



A Simple Method for the Synthesis of High Surface Area Mesoporous Carbon Monolith via Soft Template Technique

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Abstract

The aim of this work was the synthesis and characterization of a mesoporous carbon monolith (MC). The MC was prepared using the soft template method. Resorcinol and formaldehyde were used as the carbon precursor and triblock copolymer Pluronic F127 as the templating agent under acidic conditions. Several techniques were used to characterize the synthesized material such as Scanning Electron Microscopy (SEM), Brunauer-Emmett-Teller nitrogen sorption measurements (BET), Powder X-Ray Diffraction (PXRD), Fourier Transform Infrared spectroscopy (FTIR), and Thermogravimetric Analysis (TGA). The work characterized the changes due to the decomposition of the template during the carbonization process to form the pores in the carbon material. The result confirmed that the mesoporous monolithic carbon prepared had a high surface area and a narrow pore size in the mesometer range.

Keywords: Mesoporous carbon monolith, triblock copolymer, self-assembly, surface area.

1. Introduction

Porous carbon materials, are a big group of porous materials that have been studied due to their multiple applications in many fields [1-9]. Several researchers have been working on the development of innovative carbon-based materials. Porous carbons materials include a broad spectrum of carbons ranging from microporous to mesoporous such as activated carbon, carbon nanotubes, carbon particles, carbon aerogels, and carbon composites. The properties of each group of porous carbon materials reflected by their porous structure and surface chemistry are influenced by both the nature of carbon precursors and the methods of carbonization and activation [10,11].

Mesoporous carbon materials are a type of porous materials with pores size between 2 nm and 50 nm.

These materials are promising material received widespread attention due to their high surface area, excellent physical and chemical properties and thermal stability. Therefore, mesoporous carbon materials can be used in energy conversion and storage devices, catalytic support and drug delivery system [12]. Moreover, mesoporous carbon materials can be used for wastewater treatment due to their ability to adsorb and removal of many types of organic contaminations and potentially toxic metals from water [13].

There are several carbon sources, for example, biomass [14], natural graphite, thermosetting polymers, bio-waste, etc., that have been used for the preparation of different types of carbon materials. In particular, thermosetting polymers, e.g. phenolic resin-based polymers have been most commonly used due to their easy synthesis, cost effectiveness,

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and industrial attainability. In addition, they are highly versatile, having been adapted for the creation of various carbonaceous materials, for example, nanotubes [15], mesoporous carbons materials [16] and carbon aerogels [17].

One of the most commonly used methods for the synthesis of mesoporous carbon monolith is the "hard-templating method" [18-22]. This route briefly includes three steps. Firstly, the preparation of an ordered mesoporous silica followed by filling the pores of the template with suitable carbon precursors. Following this, carbonisation of the precursors and

removal of the silica template is carried out. The resulting carbon materials are inverse replicas of the template and exhibit a high surface area, large pore volume and ordered mesopores. The adsorption properties of these materials can be easily tuned by using different silica meso-structures as templates. Despite the fact that these strategies have been widely used, they have vital drawbacks. Firstly, they have several synthetic steps and are costly due to the preparation of the colloidal silica particles and silica porous. Moreover, using chemicals such as hydrofluoric acid (highly dangerous and limited to specially trained personnel) or sodium hydroxide solution (corrosive and environmentally unfriendly) for silica removal is undesirable and renders this method impractical for scale up [23]. Many researchers have therefore used a soft template method for the synthesis of mesoporous carbon materials via self-assembly of organic carbon sources such as phenol, resorcinol, and phloroglucinol in the presence of formaldehyde as a cross-linker and triblock copolymers [24]. Self-assembly of organic carbon sources such as phenolic resins and block copolymers has opened another option in the preparation of mesoporous carbon materials by using soft template route [25-28]. This methodology exploits the advantage of the interaction between thermally-decomposable surfactants and thermosetting polymers to construct ordered polymer-polymer nanocomposites. Through initial heating, the thermally unstable soft template agent (triblock copolymer) is decomposed [29]. Increasing the temperature carbonizes the thermosetting polymer to form the pore walls of porous carbon materials. In the soft templating method, fewer steps are needed compared to the hard templating route. This method

also avoids the use of hazardous materials, thereby decreasing the negative impact on the environment [30].

Evaporation-induced self-assembly (EISA) technique is the most effective and simple method utilized for the synthesis of mesoporous carbon monolith with ordered pores size [31-32], but its drawbacks include material shrinkage and crack formation, thus decreasing its applicability [33].

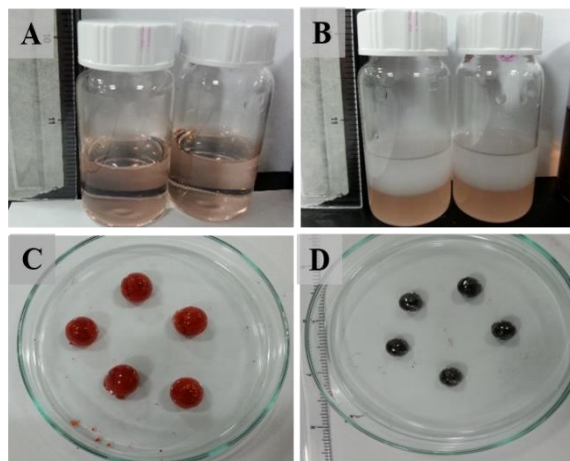


Fig. 2. Photographs show the preparation steps of mesoporous carbon material (A) sol solution (B) phase separation solution after kept the sol solution for 72 hours (C) dried polymer monolith (D) carbon monolith.

These materials can be prepared by different methods, firstly, varying the solvent system from mixture solvent mixture containing water-ethanol at different ratio [34-35] to ethanol solvent [30] or completely aqueous solvent [36]. Secondly, changing the concentration of triblock copolymer or PEO/PPO.

2. Experimental

3. Materials

Resorcinol with 99% purity was used as carbon precursor, triblock copolymer pluronic F127 was used as a soft template agent and had an average molecular weight of 12,600 these were purchased from Sigma-Aldrich. Formaldehyde (37%), used as the cross-linker agent to synthesize mesoporous carbon materials, was purchased from Scientific Fisher, UK. Absolute ethanol ($\geq 99.8\%$) and hydrochloric acid (37%) were both of analytical grade and purchased from Scientific Fisher, all these chemicals were used without any further purification.

3.1. Methods The mesoporous carbon monolith (MC) were prepared based on a modified version of the method described by Liu et al [37]. 1.65 g resorcinol (R) and 2.5 g Pluronic F127 were dissolved in 14 g of ethanol/ water mixture solvent (1/1 v/v %) under magnetic stirring at 25°C temperature. This was followed by the addition of 0.23 g hydrochloric acid to the above solution after stirring for about 30 min and followed by stirring for 2 hours at room temperature (22 ± 2 °C). The colour of the solution changed from colourless to light brown.

After stirring for two hours, 2.3 g of formaldehyde (37%) was subsequently added dropwise to the solution under vigorously stirring. The reaction solution mixture was stirred for another two hours. The final homogeneous solution was kept standing for a further 72 hours for aging. In this period the solution turned cloudy and the phases separated into two layers. The upper layer was composed of the solvents, and the bottom layer was rich in the polymer. Subsequently, the upper layer was removed while the lower layer was stirred for 12 hours until a sticky polymer was created. Then, the polymer was transferred to a range of different shaped moulds and cured at 85 °C for 24 hours to remove the residual solvent. Finally, the dried polymer was carbonized in a tubular furnace under nitrogen gas using a heating programme starting from 25 °C to 400 °C at a ramping rate of 1°C/min while the temperature increased from 400 °C to 800 °C at a heating rate of 5 °C/min and kept at the final temperature for two hours. The resulting heat-treated composite material was prepared to be mesoporous carbon monolith [37-40]. Figure 1 shows the schematic illustration of the preparation method of the MC materials and Figure 2 show photographs of the preparation stages of the MC materials.

4. Results and discussion

4.1. Blank ordered mesoporous carbon materials

The soft template route was used to synthesize mesoporous carbon materials. The synthesis mechanism reflected the strong interaction between the reaction substances as shown in Figure 3.

The formation of the mesoporous carbon monolith involves two steps. Firstly, polymerization gradually happens between the resorcinol and formaldehyde to form soluble oligomers (RF) with plenty of hydroxyl groups. The main reactions between the phenolic monomer and formaldehyde form covalent crosslinks through an addition reaction to produce methylene and hydroxymethyl bridges [41]. Secondly, the hydroxyl groups are able to interact with the surfactant triblock copolymer (pluronic F127) by hydrogen bonding [28]. The use of resorcinol and formaldehyde as a carbon precursor is vital for the successful association of organic–organic mesostructured due to it having a number of hydroxyl groups, which can interact strongly with Pluronic F127 via hydrogen bonds.

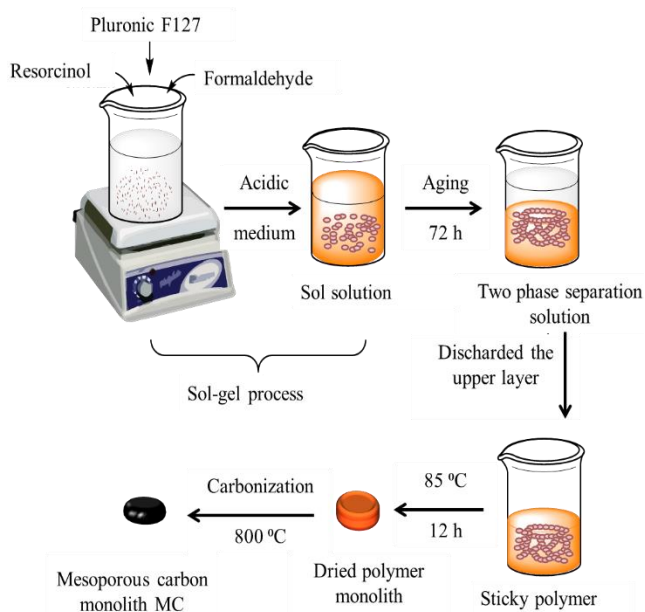


Fig. 1. Schematic illustration for preparation of mesoporous carbon materials using the soft template method.

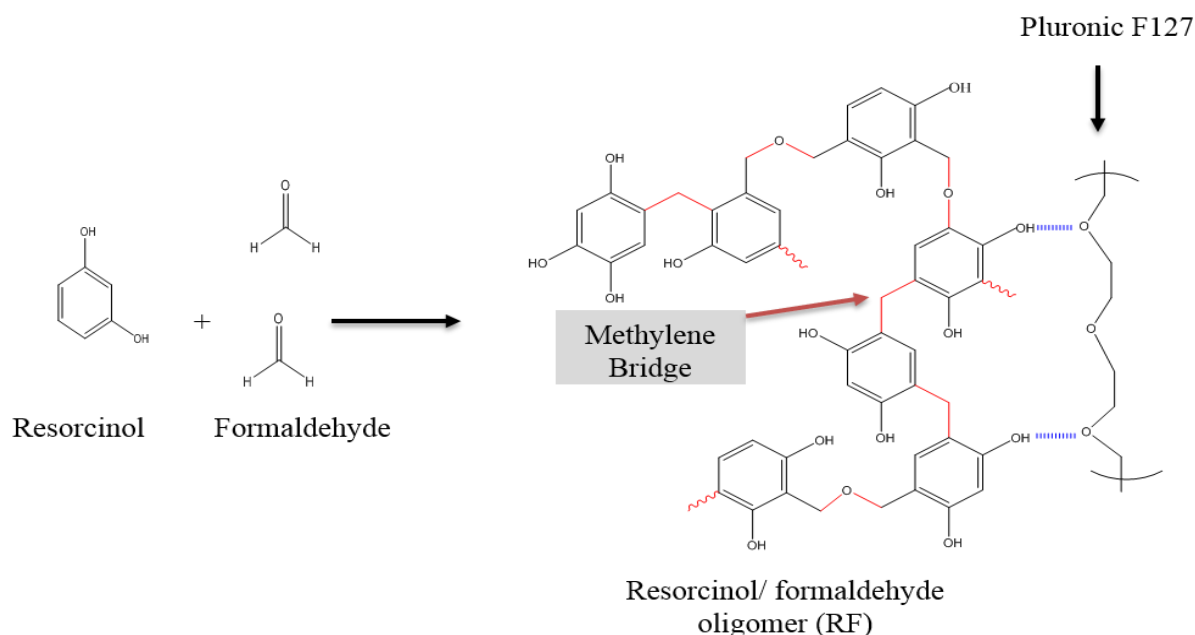


Fig. 3. Schematic of the reaction of resorcinol with formaldehyde and interaction between the F127 and the hydroxyl group in the RF via hydrogen bonding.

The carbon monoliths obtained after carbonisation at 800 °C for two hours are shown in Figure 4B. The polymer monoliths can be shaped in different forms

depending on the shape of the moulds. It can be seen that the polymer monolith exhibited shrinkage after carbonisation. The volume decreased by approximately 50% after carbonization and the colour changed from orange to black, but the monolithic morphology was still maintained.

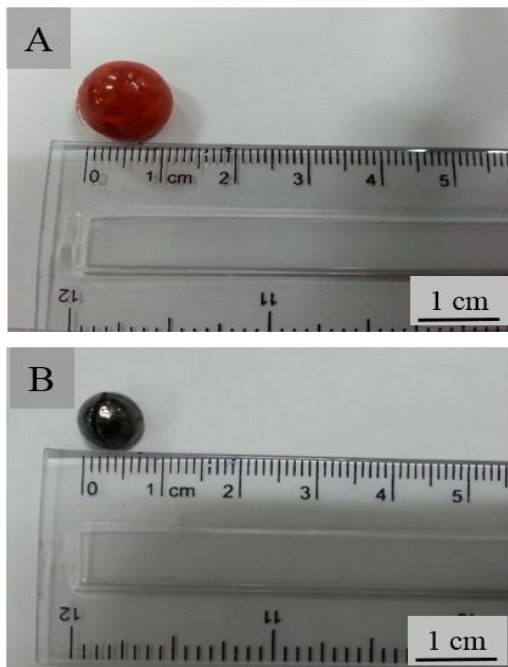


Fig. 4. Photographic images of synthesised (A) polymer monolith (B) carbon monolith using resorcinol and formaldehyde as a carbon precursor.

4.2. SEM Analysis

Scanning electron microscopy (SEM) provided information about the structural morphologies of the different materials. The SEM images of the polymer monolith and mesoporous carbon monolith (MC) are shown in Figure 5A and 5B respectively. The SEM image of the polymer monolith showed a smooth surface and confirmed that this material was non-porous (at the magnifications imaged), but the SEM of the carbon material prepared by carbonization of the same polymer monolith at 800 °C showed pores with different sizes. These pores resulted from the decomposition of the soft template (triblock copolymer F127) and the carbonization the polymer at a high temperature with the carbon precursor remaining as the wall of these pores [42].

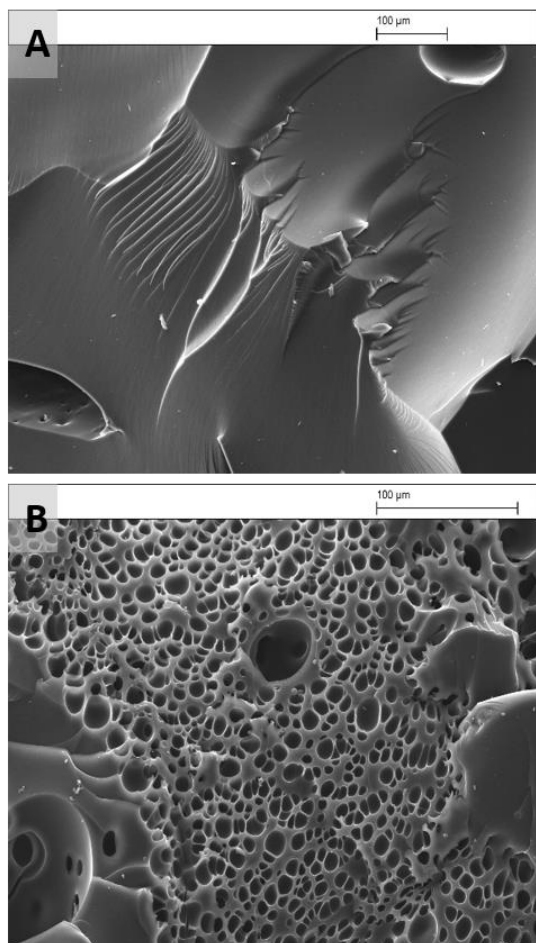


Fig. 5. Scanning Electron Microscopy (SEM) images of (A) the polymer monolith (B) the mesoporous carbon monolith MC.

3.3. BET surface area analysis

The BET (Brunauer–Emmett–Teller) surface area and porosity analyser was used in the determination of the surface area and porosity of polymer monolith and carbon monolith using nitrogen sorption measurements. Figure 6A shows the N_2 adsorption-desorption isotherms of the polymer monolith. The polymer monolith showed a Type II isotherm, which is a typical isotherm for a non-porous material [43]. The sorption isotherm of the MC shown in Figure 6B demonstrated a Type-IV isotherm, which is a characteristic of mesoporous materials [44]. The pore size distribution of the carbon monolith is shown in Figure 6C. It has a narrow pore size distribution with an average of 7.6 nm and BET specific surface area of $635 \pm 6 \text{ m}^2/\text{g}$ which is in the mesopore range. The increase in the amount of gas adsorbed at relative low

pressures ($P/P_0 < 0.1$) for the carbon monolith confirms the presence of the micropores in the framework [45].

Surface EDX analysis was carried out on cross sections of mesoporous carbon materials as shown in Figure 6D. EDX confirms the presence of high percentage of carbon element and a small percent of oxygen was also revealed in the mesoporous carbon sample, because of some surface oxidation [47].

ATR-FTIR provided further information on the chemical composition of the carbon materials. Figure 7 shows the ATR-FTIR spectra of the polymer monolith (red) and mesoporous carbon monolith (blue). The spectra have various shapes, referring to an evident change in the network structure due to the carbonization process. The main functional groups determined from the previous literature [44, 45].

A broad band at approximately $3600\text{--}3000 \text{ cm}^{-1}$ is indicative of --OH stretching, due to a number of phenolic groups in the polymer. The peak at approximately $1600\text{--}1400 \text{ cm}^{-1}$ is due to the $\text{C}=\text{C}$ stretching vibration of the aromatic ring structure of phenolic resin produced by resorcinol and formaldehyde. There are two peaks at approximately $3000\text{--}2800$ and $1200\text{--}950 \text{ cm}^{-1}$ which could be appointed to the C-H and C-O stretching vibrations of the triblock copolymer F127 and the aromatic C-H due to aromatic ring of phenolic resin. These peaks have disappeared after carbonization due to the decomposition of F127 at a temperature above $400 \text{ }^\circ\text{C}$.

Powder X-Ray Diffraction (PXRD) affords information regarding the crystallinity of the carbon materials. Figure 8 shows the wide-angle XRD patterns of the carbon monolith. This clearly shows a peak at $24^\circ (2\theta)$, together with another broad peak at $44^\circ (2\theta)$ and a weak peak at $78^\circ (2\theta)$ which correspond to diffractions from the (002), (100) and (110) planes of graphite respectively. These peaks represent different phases of graphite and reflections of the graphitic pore walls of the framework. Therefore, the wide-angle XRD pattern shows a higher degree of graphitization of the carbon framework [45, 46].

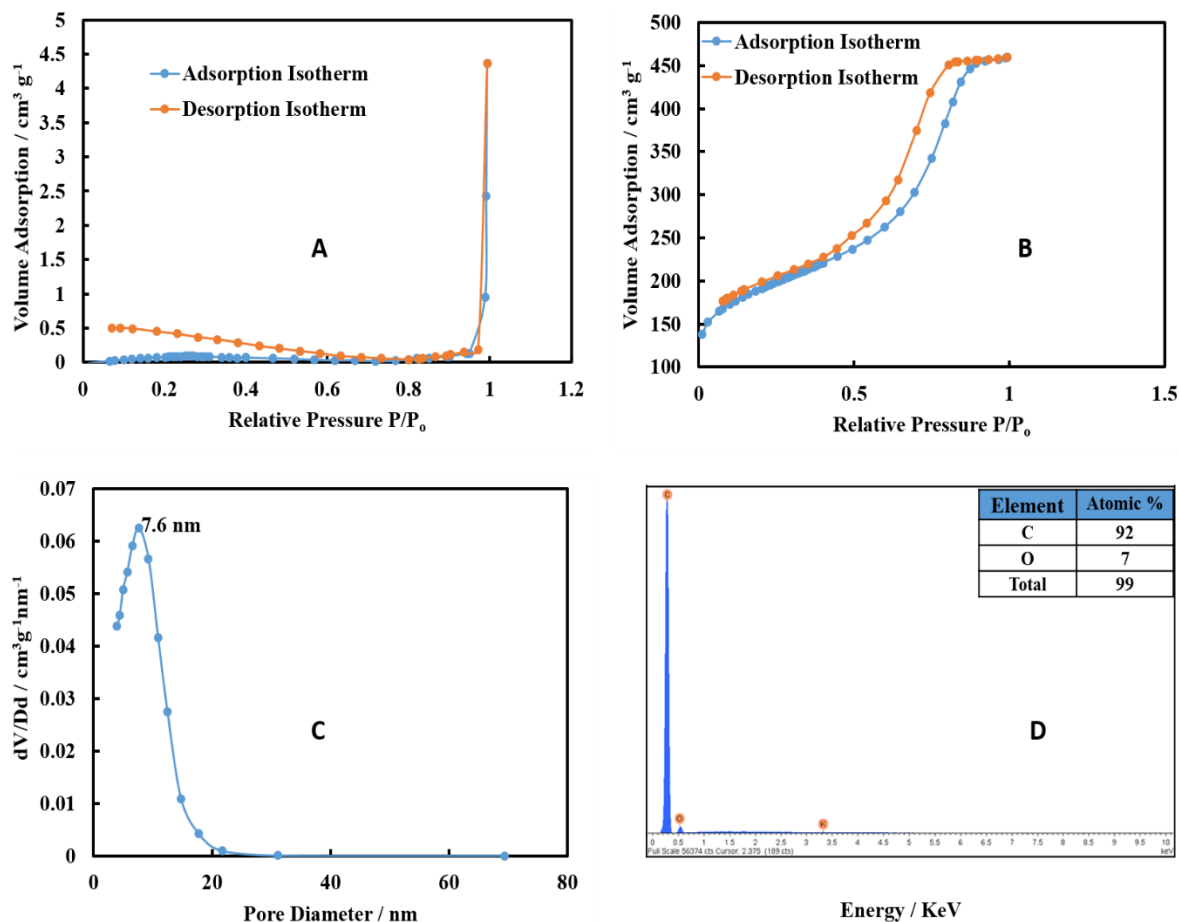


Fig. 6. (A) N₂ adsorption-desorption isotherm of the polymer monolith. (B) N₂ adsorption-desorption isotherm for carbon monolith. (C) Pore size distribution for carbon monolith. (D) EDX spectra of the carbon monolith after carbonization of the polymer monolith at 800 °C.

The TGA curves of the polymer monolith (red) and carbon monoliths (blue) under nitrogen flow are shown in Figure 9.

The TGA curve of the polymer monolith containing phenolic resin and Pluronic F127 in the temperature range 300–400 °C shows approximately 60% weight loss indicating that the soft template successfully decomposed during the carbonisation process. This is due to the decomposition of Pluronic F127 at this temperature range and the phenolic resin more thermally stable forming the mesopores walls in the carbon materials as reported in other literature [48-49]. Finally, approximately 24% of carbon residue was left at 800 °C.

5. Conclusions

In this paper mesoporous carbon materials with high surface area were successfully prepared using

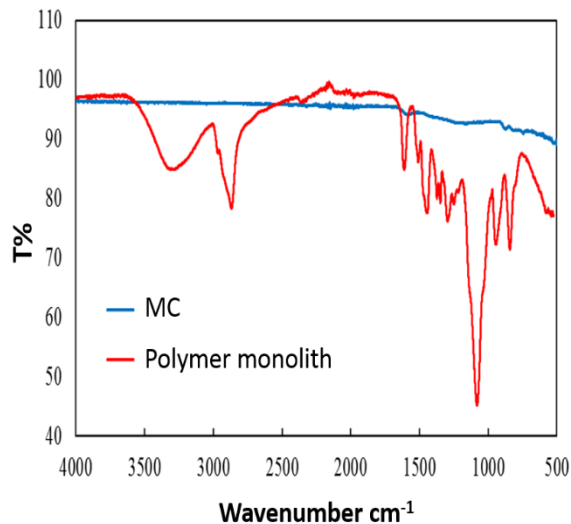


Fig. 7. ATR-FTIR spectra of the polymer monolith (red) and the porous carbon materials (blue), showing the changes in the spectra due to the decomposition of the template during the carbonization the polymer at 800 °C.

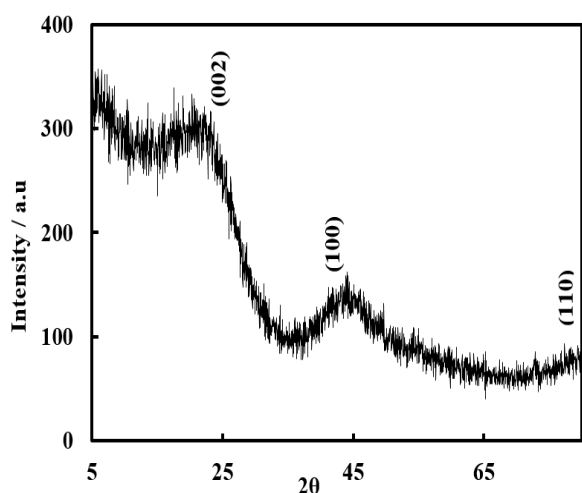


Fig. 8. Wide-angle XRD patterns for the mesoporous carbon materials prepared by soft template method.

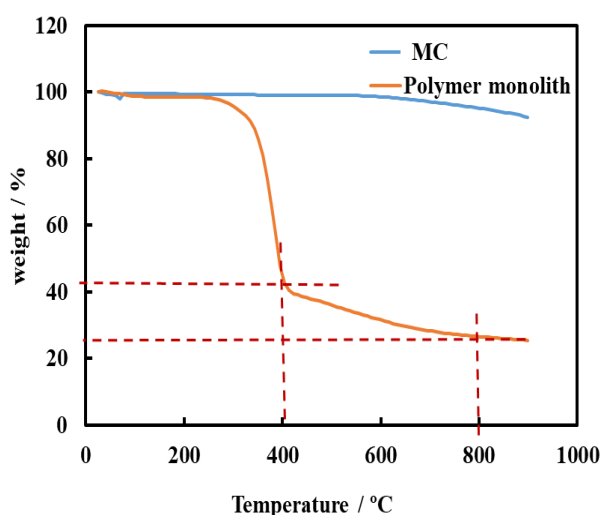


Fig. 9. Thermal gravimetric analysis (TGA) of the polymer monolith (red) and the carbon monolith (blue).

resorcinol and formaldehyde as carbon precursors and triblock copolymer F127 as the template via the soft templating method. Characterization of these materials showed that the template during the carbonization process forms the pores in the carbon materials. In comparison with other methods to prepare mesoporous carbon monolith, this method is cost-effective and simple. The results confirmed that the mesoporous carbon monoliths obtained have a high surface area, narrow pore size. Therefore, mesoporous carbon materials would have potential use in environmental remediation due to their facile separation and high selective adsorption capacity.

6. Conflicts of interest

“There are no conflicts to declare”.

7. Acknowledgments

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