area.

Table 1 Specific surface areas of the OMCs and SBA-15

Sample	SBA-15	OMC 1	OMC 2	OMC 3	OMC 4
SSA (m^2/g)	524.81	467.02	705.53	854.08	849.62

Fig. 7 FTIR spectra of different OMCs



FTIR analysis

FTIR spectral analysis allows the identification of different functional groups and chemical bonds on the surface of OMC. Figure 7 shows the FTIR spectra of the OMCs.

A set of peaks emerging in the region between 3600 and 3900 cm^{-1} can be attributed to the O–H and C–H stretching vibrations. This could be due to surface hydroxylic groups and –CH$_2$– in polyacrylic acid (Swiatkowski et al. 2004). The strong peaks in 2000–2400 cm^{-1} could be related to C–O group (Cansado et al. 2012). This is probably associated with the polymerization of the acrylic acid. The C=O bond related to the COOH group can be found around 1700 cm^{-1}. The band located at 1570 cm^{-1} is assigned to the stretching of the C=C bond due to incomplete polymerization. Fujimori et al. (2014) reported a similar conclusion in their study. In the lower region of the spectra, the band around 1050 cm^{-1} could be due to antisymmetric stretching vibrations of the C–O–C group or also to out-of-plane C–H bending. FTIR spectra show that all OMCs have similar chemical bonds. The functional groups are similar for all OMCs made from different SBA-15-to-acrylic acid ratios.

Based on the FTIR analysis, all four ordered mesoporous carbons have similar surface functional groups; also, the morphologic structures are almost the same from the TEM images. Therefore, the increase in the adsorption capacity could be mainly due to the increase in the specific surface area for the OMC materials.

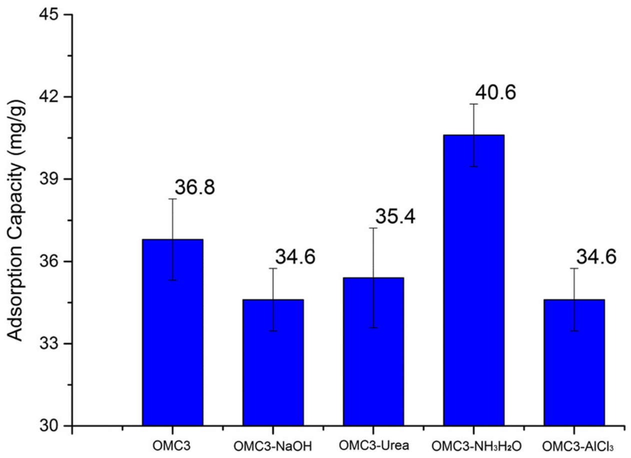


Fig. 8 Adsorption capacity of modified OMC versus original OMC

Effect of post-treatments on structure and adsorption capacity of OMC

To improve the adsorption capacity of OMC, modifications of both surface chemistry and pore structure are necessary. OMC3, with the highest adsorption capacity, was used as the original OMC for the surface modifications.

Adsorption study

The adsorption capacities of four modified OMCs are compared with the original OMC in Fig. 8.

It shows the OMC modified by NH_3H_2O has the highest adsorption capacity of 40.6 mg/g. Compared with the original OMC3, the modified samples have smaller surface areas, but their adsorption capacities were varied from each other. The phenomenon demonstrates that the modification procedures have complex effects on the OMC, which need more characterizations on the structure and chemical property.

XRD analysis

The SAXRD patterns of all the modified OMCs are shown in Fig. 9. It is clear that all the modification methods have reduced the intensity of the peak at (100) diffraction. This could be due to the erosion effect from the modifying procedures. Figure 10 shows the 2θ and pore size changes after the post-treatment. The NaOH and urea modifications caused a decrease in the two theta angle. The (100) diffraction peaks of NaOH- and urea-modified OMCs have shifted to the left compared with that of the original OMC; this meant that the pore size of the two modified OMCs had increased.

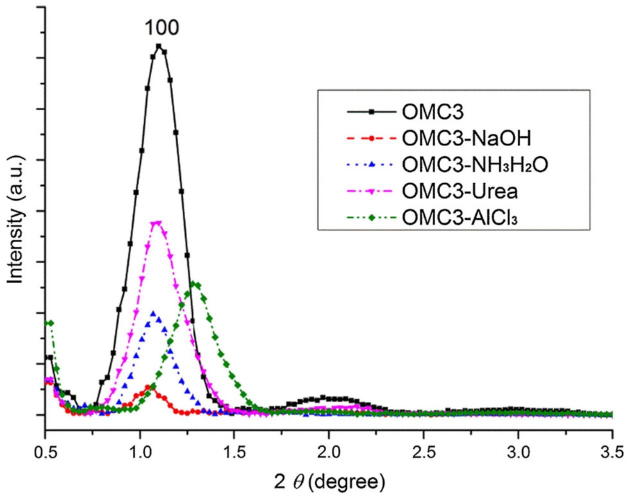


Fig. 9 XRD patterns of modified OMC

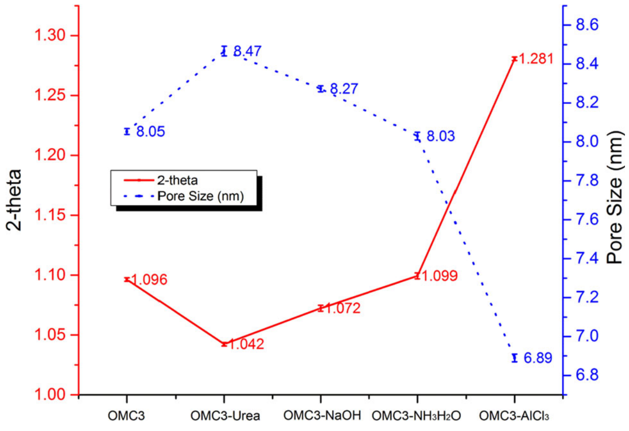


Fig. 10 Two-theta degrees and pore size of modified OMCs

On the other hand, the peak of $AlCl_3$-modified OMC shifted to a larger degree, which meant a decrease in the pore size. A possible reason for this shift is that the hydrolytic products from $AlCl_3$ may damage the pore structure of the OMC due to the amphoteric property of $AlCl_3$. The NH_3H_2O-modified OMC had a similar pore size compared with the original carbon materials.

TEM

The TEM images of the modified OMCs are shown in Fig. 11. The images of modified OMCs show that the morphologies are essentially the same as before, and the parallel channels are still preserved. A collapse of pore structure was also observed, which may reduce the adsorption capacity of the materials. Bazula et al. (2008) verified this point in their study.

BET specific surface area (SSA)

Table 2 shows the BET specific surface area of all the modified OMCs. All of the modified OMC materials have smaller SSA than the original OMC. Considering the erosion phenomenon in the TEM image, the decrease in the SSA could be mainly due to the damage of the pore structure from the immersing modification procedure. The different properties of the immersing solutions resulted in the different decreases in surface area. This phenomenon is quite similar to the result reported by Pevida et al. (2008). In their study, the ammonia treatment of activated carbon resulted in lower BET surface areas compared with the parent material. Also, Cansado et al. (2012) and Fujimori et al. (2014) found that these modifications usually decreased the surface area of activated carbon due to the shrinkage and blockage of the micropores. The modification procedures could damage the highly ordered structures and cause the ordered structures to collapse.

FTIR analysis

The FTIR spectra of original ordered mesoporous carbon and modified OMC are shown in Fig. 12. Compared with the original OMC, the NaOH-treated OMC presented strenuous vibration around 1400 and 1050 cm^{-1}. The broad band in 1400 cm^{-1} consisted of a series of overlapping absorption bands ascribable to the deformation vibration of surface hydroxyl groups. The new peak around 1030 cm^{-1} indicated the enhancement of the C–H

Fig. 11 TEM images of modified OMCs

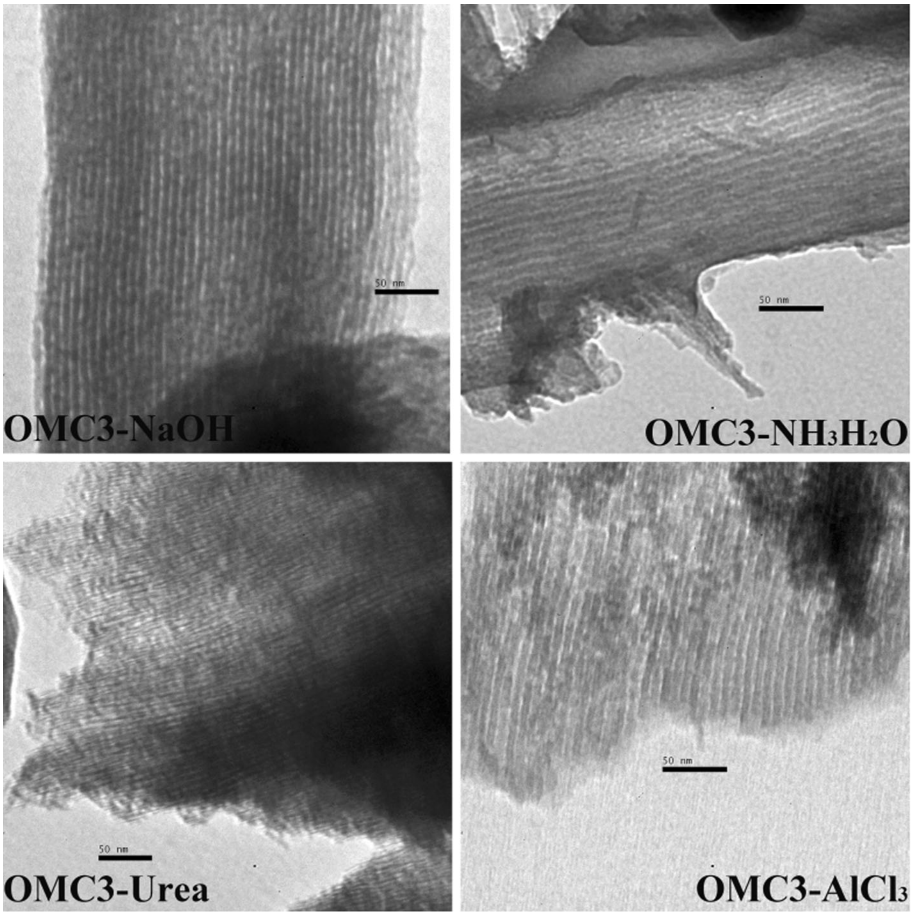
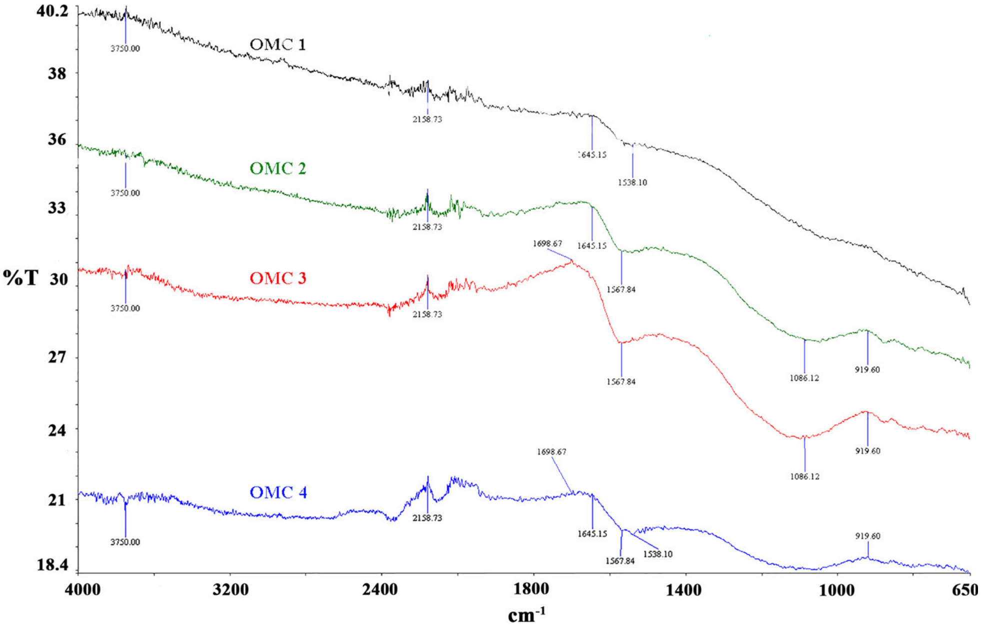


Table 2 Specific surface area of the modified OMCs

Sample	OMC3	OMC3-NaOH	OMC3-urea	OMC3-NH_3H_2O	OMC3-$AlCl_3$
SSA (m^2/g)	854.08	745.56	723.38	767.10	511.19

Fig. 12 FTIR spectra of OMC and modified OMCs



stretching vibration from the C=C group. Dehydration might occur due to the strong alkalinity of the sodium hydroxide solution.

In the FTIR spectra of ammonia-treated OMC, weak peaks around 2400 cm^{-1} could be assigned to the loss of C–O–C groups, which was mainly due to the alkalinity of the ammonium hydroxide. The similar reduction that appeared around the 1700 cm^{-1} could be attributed to the stretching vibrations of C=O moieties in carboxylic group. Swiatkowski et al. (2004) reported similar results in the modification of activated carbons with ammonium hydroxide.

OMC modified by urea presented almost the same peaks with the original samples. Despite the similarity of the FTIR spectra profiles, a deformation vibration of surface carboxylic groups happened at 1640 cm^{-1}. However, Cansado et al. (2012) reported a band at 1270 cm^{-1} in urea-modified AC. The difference of the carbon precursor and structures might lead to these differences. The black line shows the IR spectrum of OMC modified by $AlCl_3$. The intense vibrations at 3600–4000 cm^{-1} could be associated with the introduction of O–H group. Meanwhile, the aluminum chloride strengthened the C–O–C group at 1050 cm^{-1}. It agrees with the conclusion reported by Bazula et al. (2008).

Based on the FTIR tests, the surface modification has introduced various functional groups to the OMCs. Swiatkowski et al. (2004) believed that hydrogenant functional groups, such as hydroxyl, could increase adsorption capacity of the adsorbent. Based on the results from the batch adsorption tests, the introduced functional groups play the dominant role in the adsorption behavior.

Thermogravimetric analysis

Thermogravimetric Analysis (TGA) was carried out to estimate the stability of OMCs and modified OMCs. The weight loss of all the OMCs and modified OMCs is shown in Fig. 13, with the differential thermal gravity (DTG) results inserted. The results of TGA curve showed that the original carbon decomposed at temperature range 510–650 °C with total weight loss of 98 %. Gao et al. (2008) reported similar results when they investigated photo-electro-catalysis enhancement on carbon nanotubes. From the graph, it can be seen that the $AlCl_3$ modification leads to a larger residual, which is probably due to the introduction of aluminum ions. The other three modified OMCs are similar to the original OMC without any residuals.

The DTG shows that the thermal stabilities of OMC modified by urea, NaOH, and NH_3H_2O had decreased very little. So even the structure of the modified OMCs had been damaged, the thermal stability of them had kept the same level to adapt to the circumstances.

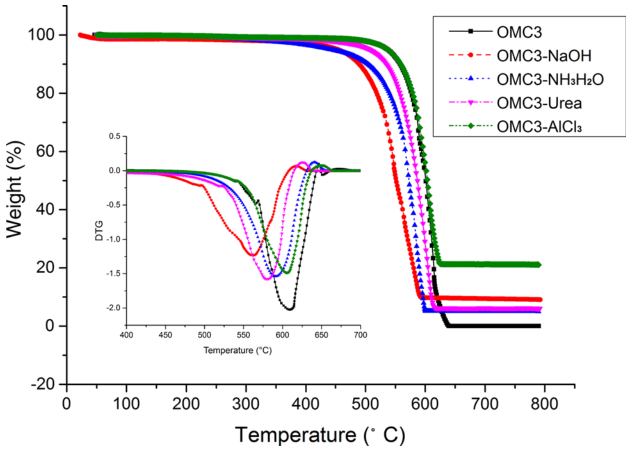


Fig. 13 TGA and DTG of the modified OMC

Conclusion

This investigation studied the effects of the ratios of silica template to carbon precursor on the OMC structures. The results showed that the increasing ratio of SBA-15 to carbon precursor could lead to a larger surface area and a higher adsorption capacity. The XRD analysis and TEM images showed the SBA-15 and OMCs have a highly ordered hexagonal structure. Also, the BET surface area analysis and adsorption behavior tests showed that OMC3 had the largest specific surface area and higher adsorption capacity of 36.8 mg/g.

The post-treatment methods have eroded the surface of the OMCs without destroying the well-ordered structure. The erosion effects have decreased the surface area of the OMC. Simultaneously, the surface functional groups of the OMCs were changed, based on FTIR analysis. The resorcinol adsorption study showed the ammonium hydroxide-treated OMC had the highest adsorption capacity than that of the original OMC, with an adsorption capacity of 40.6 mg/g.

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