Research article

The Adsorption of Perfluorooctanoic Acid on Coconut Shell Activated Carbons

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Abstract: Perfluorooctanoic acid (PFOA) is one of Per- and polyfluoroalkyl substances (PFAS) that have increasingly attracted concerns due to their global distribution in environment, persistence, high bioaccumulation and toxicity. It is important to study the effective treatment to remove PFOA from contaminated water. The feasibility of using commercial coconut shell activated carbon produced in Thailand to remove PFOA from water was investigated with regard to their adsorption kinetics and isotherms of powder activated carbon (PAC-325) and granular activated carbon (GAC-20x50). Adsorption kinetic results show that the adsorbent size significantly affected the adsorption rate of PFOA, and GAC-20x50 required at least 100 h to achieve the equilibrium, much longer than 3 h for PAC-325. Two kinetic models were fitted to the experimental data, and the pseudo-second-order model well described the adsorption of PFOA on both PAC-325 and GAC-20x50. PAC-325 trended to adsorb PFOA faster than GAC-20x50 and testing with the shortest adsorption times (5 min) still yielded substantial PFOA removal (~80% for PAC-325). The adsorption isotherms show that the adsorption capacity of PAC-325 was 0.80 mmol/g, which is 83 % higher than that for GAC-20x50 (0.13 mmol/g), according to the Langmuir fitting.

Keywords: Perfluorooctanoic acid (PFOA); coconut shell activated carbons; adsorption; water treatment

1.   Introduction

The global production and various chemical usage have been distributed in various areas to meet the needs of economic growth. This leads to environmental problems, especially contaminated water with emerging pollutants problems. Per- and polyfluoroalkyl substances (PFAS) are a group of chemicals that are emerging pollutants and have been in widely in industry [1] Some PFAS are very resistant in general environments. They are prone to accumulate in blood and serum in human beings and animals. This accumulation can damage liver, kidney and affect thyroid hormones. Prolonged exposure can cause cancer [2]. Perfluorooctane Sulfonic acid (PFOS) and Perfluorooctanoic acid (PFOA) are chemicals in the PFAS group that have been used widely in industrial plants and products such as carpets, clothing, fabrics for furniture, paper packaging for food and other materials that are resistant to water, grease or stains, fire-fighting foam and plating industry [1]. Both substances have been listed under the Stockholm Convention because they are classified as persistent organic pollutants. Additionally, PFOA is classified as a Category 2B, possibly carcinogenic to humans [2]. The minimum risk level by oral exposure reported by ATSDR for PFOA and PFOS are 3×10^{-6} and 2×10^{-6} mg/kg/day. These values show that even a small amount of substances uptake into the body over a period of time can cause effect to the body [3]. PFAS have been found in drinking water, tap water, surface water, underground water and industrial effluents in many countries including Thailand [1,4–6]. This leads to various health and environmental issues especially the problems with drinking water and contaminated with emerging pollutants. Due to problems to health and environment, many countries have issued regulations on the quantity of PFAS contaminants in various water sources. However, Thailand has not yet issued the relevant regulations. In 2009, PFOA and PFOS were detected in effluent samples from two industrial estates wastewater treatment systems located in the central and eastern regions of Thailand [7]. Their concentrations were in the range of 20–150 and 190–550 ng/L, respectively, which are higher than the USEPA drinking water standards defined at 70 ng/L. Therefore, to reduce the health risk from the exposure to PFOA and PFOS, the development of water treatment to reduce these contaminants is necessary.

S. Camalin et.al [8] reported that the primary and secondary stage of general wastewater treatment systems in industry and community were unable to remove PFAS substances. Thus, it is necessary to use an additional third-stage treatment system to remove these substances. USEPA proposed four methods for PFAS removal from water including activated carbon treatment, ion exchange resins, high pressure membranes and reverse osmosis [9–11]. However, activated carbon is a relatively inexpensive system compared to other water treatment systems. Thus, it is an attractive technology to remove PFOA or PFOS. In general, the surface of the activated carbon is non-polar and has a few other functional groups. This makes it suitable for the separation of hydrophobic pollutants. The PFOA and PFOS molecules are made up of two parts, the non-charged part and a negative charge part. The non-charged part has hydrophobic properties, which can be removed from water by sticking to the surface of activated carbon by physical adsorption (Van der Waals force).

Coconut is one of the important economic crops in Thailand which tends to expand more plantation areas due to higher industrial demand. This causes many agricultural wastes, especially the coconut shell. Nowadays, the locally available coconut shells are promoted to produce commercially activated carbon since it is the utilization and valorization of agricultural waste to create a zero-waste society. According to literatures, the surface area of CSAC was in the range of 1200–1800 m^2/g [12] and both granular activated carbon (GAC) and powder activated carbon (PAC) type could be used to

adsorb PFOA or PFOS pollutants in wastewater treatment [10,11,13,14]. Most of CSAC used in these researches came from international suppliers such as Diasorb W10-30, Calgon Carbon Japan and AquaCarb CX 1230® [15], Evoqua Water Technologies, USA [16]. There was no study in adsorption isotherms and kinetics using CSAC domestically produced in Thailand. Investigation in wastewater treatment plants (WWTP) in Thailand also indicated ineffective removal of PFOA by conventional process [17]. Therefore, study on adsorption characteristics of local CSAC to remove PFOA is necessary to understand the potential removal capacity in order to apply them in Thailand's WWTP. Therefore, this study was to use CSAC (both PAC and GAC type) produced in Thailand to absorb PFOA in spiked water samples to obtain the adsorption rate and capacity data that are useful for water treatment plant design.

2.   Material and methods

2.1.   Materials and chemicals

Perfluorooctanoic acid (PFOA, Cas No.335-67-1) was purchased from Sigma-Aldrich, and their properties were summarized in Table 1. HPLC-grade methanol was purchased from EOS Scientific (Thailand). Other chemicals were of reagent grade. The coconut shell based activated carbons (CSAC) were obtained from Right solution PCL. (Thailand), and their properties from the product specification document were summarized in Table 2. There were two types of activated carbon used in this study. The granular activated carbon (GAC-20x50) with the particle size of 0.3–0.85 mm (mesh 20x50) was selected due to its common size for water treatment and another is the powder activated carbon (PAC-325) with the particle size below 0.045 mm. The Iodine number of GAC-20x50 and PAC-325 were equal to 1100 and 700 mg/g, respectively.

Table 1. Properties of PFOA [18].

Compound	Mol. Formula	Mol. Weight (g/mol)	Water solubility (mg/L)	Log K_{ow}
PFOA	$C_8HF_{15}O_2$	414	2290 at 24 ℃	4.81

Table 2. Properties of CSAC used in this study.

Sample name	GAC-20x50	PAC-325
Type	Granular	Powder
Particle mesh size[1] (%)	+20 mesh: 5%, −50 mesh: 5%	−325 mesh: 60%
Iodine number[1] (mg g^{-1})	1100	700
Moisture content[2] (%)	5	10
Ash content[2] (%)	5	8
Hardness[1] (%)	93	
pH	9–11	9–11
Apparent density (g/cm^3)	0.40–0.60	

Notes: [1]: minimum, [2]: maximum.

2.2. Adsorbent pretreatment and characterization

Prior to perform the adsorption experiment, the CSAC was washed in deionized water several time to remove dirt until pH was constant. Then they were dried at 105 ℃ for 24 hr. The specific surface areas of activated carbons were determined by nitrogen adsorption using a surface area analyzer (ASAP 2460, Micromeritics, USA). The particle size analysis was measured in air (dispersion medium) using a particle size analyzer (Mastersizer 2000, Malvern Instruments Ltd). The true density of CSAC was measured by Ultrapyc 1200e automatic gas pycnometer (Quantachrome corporation). The characterization results were summarized in Table 4

2.3. Adsorption experiments

Batch adsorption experiments were carried out at 165 rpm in the horizontal shaker with 0.005–0.4 g of CSAC (for determination of isotherm adsorption according to ASTM 3860-98 [19]) and 0.06 g of CSAC (for determination of adsorption kinetics) in the 150 ml PP bottle containing 100 ml PFOA solution. The adsorption isotherm experiments were conducted at the initial PFOA concentrations of 20 ppm for 96 h while the adsorption kinetic experiment was performed at the concentrations of 100 ppm for 5 min to 96 h.

2.4. PFOA determination

After the adsorption experiments, the mixture was filtrated by a 3 ml plastic syringe filter with a 0.22 μm, 13 mm nylon membrane. PFOA was analyzed by Liquid chromatograph-tandem mass spectrometer (LC-MS/MS), Shimadzu model LCMS 8045. The system was equipped with inertsil ODC-C18 column (5 μm, 4.6x150 mm) from GL Sciences. The mixture of methanol and 10 mM ammonium acetate in water were used as the mobile phase at 0.7 mL min^{-1} flow rate, 70/30 v/v at starting point, changed to 90/10 at 8 min, changed to 100/0 at 10 min and 70/30 at 16 min. The appropriate HPLC gradient program for the mobile phase was determined experimentally during method development. The column temperature was set at 45 ℃ and the injection volume of sample was 10 μl. The samples were ionized by Electrospray ionization (ESI) and analyzed by multiple reaction monitoring (MRM). The selected ions for MS/MS transition were listed in Table 3. The instrument detection limit for PFOA were about 22 ng/L.

Table 3. Retention times and MRM ions.

Compound	Retention times min	Precursor ion m/z	Product ion	Qualifier ion
PFOA	7.135	413	369	169

The adsorption capacity was calculated according to the difference of PFOA concentrations before and after adsorption as shown in equation below:

$$Adsorption\ (\%) = \frac{C_0 - C_t}{C_0}\ x\ 100 \tag{1}$$

$$q_e = \frac{(C_0 - C_t)x\,V}{w} \tag{2}$$

C_0 = Initial concentration of PFOA (mg/L)
C_t = Equilibrium concentration of PFOA at time t (mg/L)
q_e = Amount of PFOA adsorbed on the adsorbents at equilibrium and time t (mg/g)
V = volume of mixture in adsorption experiment (ml)
w = weight of CSAC (g)

2.5. *Kinetic and Isotherm calculation*

Adsorption isotherms are mathematical models used to explain the distribution of the adsorbate species between liquid and adsorbent. The models based on various assumptions, related to the heterogeneity/homogeneity of the adsorbent or interaction between the adsorbate species. Two models were used to fit the experimental data to determine the adsorption capacity [11].

Langmuir model: $\qquad q_e = \frac{q_m b C_e}{1 + b C_e}$ $\qquad\qquad$ (3)

Freundlich model: $\qquad q_e = k_f C_e^{1/n}$ $\qquad\qquad$ (4)

q_e = the amount of adsorbate adsorbed on the adsorbent at equilibrium (mmol/g)
q_m = the maximum uptake per mass adsorbent (mmol/g)
C_e = the equilibrium concentration of the adsorbate (mg/L)
b = Langmuir isotherm constant (L/mmol).
k_f = Freundlich constant or capacity factor
n = the adsorption intensity

Two commonly used kinetic models, pseudo-first and pseudo-second order kinetic models were applied to calculate the adsorption kinetics [20].

Pseudo-First order: $\qquad q_t = q_e e^{-k_1 t}$ $\qquad\qquad$ (5)

Pseudo-Second-order: $\qquad q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t}$ $\qquad\qquad$ (6)

q_t = adsorption capacity, the adsorbate adsorbed on the adsorbent at time (t) (mmol/g)
k_1 = the first-order rate constant (1/hr)
k_2 = the second-order rate constant $(mmol/mg)(mmol/L)^{-n}$
k_1 and k_2 are the respective adsorption rate constants.

3. **Result and discussion**

3.1. *Physical properties of CSAC*

Porosity distribution of CSAC was shown in Figure 1 and Table 4. Average pore size of PAC-325 and GAC-20x50 were approximately the same as of 1.52 and 1.62 nm, respectively. This range of pore size could be classified as micropores according to the International Union of Pure and Applied

Chemistry, the pore size of activated carbon is classified into three groups which are micropores (size < 2 nm), mesopores (2–50 nm) and macropores (size > 50 nm)[12]. The total volume in pore of PAC-325 was about 36% lower than that of GAC-20x50, which corresponds to the BET surface area of PAC-325 (825 m^2/g) was less than GAC-20x50 (1191 m^2/g). Particle size distribution of CSAC was shown in Figure 2. The ranges of particle size were between 6–119 µm for PAC-325 and 481–1428 µm for GAC-20x50 and their median (D_{50}) size were 45 µm and 836 µm, respectively. The true density of both CSAC was similar as of 1.97 and 2.1 g/cm^3, indicating that it could submerge in water during treatment.
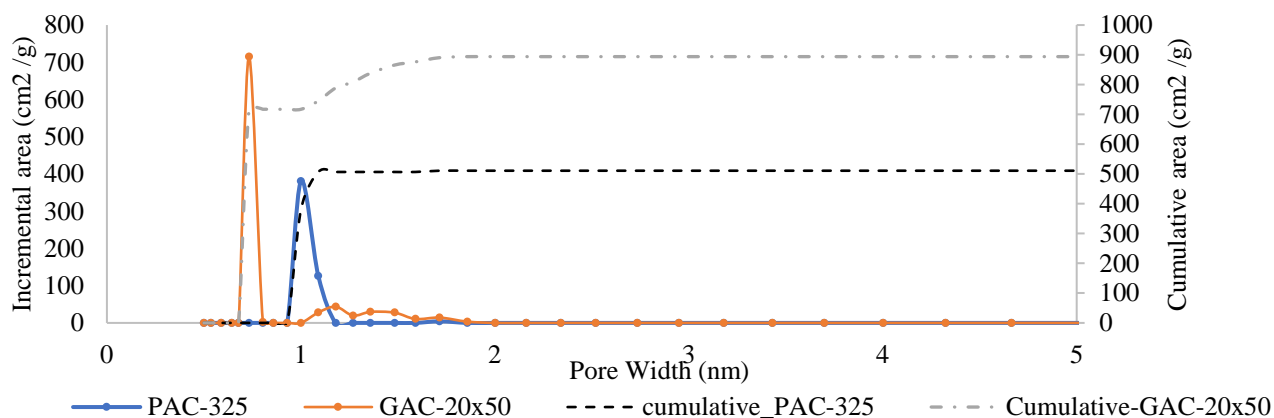

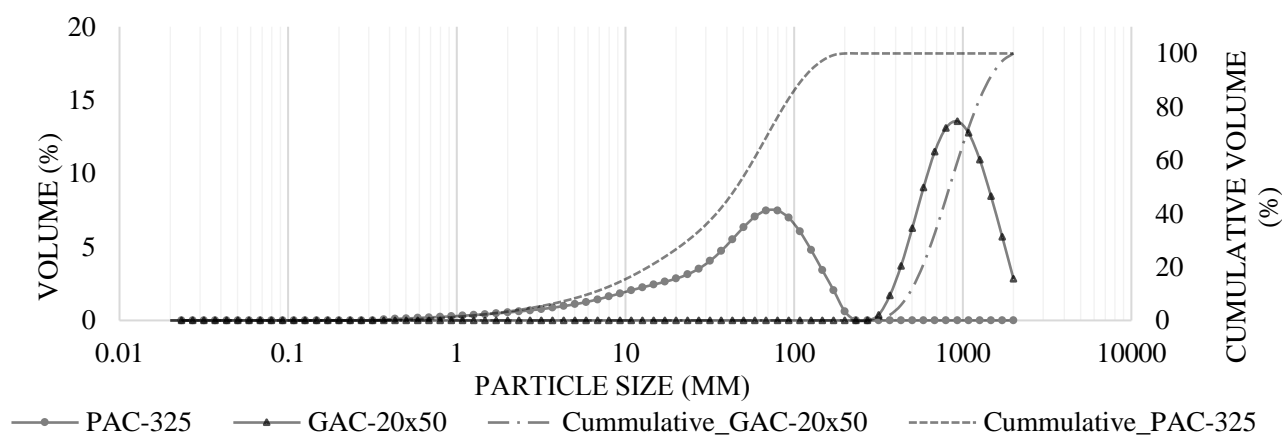**Figure 1.** Porosity distribution of commercial coconut shell activated carbon.

Figure 2. Particle size distribution of commercial coconut shell based activated carbon.

3.2. Adsorption kinetic

Figure3 shows the adsorption kinetics of PFOA on the two adsorbents including the PAC-325 and GAC-20x50. It was found that their kinetic profiles were quite different. PFOA displayed the slower adsorption kinetics on the GAC-20x50 than PAC-325, implying that the sizes of activated carbon influence the adsorption velocity significantly. The adsorbed PFOA onto both CSAC increased rapidly at the early stage (before 5 min) of the adsorption, while the slower adsorption rate was obtained later due to the decrease of active adsorption sites. The adsorption equilibrium of PFOA on

GAC-20x50 was achieved after at least 100 h whereas that on PAC-325 was achieved after 3 h. This result was consistent with research of Q.Yu et.al. [11], about 168 h and 4 h were required to reach the sorption equilibrium for the GAC and PAC. Since PFOA molecules are about 1 nm in length and large proportion of pore size of CSAC used in this study was micropores (size < 2 nm), it took long time for PFOA to diffuse into the intraparticle pores. However, due to the smaller particle of PAC-325 has larger external surface area and more available functional groups for PFOA adsorption, resulting in the faster adsorption than that of GAC-20x50 [11]. Adsorption kinetics of AC depends on the rate of external mass transfer (bulk diffusion and film diffusion) and intra-particle transport (macro pore to micro pore). The overall rate of adsorption on AC is controlled by the slowest mass transfer process [21]. When the particle size of CSAC decreases, the external surface area becomes larger, enhancing the film mass transfer rate. Thus, PAC-325 exhibited faster uptakes for PFOA than GAC-20x50. Moreover, in literature the pore distribution of the pulverized AC (PAC-325) was mainly formed mesopore and macropore rather than micropore which help the movement of adsorbate into the carbon pore matrix[22].

The pseudo first-order and pseudo second-order model were selected to fit the kinetic data to predict the rate of adsorption. The pseudo first-order model is mainly diffusion processes (physical adsorption) and it is suitable if the concentration of a reactant is assumed constant due to the excessive amount with respect to the other reactants. The pseudo second-order model is mainly chemical adsorption and it assumes that the adsorption capacity is proportional to the number of active sites on the adsorbent [23].
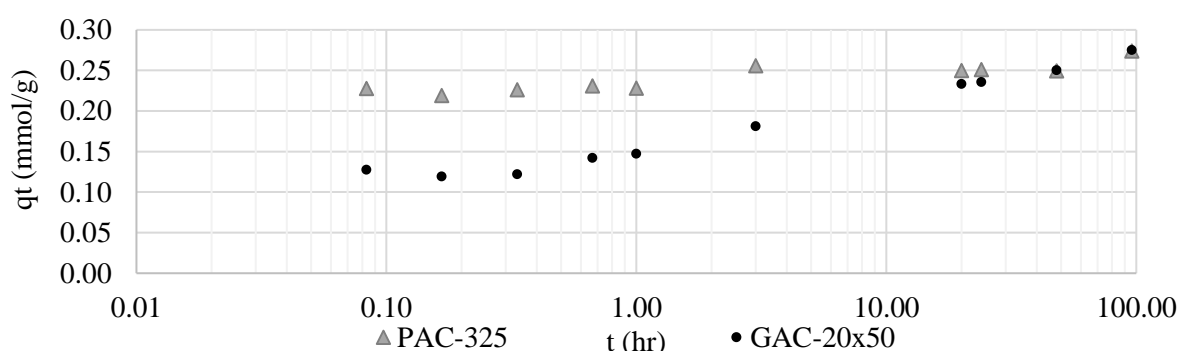

Figure 3. Adsorption kinetics of 100 ppm PFOA on the PAC-325 and GAC-20x50.

As shown in Table 5, the pseudo-second-order model fitted with the adsorption data better than the that of pseudo-first order model according to the relatively higher correlation coefficients ($r^2 > 0.83$), indicating that the chemical interactions were possibly involved in the adsorption processes. However, Q.Yu et.al. [11] reported that the electrostatic and hydrophobic interaction was also possibly involved the adsorption. In addition, it was found from Table 5 that the second-order rate constant (k_2) for the PAC-325 was 300 g/mmol/hr, which was much higher than that for the GAC-20x50 (18.8 g/mmol/ hr), indicating the fast adsorption of PFOA on the PAC. Moreover, it was found that testing with the shortest adsorption times in this study (5 min) still yielded substantial PFOA removal (~80% for PAC-325).

Table 4. Pore and particle characteristics of CSAC used in this study.

Adsorbent	Particle size distribution[1] (μm)	Accumulative pore volume[2] (cm³/g)	Avg. pore size (nm)	Pore system	Bet surface area (m²/g)	Micropore area[3] (m²/g)	External surface[3] (m²/g)	True density (g/cm³)
PAC-325	6–110	0.287	1.52	micropore	825	805	20	1.97
GAC-20x50	481–1428	0.390	1.62	micropore	1191	1175	17	2.1

Notes: [1]: D_{10}-D_{90}, [2]: BJH adsorption (0.1–700 nm), [3]: by t-Plot micropore method.

Table 5. Adsorption kinetics parameters for the adsorption of PFOA on CSAC.

Adsorbent	Initial conc. (mmol/L)	pseudo-first order q_e (mmol/g)	k_1 (1/hr)	r^2	pseudo-second order q_e (mmol/g)	k_2 (g/mmol/hr)	r^2
PAC-325	0.242	0.263	62.2	0.325	0.248	300	0.895
GAC-20x50	0.242	0.224	5.77	0.756	0.232	18.8	0.834

Notes: r^2 = Correlation coefficient.

Table 6. Adsorption isotherm parameters for the adsorption of PFOA on CSAC.

Adsorbent	Langmuir isotherm q_m (mmol/g)	B (L/mmol)	r^2	Freundlich isotherm k_f (mmol/mg) $(mmol/L)^{-n}$	n	1/n	r^2
PAC-325	0.80	1750	0.823	53	1.52	0.66	0.804
GAC-20x50	0.13	2228	0.870	0.77	2.92	0.34	0.942

Notes: r^2 = Correlation coefficient.

3.3.   Adsorption isotherm

Adsorption isotherm is important to evaluate the adsorption capacity of adsorbents. The absorbed concentration increases as the solute concentration increases. The simple model for adsorption is the linear model coefficient, K_d (solid-liquid distribution coefficient) which is the ratio of q_e and C_e as shown in Figure 4. The results show that K_d was not constant or in the other words, the linear model was not suitable to describe the sorption behavior of PFOA at high concentrations [24]. The value of K_d for GAC-20x50 was in the range of 21–19396 L/g with the average value of approximate 4377 L/g, whereas for PAC-325 was in the range of 515–31520 L/g with the average value of approximate 5335 L/g which was greater than that of GAC-20x50. Langmuir model assumes adsorption in a monolayer and a constant adsorbate affinity for all surface sites [20] while Freundlich model is an empirical equation describing the adsorption of adsorbates from liquid phase to adsorbent surface and assumes that different sites with several adsorption energies are concerned. As shown in Figure4 and Table 6, Langmuir adsorption capacity of PAC-325 was 0.80 mmol/g or 331 mg/g, which is 83 % higher than that for GAC-2050 (0.13 mmol/g or 54 mg/g). However, the adsorption isotherms of PFOA on both PAC-325 and GAC-20x50 could fitted by the Langmuir model and Freundlich model with lower correlation coefficients (r^2 = 0.82–0.94) that of Q.Yu et.al. study [11].

Adsorption capacities of commercial CSAC used in this study were compared with previous reported values as shown in VII. It shows that the adsorption capacity of PAC-325 was similar to that of other studies whereas the adsorption capacity of GAC-20x50 was less than the others.
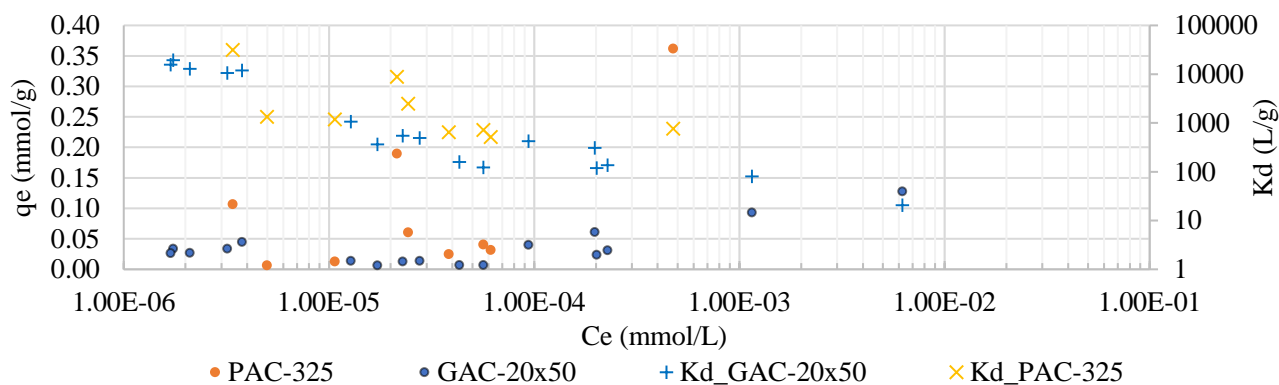
Figure 4. Adsorption isotherms of 20 ppm PFOA on the PAC-325 and GAC-20x50, shaking time 96 h.

3.4.   Application

In practice, the application of activated carbon used for water treatment can be separated in two types of fluid-sorbent contacting, PAC dosing and GAC packed columns [25]. For PAC dosing systems, AC particles are injected into the contaminated water, dispersed within the water, and then removed by sedimentation or filtration. PAC is usually added at the head of the water treatment plant to provide the longest contact time possible before applying other treatment chemicals. The adsorption isotherm and kinetic of commercial PAC-325 in this study could be used for the preliminary design for removal of PFOA in the water via PAC dosing systems. For GAC packed-bed system, contaminated water flows through a static bed of GAC designed for an appropriate contact time. This system is a process used as tertiary treatment of municipal and industrial wastewater (coagulation, sedimentation, filtration, GAC adsorption). Many researchers studied the adsorption capacity of PFAS in natural surface water by using rapid small-scale column tests (GAC packed-bed system) to simulate the fluid flow condition similar to treatment plant. The adsorption capacities which was calculated from breakthrough volume were shown in Table 8. It shows that the adsorption capacities of GAC were in the range of 0.001–0.028 mg of PFAS per g of AC, which is significantly lower than that of GAC-2050 (54 mg of PFOA per g of AC) measured in this study. This may be concerned with active site competition which is influenced by other matrix substances existing in natural surface water, especially the dissolved organic carbon [18] and the lower initial concentration of PFAS (4–1000 ng/L) compared to that used in our study. Additional studies are needed to evaluate the adsorption isotherm and kinetic of CSAC in natural surface water by packed-bed systems to perform preliminary testing to evaluate removal performance.

Table 7. Adsorption capacity of AC in other studies.

Type of activated carbon	Size (mm)	Initial conc. (mg/L)	pH	Adsorption capacity (mmol/g)
PAC[a]	<0.11	20	-	0.80
GAC[a]	0.48–1.43	20	-	0.13
GAC[b] [11]	0.9–1.0	20–250	5	0.39
GAC[c] [26]	0.60–0.85	20–250	5	1.15
GAC[d] [27]	-	15–250	5–7.2	0.27–0.38
PAC[d] [27]	-	20–300	5–7	0.42–1.26
PAC[b] [11]	<0.1	20–250	5	0.67
PAC[d] [28]	<0.149	5–40	6.1	0.038
Grape leaf litter [29]	-	0.125–1	4	0.19

Notes: [a]: this study, [b]: coal, [c]: b amboo, [d]: commercial.

Table 8. Other studies of PFAS adsorption in water contained with dissolved organic matter (modified from [18].)

Adsorbate Target PFAS	Level (ng/L)	Adsorbent Type	EBCT (min)	Organic matter Source	DOM conc. (mg/L)	CUR (kg of AC/m³)[a]	Adsorp. capacity (mg/g)[a]
PFCAs and PFSAs	1000	GAC (F300, F600, 1240C)	0.38	Surface water	1.7	0.043	0.024
PFCAs and PFSAs	100	GAC (F400)	6.1	Drinking water	1.8	0.017	0.006
PFOS, PFOA, PFDA	150–300	GAC (Norit Darco 12x40)	5.2	-DI, Surface water -Wastewater	4–7	0.008 0.013	0.028 0.017
PFOS, PFOA, PFPeA, PFHxA	4–18	GAC (Norit 830)	8	WWTP (tertiary treated effluent)	5	0.014	0.001

Notes: PFCAs = Perfluoroalkyl carboxylic acids, PFSAs = Perfluoroalkane sulfonic acids, PFDA = Perfluorodecanoic acid, PFPeA = Perfluoropentanoic acid, PFHxA = Perfluorohexanoic acid, EBCT = embedded contact time, DOM = dissolved organic matter, CUR = carbon usage rate, [a]: Calculated from breakthrough volume4. Conclusions.

The adsorption kinetics and isotherms of PFOA on the two commercial activated carbons were investigated. The PAC-325 was found to be the better adsorbent for PFOA than GAC-20x50 in terms of adsorption kinetics and adsorption capacity. The adsorption kinetic results reveal that the adsorption of PFOA on GAC-20x50 was very slow, and the adsorption equilibrium was achieved after at least 100 h, while that of PAC-324 was only about 3 h. The pseudo-second-order model can fit with the adsorption kinetic data better than pseudo-first order model, indicating that the chemical interactions were possibly involved in the adsorption processes. From the second-order rate constant (k_2), PAC-325 trended to adsorb PFOA faster than GAC-20x50. The results of adsorption isotherms show that the maximum adsorption capacities were 0.80 and 0.13 mmol/g for PAC-325 and GAC-20x50, respectively, according to the Langmuir model. This research shows the possibility of using commercial CSAC (PAC and GAC) to absorb PFOA in water treatment. However, further adsorption studies with natural surface water need to be conducted to obtain more appropriate adsorption and kinetic parameters to design the size of treatment plant.

Acknowledgment

Conflict of interest

The authors declare no conflict of interest.

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