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Grass-Like Structured Mesoporous Carbon as an Acid Red 119 Dye Adsorbent

Yuni Krisyuningsih Krisnandi

Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Indonesia, Depok 16424, Indonesia, yuni.krisnandi@sci.ui.ac.id

Tania Priantini

Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Indonesia, Depok 16424, Indonesia

Muhammad Faisal

Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Indonesia, Depok 16424, Indonesia

Rika Tri Yunarti

Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Indonesia, Depok 16424, Indonesia

Indah Revita Saragi

Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Indonesia, Depok 16424, Indonesia

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Cover Page Footnote

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Authors

Yuni Krisyuningsih Krisnandi, Tania Priantini, Muhammad Faisal, Rika Tri Yunarti, Indah Revita Saragi, and Jarnuzi Gunlazuardi

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Yuni Krisyuningsih Krisnandi*, Tania Priantini, Muhammad Faisal, Rika Tri Yunarti, Indah Revita Saragi, and Jarnuzi Gunlazuardi

Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Indonesia,
Depok 16424, Indonesia

*E-mail: yuni.krisnandi@sci.ui.ac.id

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Abstract

Grass-like structured mesoporous carbon was prepared by the soft-template method using triblock copolymer Pluronic F127, affording an organic–organic composite structure between phloroglucinol and formaldehyde. Thermogravimetric analysis revealed that the as-synthesized mesoporous carbon is stable to 900 °C, and X-Ray Diffraction (XRD) analysis confirmed the presence of carbon material structures at 2θ values of 22.020° and 42.420°. A hysteresis loop at relative partial pressures (P/P_0) between 0.45 and 0.85 was observed in the N₂ adsorption isotherm, indicating that the solid material exhibits mesoporous characteristics, with a homogeneous pore size distribution of 8.863 nm. Scanning Electron Microscope (SEM) images revealed that the bulk carbon surface exhibits a uniform grass-like structure. Furthermore, the as-synthesized mesoporous carbon was investigated for the adsorption of a diazo dye Acid Red 119, and it exhibited an adsorption capacity of 217.7389 mg/g; this capacity is 1.53 times greater than that of commercial activated carbon. Therefore, this mesoporous carbon is a potential adsorbent for the treatment of synthetic coloring waste.

Keywords: mesoporous carbon, soft-template method, adsorbent, adsorption, grass-like structure, diazo dye Acid Red 119

Introduction

Carbon-based materials demonstrate widespread applications in industrial fields due to their wide variety of structures that permit the development of several materials for different applications [1]. Due to its characteristics of a high surface area and a pore diameter of less than 2 nm, activated carbon (AC) has widely been applied in adsorption [2], catalysis [3], purification [4], and separation [5], as well as a capacitor electrode [6]. However, uniformity with respect to the pore size, shape, and volume is crucial as these factors limit the separation ability of carbon materials for different-sized molecules [7]. Therefore, microporosity is a limiting factor for industrial application when dealing with large molecules.

The introduction of mesoporous materials (i.e., pore diameter from 2 to 50 nm) is a significant step for microporous carbon applications. Such materials increase the intrapore molecule transport and facilitate large molecule adsorption. Hence, advances in mesoporous carbon materials [8] have led to the discovery of mesoporous carbon by the hard- [9] and soft- [5] template methods. Due to its excellent properties of electrical conductivity, inertness, thermal

stability, high surface area, and superior pore structure, mesoporous carbon has been extensively used in industrial applications, such as energy storage [10], gas separation [4], CO₂ capture [11], and air and water purification [12–15]. Moreover, it is possible to control the pore size, volume, and surface area via the modification of synthetic methods.

The preparation of mesoporous carbon by the hard-template method involves the use of organic and inorganic templates, which function as a mold that replicates mesoporous carbon with marginal interaction between templates and carbon precursors [14]. Hard-template synthesis has been frequently employed for producing mesoporous carbon. However, this method is expensive, it requires an extended protocol, and it is not environmentally friendly. On the other hand, the soft-template method involves the self-assembly of organic and inorganic substances to afford nanostructures. Liang and Dai [16] have used triblock copolymer Pluronic F127 as a soft template to prepare hydrogen-bonded organic–organic composite structures between phloroglucinol and formaldehyde. Sterk et al. [1] have developed a soft-template method by utilizing organic–organic self-assembly between a block copolymer template and a carbon polymer precursor. Mesoporous

carbon synthesized by the soft-template method should exhibit a high surface area and a large size with a narrow pore size distribution.

Mesoporous carbon plays a significant role in the molecular adsorption of vitamins, dyes, medicines, and amino acids [17–20]. Initially, their application as adsorbents was investigated for the adsorption of amino acids [17]. The adsorption efficiency of a molecule is determined by the molecular weight, size, molecular geometry, solubility, polarity, and functional groups of the adsorbates.

In this study, mesoporous carbon was synthesized according to the procedures reported by Liang and Dai [16,21]. In addition, the adsorption capacity of the as-synthesized mesoporous carbon was investigated for the adsorption of diazo dye Acid Red 119 (AR119), which is commonly found in synthetic coloring agent waste. Furthermore, the adsorption capacity was compared with that of commercially available AC.

Materials and Methods

Mesoporous carbon synthesis. First, 1.25 g of phloroglucinol (Sigma-Aldrich, Germany) and 1.25 g of Pluronic F127 (Sigma-Aldrich, Germany) were mixed in 9 g of an ethanol:water (10:9) solution and vigorously stirred at room temperature until they were homogeneously dissolved. Second, 0.12 mL of HCl (37 wt%, Merck) was added, and the solution was stirred for another 30 min. Third, 1.42 mL of formaldehyde (37 wt%, Sigma-Aldrich) was added into the above solution and mixed for 1 h until two layers were formed. Next, the lower layer was extracted and stirred for 12 h. The resulting elastic, non-sticky monolith was autoclaved at 100 °C for 24 h. A tubular furnace under nitrogen was utilized for thermal treatment and carbonation processes as follows: 1) The sample was heated between 100 and 400 °C at a rate of 1 °C/min, 2) the sample was next heated between 400 and 850 °C at a rate of 5 °C/min, and 3) the sample was maintained at 850 °C for 2 h. The resulting black powder of mesoporous carbon was labeled as MC-gl, denoting grass-like mesoporous carbon.

Mesoporous carbon MC-gl characterization. The surface area of the as-synthesized MC-gl was determined by nitrogen adsorption at 77 K using a surface area analyzer (QuadraSorb Station 2, Version 5.13). Its specific surface area was calculated by the Brunauer–Emmett–Teller (BET) equation. Microporous and mesoporous volumes were analyzed by the t-plot and Barrett, Joyner, Halenda (BJH) methods, respectively. The surface morphology was observed by FE-SEM (FEI INSPECT F50). Carbonation temperature profiles and mesoporous carbon crystalline structures were examined by thermogravimetric analysis (TGA

Q500) and X-Ray Diffraction (XRD) (Axios PANalytical), respectively.

Acid Red 119 dye (AR119) adsorption capacity.

Adsorption experiments were performed in an incubator shaker (120 rpm) at room temperature using 10 mg of MC-gl and 10 mL of the AR119 solution at a concentration of 300 ppm. The optimal adsorption of AR119 was determined by the variation in the contact time, pH solution, and initial AR119 concentration. The pH was controlled by the addition of 0.1 M HCl or NaOH pellets. The adsorption activity of MC-gl was compared with that of commercial AC (coconut-based AC, Bratachem) using a 1000 ppm AR119 solution for 120 min. After adsorption, the solution was subjected to centrifugation, and the adsorbents were filtered from the solution. The initial and final AR119 concentrations were analyzed on a UV–Vis spectrophotometer (Shimadzu UV-2450) with an absorption peak at 526 nm.

The amount of adsorbed AR119 (q_e) on the adsorbent surface was calculated by the following equation:

$$q_e = \frac{(C_0 - C_e) V}{M}$$

C_0 and C_e represent the initial and final AR119 concentrations, respectively, V represents the volume, respectively, and M represents the adsorbent mass.

Results and Discussion

Mesoporous carbon MC-gl synthesis and characterization. In this study, phloroglucinol and formaldehyde were used as the carbon precursors for the synthesis of mesoporous carbon. These precursors were dissolved in a mixture of ethanol:water (10:9 wt%), and polymerization was completed in ~40 min to a few hours. HCl served as the catalyst to induce the polycondensation of these two substrates. Similar to the method reported by Liang and Dai [16], Pluronic F127 was used as an agent to direct the structure of the synthesized mesoporous carbon MC-gl. XRD patterns (Fig. 1) revealed that the synthesized mesoporous carbon (MC-gl) and AC exhibit characteristics of carbon materials: Two broad peaks were observed at 22.02° and 42.02°, corresponding to the (002) and (100) lattice planes, respectively [22]. In addition, broad peaks indicated that the as-synthesized mesoporous carbon exhibits low crystallinity and amorphous nature [23]. However, the expected peak at a low angle in the XRD pattern corresponded to ordered hexagonal mesopores, which was characteristics of the mesoporous carbon synthesized by the hard-template method.

TGA was employed to examine the carbonation temperature, decomposition process, and thermal stability of the resulting MC-gl. The TGA curve (Fig. 2 area A)

revealed a weight loss of 10% between 25 and 100 °C, corresponding to the evaporation of water and ethanol, whereas a significant weight loss of 50% was observed between 300 and 400 °C, corresponding to the decomposition of Pluronic F127 induced by the soft-template method (Fig. 2 area B). This result is in agreement with those reported by Jin et al. [24]. Another weight loss was observed from 400 °C to 800 °C, possibly corresponding to the dissociation of the covalent bonds between carbon and hydrogen or oxygen in the resulting resin polymer that leaves the carbon structure (Fig. 2 area C). Upon heating the sample to 900 °C, the remaining mass was 22.40%, and no further significant mass loss was observed.

A type IV adsorption isotherm observed for MC-gl, indicated by a hysteresis loop at relative partial pressures between 0.45 and 0.85 (Fig. 3), was similar to the adsorption profile of mesoporous carbon [21,25]. This observation is related to the capillary condensation of the adsorbate against the adsorbent pore wall, leading to desorption that occurs at a lower pressure than adsorption [26]. The total MC-gl surface area and total pore volume were 447 m²/g and 0.626 mL/g, respectively. The MC-gl surface area was greater than that of the fiber type, but it less than that of the monolith type synthesized by Liang and Dai [21]. This result is further substantiated by the examination of the wide distribution of pore diameters, where the pore diameter distribution was 8.86 nm (Fig. 3 inset). Moreover, the MC-gl pore size distribution was similar to that of the monolith type, albeit different from that of the fiber type [21], while SEM images indicated that both types are present in the materials. On the other hand, AC was categorized as microporous material (Fig. 3). AC exhibited a higher

Figure 1. XRD Patterns of (A) as-synthesized Mesoporous Carbon (MC-gl) and (B) Activated Carbon

Figure 2. TGA Profile of as-synthesized Mesoporous Carbon, MC-gl

Figure 3. N₂ Physisorption Profile of as-synthesized Mesoporous Carbon (MC-gl) and Activated Carbon (AC) in the Inset: BJH Desorption Curves and Pore Size Distribution

surface area ($519 \text{ m}^2/\text{g}$). However, the isotherm profile confirmed that the structure is microporous with a pore diameter distribution of $1.907 \text{ cm}^3/\text{g}$. Table 1 summarizes the physicochemical analysis of the two carbon materials.

Fig. 4 shows the FE-SEM images of the as-synthesized mesoporous carbon (MC-gl) and AC morphological structures. The image analysis under $10,000\times$ magnification revealed that MC-gl comprises monolith and grass-like structures covering the carbon surface, affording a mesoporous configuration. This result suggested that the observed grass-like structure may increase the surface area of this unique mesoporous carbon compared to the monolith-mesoporous carbon reported by Liang and Dai [16].

Acid Red 119 (AR119) dye adsorption. Acid Red 119 (AR119) dye was selected as the adsorption model for this study. AR119 dye (IUPAC name: disodium 3-[[N-ethyl-4-[4-(3-sulfonatophenyl) diazenyl]naphthalen-1-yl]diazenylanilino]methyl]benzenesulfonate) is classified as an acid and an azo dye (Fig. 5). Generally, Acid Red 119 is used for fabric dyes and printing on materials

such as silk, wool, and polyamide, as well as leather coloring.

Contact time determination. Initially, the time required to obtain the optimum adsorption capacity, calculated as the removal of AR119 dye over MC-gl, was determined. Fig. 6 shows the experimental results obtained for the variation in the MC-gl contact time (20, 40, 60, and 180 min) against 300 ppm of AR119. Experiments revealed that the dye removal percentage increases with time and plateaus after 60 min, which is related to the oversaturation of the AR119 molecule within mesoporous carbon.

Next, the optimum contact time for the adsorption of AR119 was utilized to calculate the adsorption capacity per adsorbent unit ($\text{mg adsorbate/g adsorbent}$, Q_e). Furthermore, Fig. 6 plots the AR119 adsorption capacity of MC-gl as a function of the contact time. Over a contact time of 180 min, the maximum Q_e value of 212.73 mg/g was observed at 60 min. In subsequent experiments, contact times of 60 and 120 min were utilized.

Figure 4. FE-SEM Images of as-synthesized Mesoporous Carbon: MC-gl (A) and Activated Carbon (AC) (B) Under $10,000\times$ Magnification

Figure 5. Molecular Structure of AR119 dye: (A) Neutral, (B) Acidic, and (C) Basic Conditions. Redrawn Using ChemDraw

Figure 6. Optimization of the contact time (10 mg of MC-gl and 10 mL of a 300 ppm AR119 solution)

Initial pH effect. Subsequently, the variation of pH of the AR119 solution was investigated to observe the optimal pH that induces the adsorption of AR119. Results revealed that the maximum dye removal is observed at a pH of 3 (Fig. 7). Under acidic conditions, AR119 initially dissociated in water, affording both a negatively charged azo compound and Na^+ . Furthermore, the sulfonate group of negatively charged AR119 was protonated, affording a neutral molecule ($-\text{SO}_3\text{H}$, Fig. 5B), whereas at $\text{pH} > 7$, AR119 molecules exhibited negative charges ($-\text{SO}_3^-$) and were paired with Na^+ (Fig. 5C). The formation of neutral molecules under acidic conditions led to the elimination or reduction of repulsive forces between AR119 molecules. Furthermore, the formation of neutral molecules permitted adhesive interactions with the mesoporous carbon surface via van der Waals forces. Therefore, their adsorption on mesoporous carbon is maximized. Similar trends have been reported for the adsorption of solophenyl red 3BL on mesoporous carbon [27] with or without amine functionalization. In addition, AR119 molecules exhibited lower polarity at a $\text{pH} < 7$, indicating that mesoporous carbon undergoes preferential adsorption on nonpolar molecules. this grass-like mesoporous carbon, exhibited a high adsorption activity at pH between 4 and 5, in which $>90\%$ removal of AR119 was observed. With the increase in the solution pH, the amount of dye removal decreased due to the increased amount of anionic AR119 species in the solution. This result indicated that it is possible to apply the as-prepared mesoporous carbon for the removal of industrial dye waste, which is typically acidic in nature, as acid treatment prior to adsorption is not necessary.

Initial concentration effects. With the variation in the initial AR119 concentration (100–800 ppm), the maximum dye removal was observed at an initial dye concentration of 200 ppm (Fig. 8) before exhibiting a decreasing trend at higher initial concentrations.

Goscianska et al. [28] have reported that at low concentrations, dye adsorption is random, whereas at higher concentrations, the sulfonate functional groups of two or more dye molecules interact via van der Waals forces, leading to the formation of a densely packed dye on the adsorbent surface. Hence, at a high initial concentrations, as AR119 molecules are saturated inside MC-gl, further adsorption does not occur.

AR119 adsorption isotherm. The adsorption isotherm models in the current experiment were determined through linear regressions of the Langmuir and Freundlich equations [29]. Fig. 9A shows the Langmuir isotherm for the adsorption of AR119 over mesoporous carbon. The equation $y = 0.1231x + 0.0057$ with a linearity coefficient (R^2) of 0.8166 was obtained. The X_m value and the Langmuir constant (b) were 8.1235 mmol/g and 21.5970 L/mmol, respectively. Based on the R^2 value, the Langmuir isotherm model was not suitable for depicting the adsorption of AR119 into the MC-gl pores, signifying that chemisorption is absent and that there is an extremely weak chemical interaction between the adsorbate and adsorbent.

Figure 7. Effect of pH on Dye Removal Using Mesoporous Carbon (10 mg of MC-gl, 10 mL of 300 ppm AR119, and a Contact Time of 120 min).

Figure 8. Optimization of the Initial Concentration for Dye Removal (10 mg of Mg-gl, 10 mL of AR119, pH = 3, and a Contact Time of 120 min)

Figure 9. (A) Langmuir Isotherm and (B) Freundlich Isotherm for the Adsorption of AR199 on Mesoporous Carbon (MC-gl) (pH = 3, Contact Time of 60 min, and an AR119 Concentration Range from 200 to 1000 ppm)

For the Freundlich adsorption isotherm, the equation was $y = 0.9972x + 0.9954$, with an R^2 value of 0.9998 (Fig. 9B). The degree of adsorbent heterogeneity ($1/n$) and the Freundlich constant (K_F) were 0.9972 and 9.8946 L/g, respectively. Based on the R^2 value, the Freundlich isotherm was thought to be a more suitable model for depicting the adsorption of AR119 into the MC-gl pores. Based on these results, mesoporous carbon comprises multiple layers with the presence of physisorption processes, in which the interaction between mesoporous carbon and AR119 mainly occurs via physical van der Waals forces.

Mesoporous carbon and commercial activated carbon adsorption capacities. Finally, the adsorption of AR119 on the as-synthesized mesoporous carbon (MC-gl) was compared with that of commercially available AC. An adsorption capacity of 8.57 g for AR119 per 1 g of mesoporous carbon was observed, with a dye removal percentage of 85.67%. For AC, the adsorption capacity and dye removal percentage were 5.587 g/g and 55.31%, respectively (Fig. 10), revealing that the adsorption capacity of mesoporous carbon MC-gl is 1.53 times greater than that of commercial AC. The pore size distribution of porous materials plays a key role in the adsorption of large molecules, such as the AR119 molecule. Although the total surface area of as-synthesized MC-gl (447 m²/g) is less than that of AC (519 m²/g), it is characterized by mesoporosity (8.863 nm), whereas AC was a microporous material (i.e., pore size of 1.907 nm). Hence, mesoporous MC-gl is thought to accommodate an increased number of AR119 molecules via van der Waals interactions and exhibit layered adsorption compared to microporous AC.

Figure 10. Adsorption of AR119 on Mesoporous Carbon Compared to Commercial Activated Carbon

Conclusion

Mesoporous carbon was synthesized by the soft-template method using phloroglucinol and Pluronic F127, exhibiting a new grass-like structure morphology. The properties of the as-synthesized mesoporous carbon indicated a transition from monolith to fiber structures. The use of mesoporous carbon as an adsorbent for AR119 dye exhibited optimal dye removal under acidic to mild conditions. Moreover, the adsorption followed the Freundlich adsorption isotherm model. The capacity of the as-synthesized mesoporous carbon was 1.53 times greater than that of commercial AC for the adsorption of the azo dye due to the competition between sulfonate functional groups in AR119 and functional groups on the AC surface, demonstrating its potential for applications in the treatment of synthetic coloring agent waste.

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