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# Adsorption properties of phenolic resin-based mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58 or Brij 78 polymers

**Jerzy Choma · Anna Żubrowska · Joanna Górka · Mietek Jaroniec**

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**Abstract** Phenolic resin-based mesoporous carbons were synthesized by using mixed templates of Pluronic F127 and Brij 58 or Brij 78. For the purpose of comparison three samples of nanoporous carbons were prepared by using single templates of Pluronic F127, Brij 58 and Brij 78 polymers, respectively. Adsorption properties of the aforementioned carbons were studied by nitrogen adsorption at −196 °C. The resulting carbons featured high specific surface areas ranging from 641 $m^2$/g for the sample obtained in the presence of Brij 58 polymer to 825 $m^2$/g for the carbon prepared by using a mixed template containing 23% of Pluronic F127 and 77% of Brij 58 and from 588 $m^2$/g for the sample obtained in the presence of Brij 78 polymer to 813 $m^2$/g for the carbon prepared by using Pluronic F127 only. It was shown that the width of mesopores increases with increasing amount of Brij 58 or Brij 78 in the mixture of one of these polymers with Pluronic F127, suggesting that Brij polymers act also as micelle expanders.

**Keywords** Mesoporous carbons · Soft templating · Mixed polymeric templates · Adsorption properties · Phenolic resin

J. Choma (✉)
Institute of Chemistry, J. Kochanowski University, 25-406 Kielce, Poland
e-mail: jchoma@wat.edu.pl

A. Żubrowska · J. Górka · M. Jaroniec
Department of Chemistry, Kent State University, Kent, OH 44242, USA

M. Jaroniec
e-mail: jaroniec@kent.edu

## 1 Introduction

Ordered mesoporous carbons (OMCs) possess uniform pores of diameter in the range between 2 and 50 nm, which form 2D or 3D ordered networks. These carbons attract significant attention because of their potential applications in adsorption, catalysis, environmental cleanup, gas and energy storage (Liang et al. 2008; Choma et al. 2008b, 2008c; Ryoo et al. 2001; Wan et al. 2008). There are numerous ways to synthesize mesoporous carbons, including (i) physical activation of carbons to achieve a high burn off degree, (ii) combination of physical and chemical activation, (iii) carbonization of polymer blends with one unstable component, (iv) carbonization of polymer aerogels or cryogels, (v) use of porous silica particles as hard templates, and (vi) preparation of multiwall carbon nanotubes and their assembly. A great interest in the area of mesoporous carbons has been initiated by Ryoo et al. (1999) reporting the synthesis of ordered mesoporous carbon (OMC) by using ordered mesoporous silica (OMS), MCM-48 (*Ia3d* symmetry group), as a hard template. In this synthesis MCM-48 was impregnated with aqueous solution of sucrose with added sulfuric acid as a catalyst of carbonization, which was performed at high temperatures in the range of 800–1100 °C. The siliceous template was removed from the carbon-silica composite by using NaOH solution. The resulting OMC, denoted as CMK-1, exhibited mesopores of about 3 nm. This synthesis initiated extensive studies in the inverse replication of OMSs in order to obtain OMCs and other ordered mesoporous materials (Ryoo et al. 2001; Kang et al. 2002; Kim et al. 2005; Li and Jaroniec 2004; Fulvio et al. 2009).

In contrast to the work of Ryoo et al. (1999) the strategy proposed by Tanaka et al. (2005) eliminated the need of using hard siliceous templates, which resulted in the reduction

of the number of synthesis steps and simplified the entire process making it feasible from industrial viewpoint. This new strategy, known as soft-templating synthesis, is based on the organic–organic self-assembly, in which one organic compound, usually poly(ethylene oxide)–poly(propylene oxide)–poly(ethylene oxide) triblock copolymer (PEO-PPO-PEO), is used as a soft template, and the remaining organics are incorporated into hydrophilic nanodomains of the template and polymerize to form a polymeric-type carbon precursor. The resulting polymer-polymer composite, after thermal removal of soft template and carbonization of phenolic resin in neutral atmosphere (nitrogen), was converted to OMC (Meng et al. 2005, 2006; Liang and Dai 2006; Lin et al. 2006; Choma et al. 2008a; Górka et al. 2008; Liang et al. 2008; Liu et al. 2008; Górka and Jaroniec 2008; Wang et al. 2008).

The thermosetting polymer (carbon precursor) was obtained by polycondensation of phenol, resorcinol, or phloroglucinol with formaldehyde, crotonaldehyde or furfural in the presence of poly(ethylene oxide)–poly(propylene oxide)–poly(ethylene oxide) triblock copolymer template (Meng et al. 2005; Lin et al. 2006; Choma et al. 2008a; Górka et al. 2008). The resulting soft-templated carbons are mesoporous materials with very small fraction of micropores. The amount of micropores can be increased by additional activation. It was shown (Górka et al. 2008; Choma et al. 2010) that the KOH activation at temperatures 550–800 °C caused a significant development of microporosity, which resulted in the substantial increase of the BET surface area and total pore volume.

In this work a series of mesoporous carbons was synthesized by polymerization of resorcinol and formaldehyde in the presence of mixed templates consisting of poly(ethylene oxide)–poly(propylene oxide)–poly(ethylene oxide) triblock copolymer (Pluronic 123) and polyoxyethylene(20) cetyl ether (Brij 58) or polyoxyethylene(20) stearyl ether (Brij 78).

It was found that the use of mixed templates promotes the micropore formation and/or leads to the enlargement of primary mesopores, which is an interesting alternative to the post-synthesis activation. The BET surface area, pore volume and pore size distribution of the resulting mesoporous carbons were evaluated by nitrogen adsorption.

## 2 Experimental

*Chemicals.* Mesoporous carbons were synthesized using: poly(ethylene oxide)–poly(propylene oxide)–poly(ethylene oxide) triblock copolymer ($EO_{106}PO_{70}EO_{106}$; Pluronic F127) provided by BASF Corp. Germany; resorcinol ($C_6H_4(OH)_2$; 98%), formaldehyde (HCHO), polyoxyethylene(20) cetyl ether ($C_{56}H_{114}O_{21}$, Brij 58), and polyoxyethylene(20) stearyl ether ($C_{58}H_{118}O_{21}$, Brij 78) purchased from Acros Organics, USA; HCl (35–38%) acquired from Fischer, USA and ethanol (95%) from Pharmco., USA.

*Synthesis of mesoporous carbons.* Mesoporous carbons were prepared according to a slightly modified recipe of Wang et al. (2008). In a typical synthesis, 1.25 g of resorcinol and the specified amounts of poly(ethylene oxide)–poly(propylene oxide)–poly(ethylene oxide) triblock copolymer (Pluronic F127) and polyoxyethylene(20) cetyl ether (Brij-58) or polyoxyethylene(20) stearyl ether (Brij 78) were dissolved in 8.1 g of ethanol-water (10:7 wt ratio) solution, to achieve the desired concentration of both copolymers. The F127:Brij 58 molar compositions were 100%:0%, 80%:20%, 75%:25%, 23%:77%, 10%:90% or 0%:100%, which correspond to the F127:Brij 58 ratios: 1:0, 4:1, 3:1, 0.3:1, 1:9, 0:1. In the case of series prepared with Brij 78, the F127:Brij 78 molar compositions were 100%:0%, 90%:10%, 75%:25%, 50%:50%, 23%:77%, 10%:90% or 0%:100%, which correspond to the F127:Brij 78 ratios: 1:0, 9:1, 3:1, 1:1, 0.3:1, 1:9, 0:1. The resulting mixture was stirred vigorously at room temperature. After complete copolymer dissolution, 1.1 mL of 37% HCl was added to the solution as a catalyst and stirred for additional 30 min. Next, the synthesis mixture was supplied with 1.25 mL of 37% formaldehyde, stirred until turned cloudy (45 min–1.5 h) and allowed to separate into two layers (usually ∼1–2 h). The polymer-rich bottom layer was spread onto Petri dish and transferred to an oven at 100 °C for 24 h. Thermal treatment and carbonization of the resulting film were performed in the tube furnace under nitrogen flow using a heating rate of 2 °C/min up to 180 °C, keeping the sample at this temperature for 5 h, resuming heating with 2 °C/min up to 400 °C and with 5 °C/min up to 850 °C, and finally keeping the sample at 850 °C for 2 h. The samples were labeled respectively to the concentration of Brij 58 used in the synthesis: B58-0%, B58-20%, B58-25%, B58-77%, B58-90% and B58-100% and to the concentration of Brij 78 used in the synthesis: B78-0%, B78-10%, B78-25%, B78-50%, B78-77%, B78-90% and B78-100%. Labels B58-0% and B78-0% refer to the same carbon templated by Pluronic F127 only.

*Measurements.* Nitrogen adsorption isotherms were measured at −196 °C, using ASAP 2010 volumetric analyzer manufactured by Micromeritics, Inc. (Norcross, GA, USA). Prior to each adsorption measurement all samples were outgassed at 200 °C for 2 h.

*Calculations.* Parameters of mesoporous carbons were determined on the basis of experimental nitrogen adsorption isotherms. The BET specific surface area, $S_{BET}$, was calculated in the relative pressure range of 0.05–0.2 (Gregg and Sing 1982) assuming the cross-sectional area for nitrogen molecule equal to 0.162 $nm^2$. The total pore volume, $V_t$, was estimated from amount adsorbed at a relative pressure $p/p_o$ of $\sim$ 0.99 (Gregg and Sing 1982).

Porous structure of materials was analyzed using the $\alpha_s$-plot method (Gregg and Sing 1982; Jaroniec and Kaneko 1997; Kruk et al. 1998), where $\alpha_s$ denotes the standard reduced adsorption on the reference solid defined as the ratio of the amount adsorbed at a given relative pressure to the amount adsorbed at the relative pressure = 0.4. The $\alpha_s$-plots for the carbons studied were obtained using nitrogen adsorption isotherm for non-graphitized carbon black Cabot BP280 reported by Kruk et al. (1997a) as reference data. These plots were used to obtain the total volume of micropores and mesopores, $V_{mi} + V_{me}$, in the range of $\alpha_s$ from 2 to 4. The volume of micropores, $V_{mi}$, was estimated in the range of $\alpha_s$ from 0.8 to 1.2. The difference between two last volumes was used to determine the mesopore volume, $V_{me}$.

The pore size distributions (PSDs) were obtained from adsorption branch of the nitrogen adsorption–desorption isotherm by using the Barrett-Joyner-Halenda (BJH) procedure (Barrett et al. 1951) developed for cylindrical mesopores. In contrast to the original work (Barrett et al. 1951) this study employed the statistical film thickness for nitrogen on the Cabot BP280 carbon black (Kruk et al. 1997a), which was obtained by fitting nitrogen adsorption isotherm on this carbon in the multilayer range to the statistical film thickness established on the basis of adsorption data for the MCM-41 silicas (Kruk et al. 1997b). Also, an improved Kelvin relation was used (Kruk et al. 1997b). This modified BJH method, known as KJS method (Kruk et al. 1997b), was used to obtain PSDs. The maxima on the PSD curves were used to estimate the size of micropores, $w_{mi}$, and mesopores, $w_{me}$.

## 3 Results and discussion

Synthesis of phenolic resin-based mesoporous carbons in the presence of mixed Pluronic F127 and Brij 58 or Brij 78 soft templates is illustrated on Fig. 1. As can be seen from this scheme polymerization of resorcinol and formaldehyde in hydrophilic domains of the mesostructured template leads to the polymer–polymer composites, which during controlled thermal treatment and carbonization in nitrogen give mesoporous carbons. Figure 2 shows nitrogen adsorption isotherms for a series of carbons synthesized with Brij 58 (samples denoted as B58-$x$%, where $x$ is 0, 20, 77, 90 and 100% of Brij 58, respectively), while Fig. 3 shows the set of nitrogen adsorption isotherms for a series of carbons prepared with Brij 78 instead of Brij 58 (samples labeled B78-$x$%, where $x$ correspond to 0, 10, 50, 77, 90 and 100% of Brij 78, respectively). As can be seen from Fig. 2 the best adsorption properties in terms of high nitrogen adsorption at low relative pressures, and well-developed and steep hysteresis loop has the carbon sample, B58-77%, obtained by using 77% of Brij 58 and 23% of Pluronic F127 in the mixed template. Adsorption parameters of the remaining two samples, obtained with the help of mixed templates, B58-20% and B58-90%, are between those characterizing carbons obtained by using single templates Pluronic F127 and Brij 58, respectively. It is interesting that also in the second series (see Fig. 3), the best adsorption properties has the carbon sample, B78-77%, obtained by using 77% of Brij 78 and 23% of Pluronic F127 in the mixed template. However, the adsorption properties of remaining carbons do not change much in respect to their composition and stay close to the 100% Pluronic F127-templated carbon.

Nitrogen adsorption isotherms shown in Figs. 2 and 3 were used to calculate the BET specific surface area, $S_{\mathrm{BET}}$, and the single-point pore volume, $V_t$, for the carbon samples studied. The BET surface area varies from 339 $m^2/g$ to 825 $m^2/g$ for the B58-100% and B58-77% samples, respectively (Table 1). However, the pore volume $V_t$ changes from 0.40 $cm^3/g$ for B58-100% to 0.78 $cm^3/g$ for B58-77% (Table 1). These data indicate that the addition of about 20% of Pluronic F127 to Brij 58 double the values of the BET surface area and single-point pore volume, showing advantage

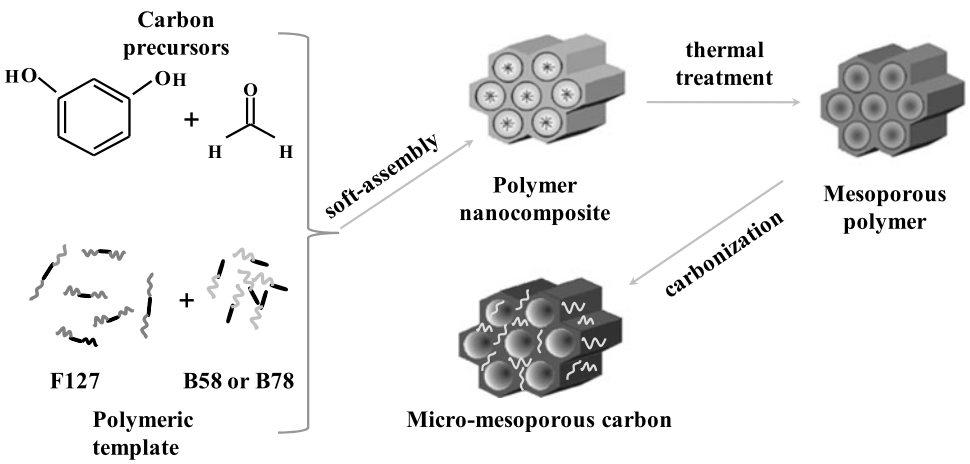


**Fig. 1** Schematic illustration of synthesis of mesoporous carbons obtained by using mixed soft templates consisting of Pluronic F127 and Brij 58 or Brij 78

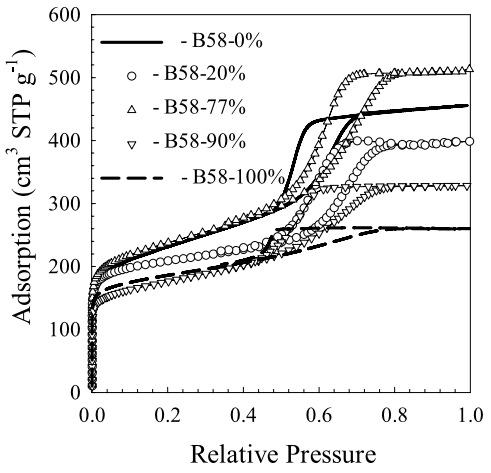


**Fig. 2** Nitrogen adsorption isotherms for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58

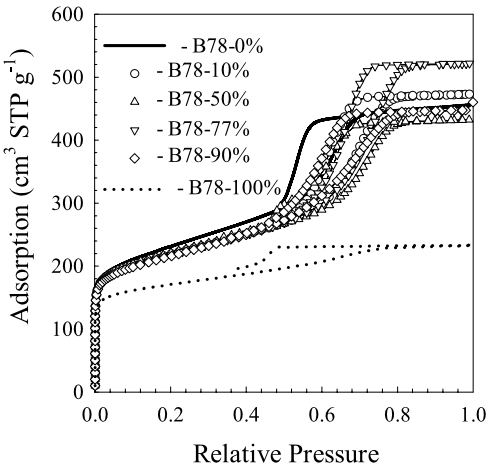


**Fig. 3** Nitrogen adsorption isotherms for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 78

of using mixed templates for the synthesis of mesoporous carbons. In the case of series of samples prepared with Brij-78, the BET surface area does not vary as much as in previously discussed series and stays in the range from 588 $m^2/g$ to 813 $m^2/g$ for the B78-100% and B78-0% samples, respectively (Table 2). Comparing both series of samples, the total pore volumes $V_t$ show similar values for the same percentage of the co-surfactant used.

The $\alpha_s$-plots were used for the estimation of the volumes of micropores, $V_{mi}$ and mesopores, $V_{me}$. Linear segments of these plots in the range of $\alpha_s$ from 0.8 to 1.2 were used to evaluate $V_{mi}$ for the carbons studied (Gregg and Sing 1982; Jaroniec and Kaneko 1997); however, linear segments in the range of $\alpha_s$ from 2 to 4 were employed to estimate the total volume of micropores and mesopores, $V_{mi} + V_{me}$ (Gregg and Sing 1982; Jaroniec and Kaneko 1997). These data and their difference, which represents the mesopore

**Table 1** Adsorption parameters of mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58*

| Mesoporous carbons | $S_{BET}$ $m^2/g$ | $V_t$ $cm^3/g$ | $V_{mi}$ $cm^3/g$ | $V_{me}$ $cm^3/g$ | $w_{mi}$ nm | $w_{me}$ nm |
|---|---|---|---|---|---|---|
| B58-0% | 813 | 0.71 | 0.15 | 0.54 | 1.45 | 5.97 |
| B58-20% | 718 | 0.62 | 0.22 | 0.40 | 1.41 | 6.65 |
| B58-25%** | 724 | 0.62 | 0.21 | 0.41 | 1.44 | 7.29 |
| B58-77% | 825 | 0.78 | 0.16 | 0.62 | 1.45 | 7.11 |
| B58-90% | 610 | 0.51 | 0.11 | 0.40 | 1.24 | 5.72 |
| B58-100% | 641 | 0.40 | 0.17 | 0.23 | 1.45 | 4.00 |

$S_{BET}$—BET specific surface area; $V_t$—single-point total pore volume calculated for $p/p_o = 0.99$; $V_{mi}$—volume of micropores obtained by $\alpha_s$-method; $V_{me}$—volume of mesopores obtained by $\alpha_s$-method; $w_{mi}$ and $w_{me}$—micropore and mesopore diameters at the maxima of the PSD curve

*Data for mesoporous carbons prepared in the presence of Brij-58 and Pluronic F127 were partially presented at the 16th Polish Zeolite Forum in Skorzecin, Poland, June 29–July 3, 2009

**This carbon is not shown in Figs. 2 and 6 but in Fig. 9

**Table 2** Adsorption parameters of mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 78

| Mesoporous carbons | $S_{BET}$ $m^2/g$ | $V_t$ $cm^3/g$ | $V_{mi}$ $cm^3/g$ | $V_{me}$ $cm^3/g$ | $w_{mi}$ nm | $w_{me}$ nm |
|---|---|---|---|---|---|---|
| B78-0% | 813 | 0.71 | 0.15 | 0.54 | 1.45 | 5.97 |
| B78-10% | 760 | 0.73 | 0.17 | 0.56 | 1.45 | 6.88 |
| B78-25%* | 718 | 0.70 | 0.19 | 0.51 | 1.33 | 7.80 |
| B78-50% | 767 | 0.67 | 0.19 | 0.48 | 1.45 | 7.54 |
| B78-77% | 755 | 0.81 | 0.15 | 0.65 | 1.48 | 7.85 |
| B78-90% | 749 | 0.69 | 0.13 | 0.55 | 1.47 | 6.46 |
| B78-100% | 588 | 0.36 | 0.18 | 0.18 | 1.17 | 5.68 |

$S_{BET}$—BET specific surface area; $V_t$—single-point total pore volume calculated for $p/p_o = 0.99$; $V_{mi}$—volume of micropores obtained by $\alpha_s$-method; $V_{me}$—volume of mesopores obtained by $\alpha_s$-method; $w_{mi}$ and $w_{me}$—micropore and mesopore diameters at the maxima of the PSD curve

*This carbon is not shown in Figs. 3 and 7 but in Fig. 9

volume, $V_{me}$, are summarized in Tables 1 and 2. Tables 1 and 2 indicate that the adsorption properties of the samples synthesized by using mixed templates or Pluronic F127 are much better than those obtained for the Brij 58 or Brij 78-templated carbons. Interestingly, the use of mixed templates of Brij 58 or Brij 78 and Pluronic F127 improves slightly microporosity in comparison to that present in the carbon templated by Pluronic F127 only. This result is promising if one considers that the phenolic resin-based carbons templated by Pluronic F127 are mainly mesoporous (Górka et al. 2008).

Shown in Fig. 4 are the changes in the specific surface areas in respect to increasing percentage of Brij 58 or 78 in

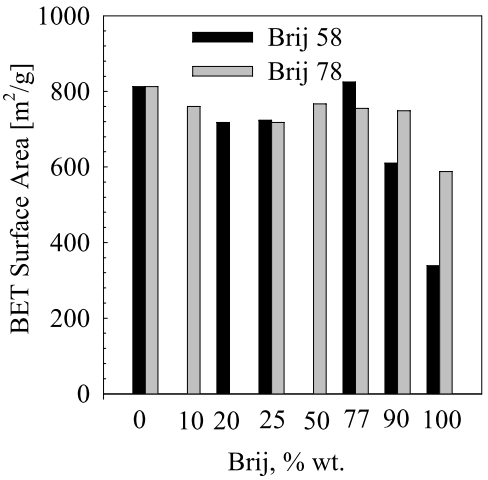


**Fig. 4** Comparison of the BET surface area for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58 or Brij 78

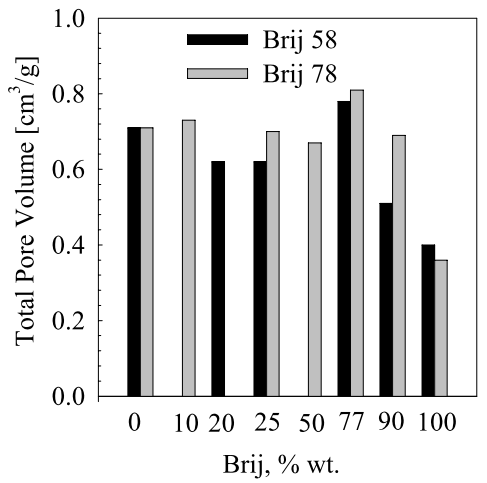


**Fig. 5** Comparison of the total pore volume for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58 or Brij 78

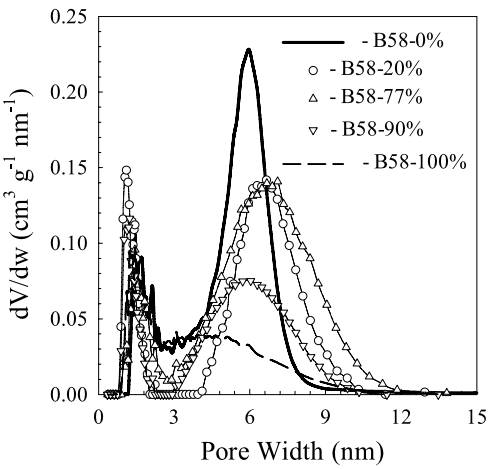


**Fig. 6** Pore size distributions (PSDs) for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58

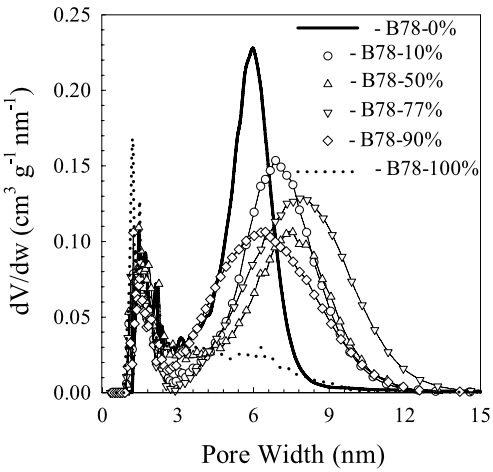


**Fig. 7** Pore size distributions (PSDs) for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 78

the mixed templates used. Although the changes are rather mild we can easily distinguish that the maximum surface area (825 $m^2/g$) corresponds to the template mixture composed of ∼77% of Brij 58 and 23% of Pluronic F127. Analogous results are obtained comparing the total pore volume and the percentage composition of the mixed template (see Fig. 5). In this case the maximum of the total pore volume also corresponds to the sample, where Brij 58/78 constitutes 77% of the template used.

Figures 6 and 7 shows pore size distributions (PSD) for the carbons studied. These PSD curves were calculated from adsorption branches of nitrogen adsorption-desorption isotherms using the Barrett, Joyner and Halenda (BJH) (Barrett et al. 1951) method modified substantially by Kruk, Jaroniec and Sayari (KJS) (Kruk et al. 1997b). These distributions possess two peaks reflecting the presence of micropores and mesopores. The widths of micropores and mesopores corresponding to the suitable peaks on the PSD curves are summarized in Tables 1 and 2. As can be seen from these tables the mesopore width shows a tendency of increase with increasing amount of Brij 58 or Brij 78 in the mixed template; however, the smallest mesopore width was obtained for the carbon templated by Brij 58 or Brij 78 only. This behavior suggests that Brij 58 or Brij 78 act rather as micelle expanders.

The comparison of the mesopore width as a function of the template composition is shown in Fig. 8. Interestingly, the biggest mesopores around 8 nm were afforded when the percentage of Brij in the mixed templates was in the range from 25 to 77%. The samples prepared from single template (0 and 100% of Brij 58/78, respectively) are characterized by smaller mesopores usually between 4 and 6 nm.

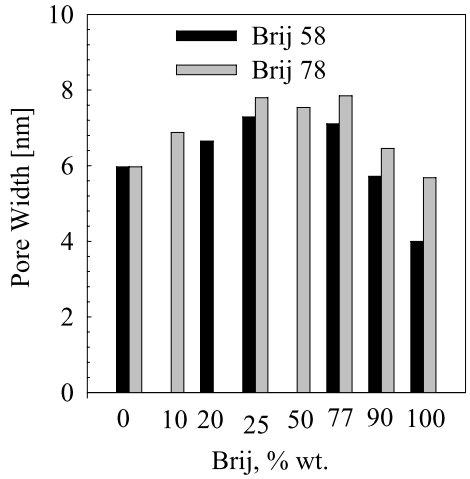


**Fig. 8** Comparison of the pore width for mesoporous carbons obtained by using mixed templates of Pluronic F127 and Brij 58 or Brij 78

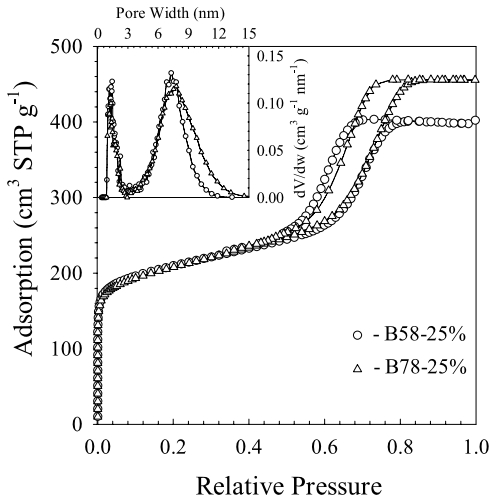


**Fig. 9** Comparison of nitrogen adsorption isotherms and the corresponding pore size distributions for mesoporous carbons obtained by using mixed templates of Pluronic F127 (75% wt) and Brij 58 (25% wt) or Brij 78 (25% wt)

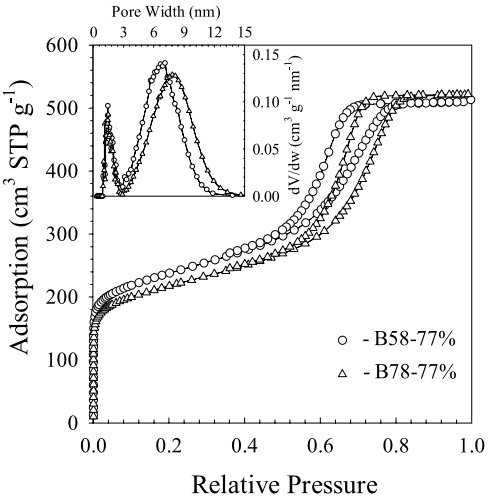


**Fig. 10** Comparison of nitrogen adsorption isotherms and the corresponding pore size distributions for mesoporous carbons obtained by using mixed templates of Pluronic F127 (23% wt) and Brij 58 (77% wt) or Brij 78 (77% wt)

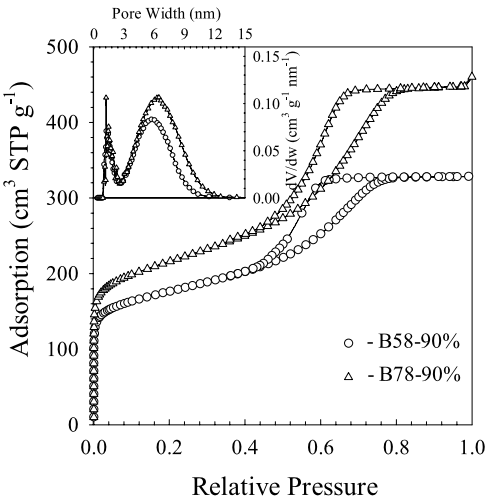


**Fig. 11** Comparison of nitrogen adsorption isotherms and the corresponding pore size distributions for mesoporous carbons obtained by using mixed templates of Pluronic F127 (10% wt) and Brij 58 (90% wt) or Brij 78 (90% wt)

Based on the structural parameters obtained for the carbons prepared from Brij 58-Pluronic F127 (see Table 1) vs. Brij 78-Pluronic F127 (see Table 2), the latter system seems to lead to much better carbons in terms of adsorption properties, as shown in Figs. 9–11, where the nitrogen adsorption isotherms and the corresponding pore size distributions are compared for the carbons prepared with the same Brij to Pluronic F127 ratio. The C-18 hydrophobic alkyl chain of Brij 78 seems to interact stronger with hydrophobic domains of Pluronic F127 triblock copolymer than C-16 alkyl chain from Brij-58, which results in better mesoporosity development in the final carbons. The observed irregularity in the sequence of adsorption isotherms for the carbons prepared with increasing percentage of Brij 58 or Brij 78 in the mixed template is probably caused by competing effects of polymer interactions and micelle expansion. Analysis of these adsorption isotherms suggest that Pluronic F127 triblock copolymer is mainly a pore forming agent, while Brij 58 or Brij 78 may act as micelle expander because its addition to Pluronic F127 led to the pore size enlargement. In addition, Brij 58 and Brij 78 seem to promote the formation of microporous structure because the resulting carbons possess a larger fraction of micropores in comparison to that observed in the carbons templated by Pluronic F127 only.

## 4 Conclusions

Adsorption properties of mesoporous carbons synthesized by polymerization of resorcinol and formaldehyde in the presence of mixed templates of Pluronic F127 and Brij 58 or Brij 78 were studied. It is shown the use of mixed templates of Pluronic F127 and Brij 58 or Brij 78 afforded mesoporous carbons with improved microporosity, high surface area (exceeding 800 $m^2/g$), and large pore volume (close to 0.8 $cm^3/g$). Pore size distributions consist of two peaks reflecting the presence of mesopores and micropores. The mesopore width showed a tendency of increase with increasing amount of Brij 58 or Brij 78 in the mixed template, suggesting that Brij 58 and Brij 78 act as micelle expander.

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