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New mesoporous carbon materials synthesized by a templating procedure

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Abstract

The new porous carbon materials were obtained by templating procedure using mesoporous silica (SBA-15) as template. The ordered mesoporous silica materials were synthesized by using Pluronic P123 (non-ionic triblock copolymer, $EO_{20}PO_{70}O_{20}$). SBA-15/cryogel carbon composites were obtained by sol–gel polycondenzation of resorcinol and formaldehyde in the presence of different amount of SBA-15. The polycondenzation was followed by freeze drying and subsequent pyrolysis. One set of SBA-15/sucrose carbon composites was prepared by using sucrose as carbon source. The silica template was eliminated by dissolving in hydrofluoric acid (HF) to recover the carbon material. The obtained carbon replicas were characterized by nitrogen adsorption–desorption measurements, X-ray diffraction and scanning electron microscopy (SEM). It was revealed that the samples have high specific surface (533–771 $m^2\,g^{-1}$), developed meso- and micro-porosity and amorphous structure. Porous structure of carbon replicas was found to be a function of the carbon source, properties of SBA-15 and silica/carbon ratio. Room temperature adsorption of nitrogen and adsorption of phenol from aqueous solutions were investigated.

Keywords: A. Sol–gel method; Carbon replica; Mesoporosity; Nanomaterial

1. Introduction

Since the mesoporous siliceous materials were synthesized [1–5], there has been an increasing interest in the tailoring of these materials for potential applications in separation and adsorption processes, catalysis etc. One of the most studied mesoporous materials is SBA-15 (Santa Barbara No. 15), which can be synthesized in large quantities from tetraethyl orthosilicate (TEOS) in the presence of amphiphilic poly (alkylene oxide)-type triblock copolymers [6,7]. This material has a highly ordered mesoporous hexagonal structure with mesopore diameters varying from 5 to 30 nm [8,9]. The pore size and the thickness of silica walls can be adjusted by varying the temperature and time of thermal treatment of the reaction solution. Careful investigation of the SBA-15 structure has revealed a certain amount of micropores, which connect neighboring mesopores [8–11].

Ordered mesoporous carbon, synthesized by using ordered mesoporous silica as template, is porous material with structure which is usually an inverse replica of the used template. In general, sucrose is most often used as a carbon source [11–33], however, carbon replicas can also be successfully obtained by using organic compounds such as furan resin and acetone [34,35], resorcinol–crotonaldehyde polymer [36], acrylic acid [37], furfuryl alcohol [27,38,39], petroleum pitch and propylene [16,18,21], para-toluene sulfonic acid and furfuryl alcohol [40], Pluronic P123 [41] and poly-vinyl chloride [42].

It has been reported that carbon replicas obtained from silica templates can be effectively used as material for supercapacitors [18,21], catalyst support [20,35], hydrogen storage [29], electrocatalytic oxidation of dopamine [33], in adsorption of methane [17,25], light gases [24], methyl mercaptan [22], n-heptane and cyclohexane [19], dyes [31],

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aqueous alkylphenol ethoxylate surfactant [26], L-histidine [28]. Investigations have shown that the efficiency of material is strongly affected not only by its structural properties but also by its surface functionality.

Carbon obtained from cryogel is a special class of porous carbon material [43–47] which is usually formed from the sol–gel polycondenzation of resorcinol and formaldehyde, followed by freeze drying and subsequent pyrolysis. This material has high specific surface and mesoporous structure which can be controlled by varying the concentration of starting resorcinol–formaldehyde solutions and catalyst.

In this paper, a completely new method for preparation of porous carbon material by using SBA-15 as template and resorcinol–formaldehyde (RF) gel as a source of carbon is presented. New carbon materials were characterized by nitrogen adsorption, XRD and SEM method. Room temperature adsorption of gaseous nitrogen and phenol adsorption from aqueous solutions were also investigated.

2. Experimental

2.1. Synthesis of SBA-15 samples

SBA-15 samples were synthesized according to the standard procedure [4,48], using Pluronic P123 (non-ionic triblock copolymer, $EO_{20}PO_{70}O_{20}$, BASF) as a surfactant and tetraethoxysilane (TEOS, 98%) as a source of silica. Although the synthesis of SBA-15 was described in detail in our previous study [48] it is important to stress that the mixtures of Pluronic P123 and TEOS were subjected to two different aging treatments.

Detailed procedure of preparation of SBA-15/carbon composites is presented in reference [49]. Different amounts of previously prepared SBA-15 were added to water mixture of resorcinol ($C_6H_4(OH)_2$), formaldehyde (HCHO) and sodium carbonate (Na_2CO_3), which was used as a basic catalyst. The volume of resorcinol–formaldehyde (RF) solution was adjusted to yield 4 g of carbon. The synthesis conditions are listed in Table 1. The sample notation gives information about source of carbon, SBA-15 aging temperature and Si/C ratio in the sample. For instance: Si80C 0.2, means that SBA-15 was aged at 80 °C, source of carbon was carbon cryogel (C) and Si/C ratio was 0.2.

Table 1
Synthesis conditions of SBA-15/carbon cryogel composites.

C80 *x*	SBA-15/80 (g)	*C* (g)
C80 0.20	0.8	4.0
C80 0.50	2.0	4.0
C80 0.75	3.0	4.0
C100 *x*	SBA-15/100 (g)	*C* (g)
C100 0.20	0.8	4.0
C100 0. 50	2.0	4.0
C100 1.00	4.0	4.0

Similar to the method proposed by Pekala [43], the suspensions were decanted in glass tubes (inner diameter = 10 mm) and sealed. In order to cause gelation of RF, the tubes were held 2 days at 25 °C, 1 day at 50 °C and finally 4 days at 85 °C. RF cryogels with SBA-15 were prepared by freeze drying according to the procedure of Tamon et al. [44–47]. SBA-15/cryogel carbon composites were prepared by carbonization of the samples in a conventional furnace at 800 °C in nitrogen flow. After pyrolysis, the furnace was cooled to room temperature.

For the comparison, SBA-15/sucrose carbon composites were prepared according to procedure given elsewhere [12]. Briefly, 1 g of SBA-15/80 and SBA-15/100 was added to a solution obtained by dissolving 1.5 g of sucrose (BDH Prolabo) and 0.19 g of H_2SO_4 (p.a., Centrohem) in 5 ml of distilled water. The mixtures were heated for 6 h at 100 °C and for 6 h at 160 °C. After the addition of 1 g of sucrose, 0.1 g of H_2SO_4 and 5 ml of distilled water, the samples were treated again for 6 h at 100 °C and for 6 h at 160 °C. SBA-15/sucrose carbon composites were prepared by carbonization of the samples in a conventional furnace, at 900 °C in nitrogen flow. After pyrolysis, the furnace was cooled to room temperature. The Si/C ratio in these samples was 0.5 and they will be denoted as Si80S 0.5 and Si100S 0.5 depending on SBA-15 aging temperature.

Carbon replicas were obtained by the immersion of SBA-15/carbon composites in solution of HF. Typically, 1 g of SBA-15/carbon composites was mixed with 20 ml of HF (p.a. 40%, E. Merck) for 20 h. After that, replicas were washed in 100 ml of ethanol and about 250 ml of distilled water, until the neutral pH reaction. The samples were dried in air, at 120 °C, for 24 h.

2.2. Characterization of carbon replicas

Adsorption and desorption of N_2 on carbon replicas were measured at −196 °C using the gravimetric McBain method. The specific surface area (S_{BET}), pore size distribution, mesoporous surface area including external surface area (S_{meso}) and micropore volume (V_{mic}) of the samples were calculated based on the obtained adsorption and desorption isotherms. The pore size distribution was estimated by applying the BJH method [50] to the desorption branch of isotherms whereas mesoporous surface area and micropore volume were estimated using the high-resolution α_s plot method [51–53]. Microporous surface area (S_{mic}) was calculated by subtracting S_{meso} from S_{BET}.

Carbon replicas were characterized by recording powder X-ray diffraction (XRD) patterns on a Siemens D500 X-ray diffractometer using Cu Kα radiation with a Ni filter. The 2θ angular region between 5° and 80° was explored at a scan rate of 1°/s with the angular resolution of 0.02° for all the XRD tests. The scanning electron microscopy (SEM) analysis was carried out on the carbon replicas using the JEOL 6300F microscope. Nitrogen adsorption isotherms on the carbon replicas at room temperature were measured using the gravimetric method.

The adsorption of phenol from aqueous solutions was determined at 25 °C. Starting concentration of phenol solutions was ranging from 50 to 400 mg dm^{-3}. 25 mg of carbon replica was immersed in 25 ml of phenol solution and equilibrated by 20 h long vigorous stirring. After filtration, the equilibrium concentration of phenol was measured by using UV/VIS Perkin Elmer Lambda 2 Spectrophotometer.

3. Results and discussion

3.1. Adsorption isotherms—BET experiments

In order to better understand the properties of carbon replicas it would be essential to point out some important results regarding SBA-15 [48] and SBA-15/carbon composites [49] which are presented in our previous reports. It was shown that SBA-15 samples aged at two different temperatures are mesoporous materials. The specific surface areas of SBA-15/80 (aged at 80 °C) and SBA-15/100 (aged at 100 °C) were 710 m^2 g^{-1} and 641 m^2 g^{-1}, respectively. Furthermore, the pore radius of SBA-15/80 varied between 2 and 6 nm whereas the pore radius of SBA-15/100 varied between 2 and 25 nm which indicates that the use of SBA-15 template aged at lower temperature gives samples with narrower pore size distribution. As far as silica/carbon composites are concerned, it was found that silica/cryogel carbon composite materials had high specific surface (350–520 m^2 g^{-1}), developed meso- and micro-porosity and amorphous structure. However, mesoporosity of silica/sucrose carbon samples was negligible. The amount of micro- and meso-pores in silica/cryogel carbon composites was successfully controlled by Si/C ratio.

Adsorption and desorption isotherms of N_2 on carbon replicas with different Si/C ratios and different carbon source are shown in Fig. 1. The isotherms are given as functions of N_2 relative pressure at −196 °C. According to the IUPAC classification [54], the isotherms for all carbon replicas are of type-IV and with a hysteresis loop typically for mesoporous materials. The relatively high adsorption of nitrogen at low relative pressure indicates a presence of significant amount of micropores in the samples. The specific surface areas, calculated by the BET equation (S_{BET}) are listed in Table 2. It is obvious that the specific surface of samples obtained by using carbon cryogel as source of carbon is not a function of silica/carbon ratio. Carbon replicas obtained by using sucrose as carbon source have larger specific surface than those obtained by using carbon cryogel. These results can be explained by the fact that carbon replica obtained by carbonization of sucrose retained the structural properties of silica template. In the carbon cryogel replicas, structure is a combination of the porous network established during the gelation process and repeated structural properties of silica template. In our previous paper [49] we showed that the amount of carbon particles in SBA-15/cryogel carbon composite decreases as the Si/C ratio increases which implies that RF gel just partially penetrates into the mesopores of SBA-15. Therefore, the excess graphite, i.e., the graphite that cannot penetrate into silica pores, is located on the surface of peanut-like silica

Table 2
Porous properties of the carbon replicas (please note that S and C in sample notation refer to carbon source).

Sample	S_{BET} (m^2 g^{-1})	S_{meso} (m^2 g^{-1})	S_{micro} (m^2 g^{-1})	V_{micro} (m^3 g^{-1})
S80 0.50	771	254	517	0.4
C80 0.20	604	181	423	0.20
C80 0. 50	569	109	460	0.24
C80 0.75	533	145	388	0.21
S100 0.50	739	343	396	0.27
C100 0.20	584	149	435	0.26
C100 0.50	571	157	414	0.23
C100 1.00	533	162	371	0.19

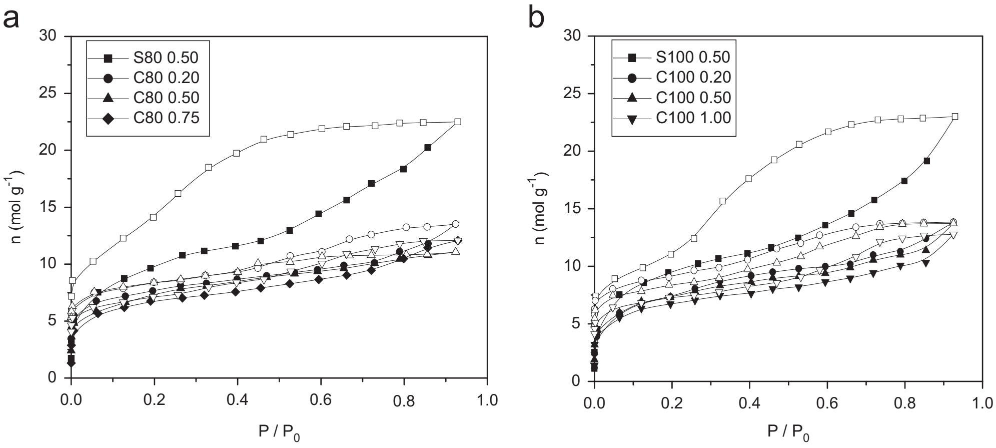

Fig. 1. Nitrogen adsorption and desorption isotherms for carbon replicas with different carbon source and Si/C ratio synthesized by using SBA-15 aged at (a) 80 °C and (b) 100 °C. Solid symbols—adsorption, open symbols—desorption.

domains. It is believed that polycondenzation of resorcinol and formaldehyde and therefore the formation of RF clusters is a relatively fast reaction. The large size of newly-formed clusters actually prevents their penetration into the pores of SBA-15. However, aqueous solution of sucrose very easily fills the mesopores and almost all carbon goes into porous SBA-15.

The pore size distributions (PSD) of the carbon replicas are shown in Fig. 2. All samples have developed micro- and meso-porosity and the pore radius is below 4 nm. All replicas have narrower pore size distribution in comparison with silica templates which is in agreement with the literature data [12,23,34,37]. The samples with sucrose as carbon source are microporous with certain amount of mesoporosity.

The α_s plots, obtained using standard nitrogen adsorption isotherms, are shown in Fig. 3. The mesoporous surface area which includes the contribution of the external surface (S_{meso}) was determined by the slope of straight line in the high α_s region. At the same time the micropore volume (V_{mic}) was determined by the vertical axis intercept. The calculated porosity parameters (S_{meso}, S_{mic}, V_{mic}) are presented in Table 2. Analysis of the experimental data confirms that, in the carbon replicas, a significant part of specific surface is in micropores. The carbon replicas obtained from sucrose have larger overall specific surface (S_{BET}) and mesoporous surface (S_{meso}) in comparison with the samples obtained from carbon cryogels. The properties of porous carbon replicas obtained from sucrose are affected by the source of silica (aging temperature of SBA-15), which is not in agreement with the literature data [27]. As presented in Table 2, the overall specific surface (S_{BET}) and microporous surface (S_{mic}) of replicas obtained from carbon cryogels slightly decrease whereas the mesoporous surface (S_{meso}) slightly increases with increasing Si/C ratio. These results are considered to be the consequence of silica framework shrinkage during carbonization, which is in an agreement with literature data for replicas obtained from other precursors [30]. The results presented in Table 2 also indicate that the properties of porous replicas

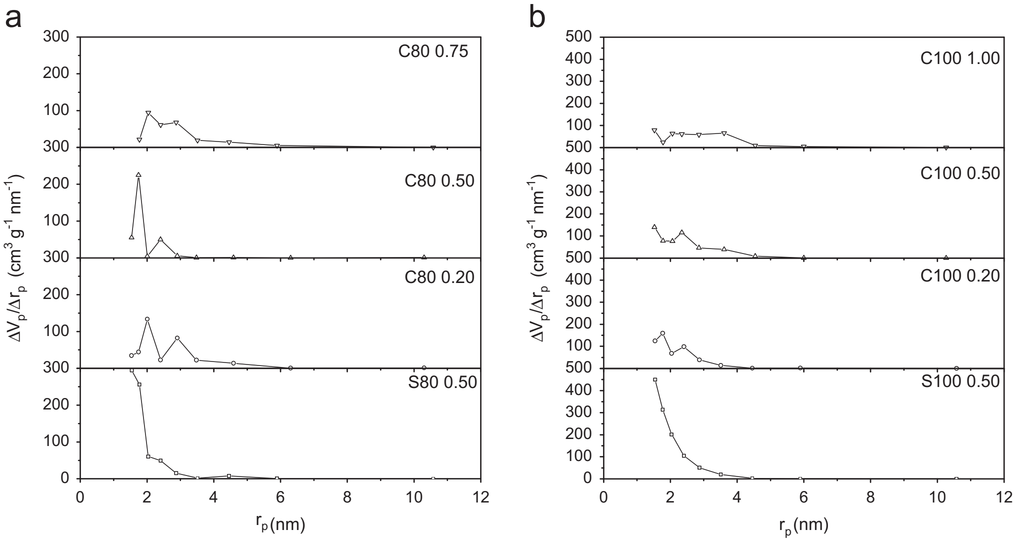

Fig. 2. Pore size distribution (PSD) of the carbon replicas with different carbon source and Si/C ratio synthesized by using SBA-15 aged at (a) 80 °C and (b) 100 °C.

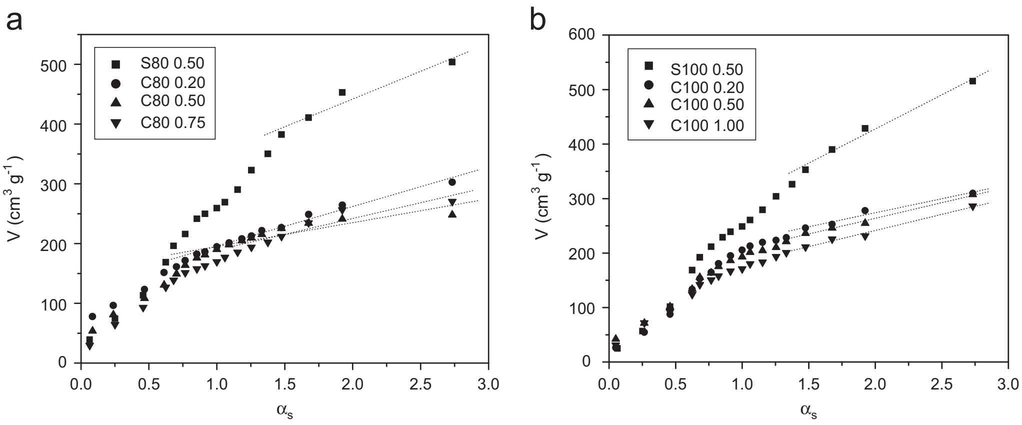

Fig. 3. α_s-plots of the nitrogen adsorption isotherm of carbon replicas with different carbon source and Si/C ratio synthesized by using SBA-15 aged at (a) 80 °C and (b) 100 °C.

obtained from carbon cryogels are very similar. This can be explained by the fact that RF gel undergoes a substantial volume contraction during pyrolysis. The comparison of these properties with the properties of porous carbon cryogel synthesized by standard procedure [55] indicates that carbon cryogel replicas have significant increase of microporosity. It is believed that the microporosity comes from repeated SBA-15 structure whereas mesoporosity comes from the carbon cryogel structure. Again, these results indicate that during synthesis of SBA-15/cryogel carbon composite, the mesopores of SBA-15 are not completely filled with the cryogel. This conclusion is also supported by the fact that microporosity decreases with the increase in Si/C ratio.

It is interesting to note that the obtained samples have very similar adsorption isotherms with the carbon replicas described in [12,15,16]. It is especially true when it comes to low relative pressure at which high amount of adsorbed nitrogen was measured. Although this shape of isotherms confirms the presence of micropores, only few authors have reported that the non heat-treated carbon replicas contain micropores [38,39,46]. According to our experimental results and literature data it is easy to conclude that all ordered mesoporous carbon materials have significant amount of micropores which can be eliminated only by thermal treatment at high temperature in an inert atmosphere [15].

3.2. XRD analysis

The XRD patterns of the carbon replicas are shown in Fig. 4. As can be seen, the patterns of all samples are quite similar without well defined peaks. The intensity of two broad humps at 26° and 43° which are characteristic of amorphous carbon is a function of the amount of carbon in samples.

3.3. SEM analysis of SBA-15/cryogel carbon composites

SEM images of carbon replicas are presented in Figs. 5 and 6. Fig. 5 shows images of replicas obtained by using SBA-15/80 silica while Fig. 6 shows images of samples obtained by using SBA-15/100 silica as template. Fig. 5d and Fig. 6d show that the samples obtained from sucrose as source of carbon are porous materials with structure which is an inverse replica of the SBA-15 template. As presented in our previous study [48], SBA-15 consists of many peanut-like domains with relatively uniform sizes (about 1 μm) which are aggregated into wheat-like structures. However, the carbon replicas obtained from carbon cryogel as a source of carbon (Fig. 5a–c and Fig. 6a–c) have structure similar to the structure of carbon cryogels synthesized by the standard procedure [43–47]. This confirms our assumption that RF gel just partially penetrate into the mesopores of the SBA-15.

3.4. Room temperature adsorption of nitrogen

Room temperature adsorption isotherms of nitrogen, as a functions of relative pressure for carbon replicas are shown in Fig. 7. The quantities of adsorbed nitrogen are in agreement with the literature data for the carbon/silica composite synthesized from MCM-41 and polyfurfuril alcohol as a carbon source [24]. The isotherms presented in Fig. 7 show that carbon replicas obtained from different SBA-15 precursors adsorb almost the same quantities of nitrogen at the same relative pressures. Also, the samples with higher amounts of mesopores are better adsorbents which indicate that the presence of mesopores is beneficial for nitrogen adsorption. It is also evident from Fig. 7 that carbon cryogel replicas are as good as, or even better adsorbents than sucrose replicas.

3.5. Phenol adsorption

Phenol adsorption isotherms for carbon replicas C80 0.75, C100 1.0 and S80 0.50, at 25 °C, are presented in Fig. 8. Adsorption isotherms are obtained at pH = 6. Isotherms may be classified according to Giles et al. [56] as B2 type. The shape of isotherms shows that dispersion interactions are dominant in adsorption of phenol. At higher equilibrium concentrations, the shape of isotherms is a consequence of multilayer adsorption. The quantities

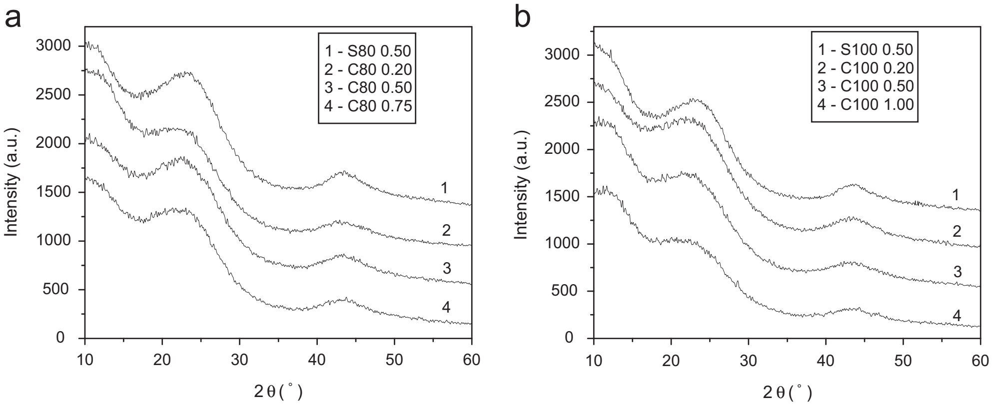

Fig. 4. X-ray diffraction (XRD) patterns of the carbon replicas with different carbon source and Si/C ratio synthesized by using SBA-15 aged at (a) 80 °C and (b) 100 °C.

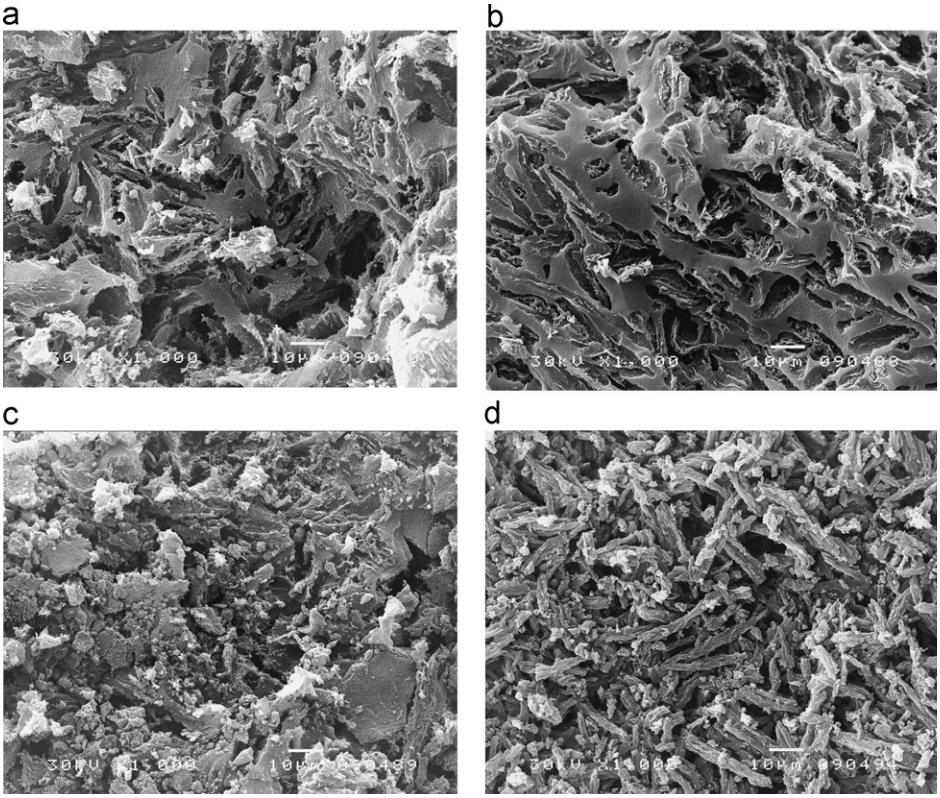

Fig. 5. SEM images of carbon replicas obtained from SBA-15 aged at 80 °C using (a–c) cryogel and (d) sucrose as carbon source.

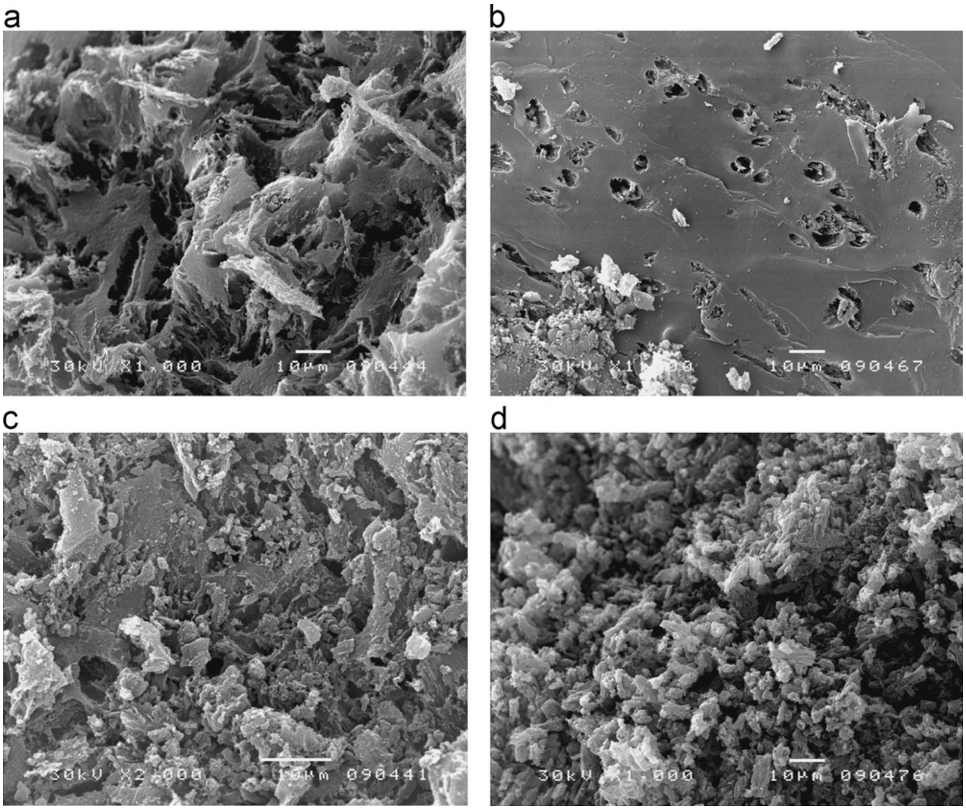

Fig. 6. SEM images of carbon replicas obtained from SBA-15 aged at 100 °C using (a–c) cryogel and (d) sucrose as carbon source.

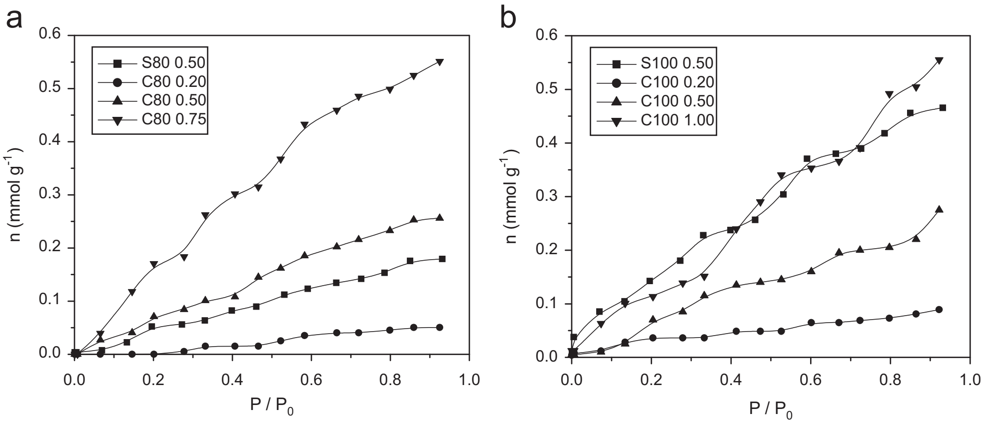

Fig. 7. Room temperature adsorption isotherms of nitrogen, as a functions of relative pressure for carbon replicas with different carbon source and Si/C ratio synthesized by using SBA-15 aged at (a) 80 °C and (b) 100 °C.

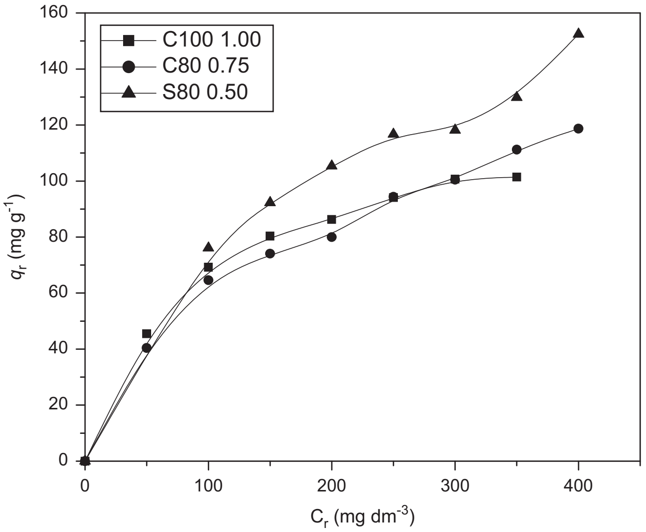

Fig. 8. Phenol adsorption isotherms for carbon replicas C80 0.75, C100 1.00 and S100 0.50, at 25 °C.

of adsorbed phenol are in an agreement with the literature data for the CMK-3 carbon replicas obtained by using sucrose as a source of carbon [57,58]. Adsorption on carbon replica samples obtained from carbon cryogel is lower at higher concentrations in comparison with carbon replica sample obtained from sucrose as a source of carbon. This implies that the presence of mesopores slightly decreases the amount of adsorbed phenol which is in an agreement with the literature data [59,60].

4. Conclusion

The new porous carbon materials were obtained by the templating procedure using SBA-15 as silica matrix and carbon cryogel and sucrose as a source of carbon. Characterization by nitrogen adsorption–desorption measurements, X-ray diffraction and scanning electron microscopy (SEM) showed that samples have high specific surface (533–771 m^2 g^{-1}), developed meso- and microporosity and amorphous structure. The properties of porous replicas are affected by the carbon source, silica source and silica/carbon ratio. Characterization of samples revealed that carbon replicas obtained from carbon cryogel have structure which is a combination of structure of carbon cryogel and inverse replica of the used silica template. Room temperature adsorption of nitrogen and adsorption of phenol from aqueous solutions showed that these materials can be successfully used as adsorbents in gaseous and liquid solutions.

Acknowledgments

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