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Removal of benzo (a) anthracene from water using a novel UAE sludge-based activated adsorbent

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ABSTRACT

A new adsorbent material from sewage sludge of relatively high surface area (2,230 m^2/g) was prepared by carbonization and chemical activation using steam at controlled semi-static conditions. The pore size distributions of the sludge-based activated adsorbent (SW-A2) and commercial activated carbon were compared and found to be remarkably different with noticeable presence of larger micro-pores and small meso-poresfor SW-A2. The newly developed activated adsorbent was then evaluated for the adsorption of a polycyclic aromatic hydrocarbon (PAH); benzo (a) anthracene. The adsorption isotherms of benzo (a) anthracene on the adsorbent materials were fitted to both Langmuir and Freundlich isotherms. The Langmuir isotherm was found to be the best fit for the adsorption of the benzo (a) anthraceneon ACwith K and q_{max} of 0.43L/mg and 70.5 mg/g, respectively. On the other hand, the adsorption isotherm for SW-A2 was found to follow the Freundlich isotherm with K_f and n of 100.8 mg/g $(L/mol)^{-0.763}$ and 1.31, respectively. The adsorption kinetics for both adsorbents were found to follow pseudo second order kinetics with rate constant of 1.55×10^{-4} g mg^{-1} min^{-1} for SW-A2 and 1.44×10^{-4} g mg^{-1} min^{-1} for AC. The results of this study indicate the high potential of the newly developed sludge-based adsorbent as an efficient and successful material for the removal of carcinogenic PAHs from water body.

Keywords: Benzv (a) anthracene; Adsorbent materials; PAHs; Water remediation; Adsorption isotherms

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a large family of compounds characterized by their fused benzene rings. PAHs vary in their physical and chemical characteristics based on the number of aromatic rings and the pattern of their ring linkage. PAHs have unique UV absorbance features and most of them can fluoresce with characteristic fluorescence spectra upon excitation [1]. They are introduced into the environment from natural and anthropogenic sources, as products of the thermal decomposition and reformation of organic molecules [2]. PAHs are considered to be amongst the most widespread contaminants of organic nature in air, soils, water and wastewater [3, 4]. There are various sources of PAHs in the environment including industrial emissions, agricultural activities, and medical wastes. Furthermore, they could be present in dyes, plastics, pesticides, and wood preservatives [5]. Exposure of organisms to PAHs occurs through ingestion, inhalation, and dermal contact and/or a combination of one or more of these routes; hence, impacting the dosage and the uptake [1,6]. PAHs are carcinogenic, teratogenic and mutagenic, and their toxicity increases with molecular weight [7]. Due to their known toxicity, the US Environmental Protection Agency (US EPA) and the International Agency for Research of Cancer(IARC) classified some PAHs including: benz[*a*]anthracene, chrysene, and indeno[1,2,3-*c,d*]pyrene, amongst others, as probable human carcinogens [5,8]. The harmful effects of PAHs to humans are attributed to their ability to bind to human tissues and due to their ability to

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be activated metabolically by the cytochrome p450 enzymes [9,10]. Moreover, sixteen PAH compounds have been listed as priority pollutants by US-EPA [11]. Their presence in water poses risk to aquatic life and human heath due to their persistence and stability; hence, may result in bioaccumulation and biomagnification in aquatic food chains [12].

In recent years, the removal of PAHs has become the center of major drive for scientific research. Several studies have focused on the removal of PAHs from water using poly-cyclodextrin cryogels with aligned porous structure. These studies showed that cryogels can be applied with high efficiency for removing PAH molecules from water bodies as a result of the volume based scavenging mechanism and the interfacial adsorption [13]. Microalgae and dead algal cells have also been applied in wastewater treatment processes to remove toxic organic pollutants, including PAHs [14]. Furthermore, magnetic permanently confined micelle arrays (Mag-PCMAs) with a maghemite core and a silica porous layer, have been synthesized to remove PAHs and other metal contaminants [15]. Similarly, carbon nanofibers coated with magnetite (CNF-Fe_3O_4) was found to be efficient in the removal of some polycyclic aromatic hydrocarbons, including benz(a)anthracene [16].

Another effective technique for the removal of PAHs from wastewater is based on the adsorption using activated carbons. This technique is very attractive due to the persistency of the carbon material. Several studies have presented the rates and mechanisms of the adsorption process and the characteristics of various activated carbon adsorbents, denoting their effectiveness [17–22]. In particular, publications such as those coauthored by Moradi and Zhang, reported the effectiveness of the phenanthrene adsorption and desorption processes on activated carbons of from aqueous solutions using organic solvents [23,24].

In a previous work, activated adsorbents from sewage sludge were prepared using a novel semi-static carbonization ad activation processes [25,26]. The activated adsorbents were applied for the removal of lead ions from aqueous solution. The adsorbents were not evaluated for organic compounds removal. Also, the effect of conducting the activation at higher temperature and longer time were not studied.

The aim of this study is to modify the method of activation and develop a series of adsorbent materials using sewage sludge waste as a precursor and evaluate their efficiencies for the removal of PAHs from water. Benzo (a) anthracene is used as a model compound for PAHs in this study. The choice of benzo (a) anthracene compound was based on its carcinogenicity and its presence in tobacco smoke. The specific objectives include the optimization of the adsorption parameters of benzo (a) anthraceneon the newly developed adsorbents, the measurements of the adsorption isotherms on the most efficient adsorbent, and the study of the adsorption efficiency and kinetics. In addition, a commercially available activated carbon was used for comparison purposes.

2. Experimental

2.1. Benz[a] anthracene preparation

Benzo (a) anthracene neat standard was purchased from Supelco (Bellefonte, PA, USA). A stock solution was prepared by dissolving 10 mg of benzo (a) anthracene in 100 mL of methanol. Diluted solutions were prepared from the stock solution using a 70:30 (v/v) ratio of methanol: water.

2.2. Activated adsorbent preparation

Sewage sludge samples were obtained from a wastewater treatment plant in Dubai, UAE. The design of the carbonization and activation processes were done in the absence of any external flow as described in previous publications [25,26]. The carbonization and activation were done using a home-made stainless steel apparatus constructed in our facilities [26]. In briefly, the washed and un-washed sludge samples were dried inside an oven at 105°C followed by carbonization at 680°C for one hour in nitrogen environment. The carbonized samples were then homogeneously mixed with potassium hydroxide (KOH) fine powder at 1:3 (w/w) followed by activation at 680°C for one hour using internally generated steam at the same temperature [25,26]. The activated samples were washed with 1.0 M HCl then rinsed with distilled water until pH of deionized water was reached. The unwashed and washed samples are denoted as S-A and SW-A, after activation. To improve the structure of the developed adsorbent, further modification was made where the carbonization and activation temperatures were done at 750°C on the washed sludge sample while maintaining all previous conditions constant. The new sample was denoted as SW-A2. For comparison purposes, commercial activated carbon (Calgon, USA) was purchased. The commercially available activated carbon sample was denoted as AC.

2.3. Sorption of Nitrogen

The Brunauer–Emmett–Teller surface area (S_{BET}) for all activated sludge-based adsorbents were determined from the nitrogen adsorption isotherms. In addition the pore size distribution and structural parameters such as micropore volume (V_{mic}), and total pore volume (V_t) were calculated using density functional theory (DFT) [27]. The nitrogen adsorption and desorption isotherms were measured at 77 K using quanta chromegas sorption analyzer (AutosorbiQ, USA). Before the experiment, the activated samples were outgassed at 423 K under a vacuum of 10-5 torr to constant pressure.

2.4. Adsorption studies

2.4.1. Choice of adsorbent material

To select the optimized adsorbent for the adsorption study, 20.0 ml of different concentrations of benzo (a) anthracene ranging between 1.00–10.0 ppm were added to 10.0 mg of a specific adsorbent from the samples listed above. The suspensions were mixed in a thermal shaker (Edmund Buhler KS-15/TH-15 shaker, Germany) at150 rpm and 25°C for 24 h. The pH of the solution was maintained at 7 using either 0.10 M HCl or 0.10 M NaOH solution. The suspensions were centrifuged at 4000 rpm for 5 min using a centrifuge (HERMLE centrifuge, Model 2206A, Germany) and the concentration of benzo (a) anthracene was determined using UV/VIS spectrophotometer at 287 nm (Cary a 50 spectrophotometer, Varian, Australia). The percent removal of benzo (a) anthracene by the newly developed adsorbents and by the commercial activated carbon were determined using Eq. (1).

$$\% \text{ Removal} = (C_o - C_e)/(C_o) * 100 \tag{1}$$

where C_o and C_e are the initial and equilibrium concentrations in ppm, respectively.

2.4.2. Effect of adsorbent dosage and contact time

Once the percent removal of benzo (a) anthracene was measured using all of the adsorbents, the effect of adsorbent dosage on the % removal was studied using two hours contact time. For this purpose, 50.0mL solution of 10 ppm of benzo(a) anthracene with varying dosages of adsorbents (2.50, 5.00 and 10.0 mg which give an adsorbent dosage of 0.0500, 0.100, and 0.200 g/L, respectively) were mixed at 25.0°C.

To study the effect of contact time, batch experiments were conducted by mixing a 50.0 mL solution of 10 ppm of benzo (a) anthracene with a fixed dosage of activated carbon (5.00 mg) samples were taken at different time intervals of 15, 30, 60, 120, 180, 300 and 480 min, centrifuged and the percent removal was determined as described in section 2.3.1.

2.4.3. Adsorption isotherms

After the selection of the best adsorbent, adsorption isotherms of benzo (a) anthracene were obtained at the optimum conditions and a temperature of 25.0°C. The adsorption isotherms were also obtained on the commercial activated carbon for comparison purposes. The experimental isotherms were fitted to both Langmuir [Eq. (2)] and Freundlich [Eq. (3)] models.

$$\frac{C_e}{q_e} = \frac{1}{K'q_m} + \frac{C_e}{q_m} \tag{2}$$

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \tag{3}$$

where q_e, q_m and k_f represent the amount of benzo (a) anthracene being adsorbed at equilibrium per gram of adsorbent (mg/g), its maximum adsorption capacity (mg/g) and its Freundlich capacity parameter ($\text{mg}^{(1-1/n)}\ \text{L}^{1/n}/\text{g}$), respectively; K' and n are adsorption constants for Langmuir and Freundlich isotherms, related to energy of adsorption and adsorption intensity, respectively.

2.4.4. Adsorption kinetics

Adsorption kinetics provide important information on the rates of adsorption and as such furnish the bases of understanding the extent of adsorbate uptake by the adsorbent at different times and help in determining the mechanisms of the adsorption process itself [28–31]. Several kinetics models are reported in the literature to describe the adsorption rate on heterogeneous surfaces [28–31]. These are pseudo first order (Lagergren model), pseudo second-order models, and Langmuir Kinetics. In this work, pseudo first order and pseudo second-order models are used to fit the experimental data of adsorption of benzo (a) anthracene on SW-A2 and AC. These two models give the overall observed rate constant for the adsorption process based on a two proposed mechanisms for the adsorption process [28]. In this work, the linearized forms of the Lagergren model (pseudo first order kinetic model) and pseudo second order kinetic model are used to fit the experimental data. These forms are given by Eqs. (4) and (5), respectively.

$$\ln\left(q_e - q_t\right) = -k_I t + \ln q_e \tag{4}$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{K_{II} q_e^2} \tag{5}$$

where q_t, k_I and k_{II} represent instantaneous amount of benzo (a) anthracene being adsorbed per gram of adsorbent (mg/g), pseudo first order rate constant (1/min) and pseudo second order rate constant (g/mg·min), respectively.

3. Results and discussion

3.1. Nitrogen sorption isotherms

Fig. 1 shows the nitrogen adsorption/desorption isotherms for AC, S-A, SW-A and SW-A2 adsorbent samples. The curves on the first three adsorbents showed minimal hysteresis at high P/P_o which indicates that these adsorbents are mainly microporous [25]. The presence of hysteresis on the SW-A2 curve indicate the presence of additional larger pores within the structure of this adsorbent. Moreover, the higher nitrogen uptake at lower relative pressure ($P/P°$) on SW-A2 compared to the other adsorbents reflects the enhanced microporous structure for this adsorbent as well. The KOH activation at higher temperature seems to enhance the distribution in the pore sizes for the activated adsorbents prepared from sewage sludge. The results observed in Fig. 1 are supported by the pore-size distribution (PSD) curves presented in Fig. 2. Although washing

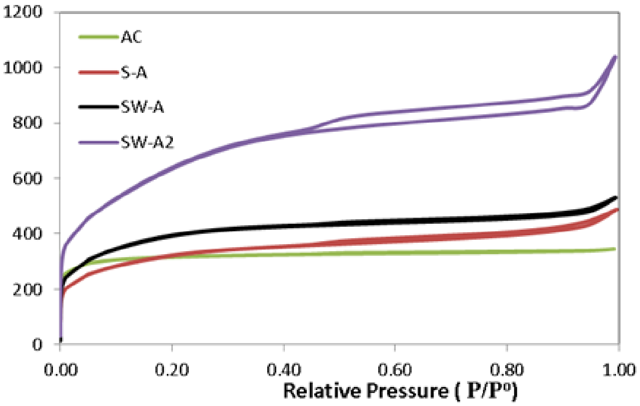

Fig. 1. Adsorption/desorption isotherms of N_2 at 77 K for the commercially available activated carbon (AC) and the sludge-based activated adsorbents (S-A, SW-A and SW-A2).

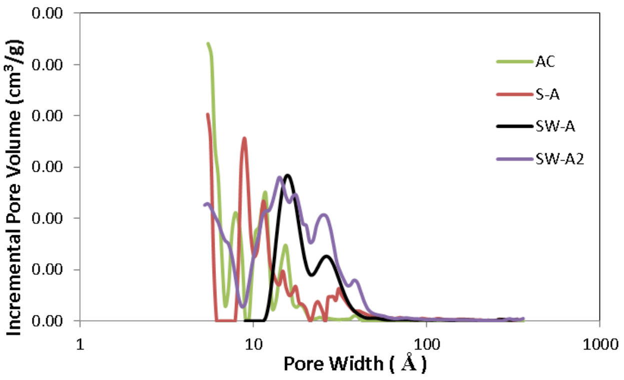

Fig. 2. Pore size distribution of the commercially available activated carbon (AC) and the sludge-based activated adsorbents (S-A, SW-A and SW-A2) using density functional theory (DFT).

Table 1
Comparison of surface structural parameters of the commercial and sludge-based adsorbents*

Sample	S_{BET} (m²/g)	V_{mic} (cm³/g)	V_t (cm³/g)
AC	1236	0.458	0.492
S-A	1183	0.352	0.662
SW-A	1552	0.321	0.792
SW-A2	2189	0.633	1.37

*AC: commercial activated carbon, S-A: unwashed sludge-based adsorbent activated and carbonized at 680°C, SW-A: washed sludge-based adsorbent activated and carbonized at 680°C, SW-A2: washed sludge-based adsorbent activated, carbonized at 750 °C, S_{BET}: BET surface area, V_{mic}: micropore volume, V_t: total pore volume.

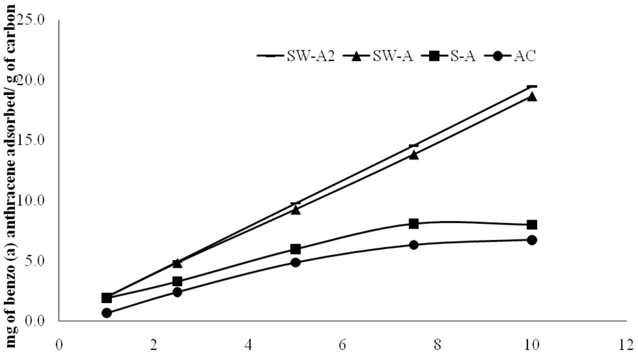

Fig. 3. Adsorption capacity of Benzo (a) anthracene on the sludge-based activated adsorbents (S-A, SW-A and SW-A2) and the commercially available activated carbon (AC) at 25°C, 24 h contact time, shaking speed of 150 rpm, and adsorption dosage of 0.500 g/L.

the sludge prior to carbonization introduced larger micropores into the structure; it is evident from SW-A2 curve that activation of the washed sludge at higher temperature enhanced the overall distribution of pores, mainly in the micropore and small mesoporous region. The presence of such wide range of micropores indicates the effectiveness of this adsorbent for the removal of molecules such as volatile and semi-volatile organic matter. The commercial activated carbon seems to have small micropores only; mainly those smaller than 10 . Such results are expected since the activated carbon is designed by the supplier to remove volatile organic compounds from air stream [32].

The BET surface area (S_{BET}) and the structural parameters of the commercial and produced adsorbents were calculated for all samples and are summarized in Table 1. The S_{BET} for the commercial activated carbon is 1236 m²/g while it was found to be 1183 m²/g and 1552 m²/g for the S-A and SW-A samples, respectively. The activation of the washed sludge at higher temperature resulted in increasing the surface area to 2189 m²/g (SW-A2) which reflects the observed changes in the pore size distribution as described earlier. The surface area obtained in this study was higher than that reported for many adsorbents prepared from sewage sludge using different methods [33–43]. The total pore volume (0.492, 0.662, 0.792, 1.37 cm³/g) and micropore volumes (0.458, 0.352, 0.321 and 0.633 cm³/g) were also calculated for the AC, S-A, SW-A and SW-A2, respectively. The higher micropore and total pore volumes for the SW-A2 sample is evident and it is in agreement with the above results and discussions.

3.2. Adsorption study

3.2.1. Selection of adsorbent

In spite of the fact that SW-A2 showed the highest surface area with higher volume and broader distribution of pores, the adsorbent capacity of the three sludge-based adsorbents were evaluated for benzo (a) anthracene removal. Fig. 3 clearly shows that the adsorption capacity of the SW-A and SW-A2 adsorbents was higher than the commercial (AC) and the non-washed sludge based adsorbent (S-A). The difference in the uptake becomes evident at higher initial concentrations, especially for SW-A2. The results indicate that SW-A2 is the best adsorbent for benzo (a) anthracene; hence it was used to further optimize the adsorption isotherm parameters and for the kinetics studies.

3.2.2. Effect of dosage and contact time

Adsorbent dosage plays a major role in the adsorption process. As shown in Fig. 4, an increase in the adsorbent dose can improve adsorption of benzo (a) anthracene. Furthermore, an adsorbent dosage of 0.1 g/L–0.2 g/L seems to be the most ideal dosage for the adsorption of the benzo (a) anthracene. When comparing the SW-A2 adsorbent to the commercially available adsorbent (AC), SW-A2, showed a percent removal of 82% and 99% compared to 56% and 88% on AC, for an adsorbent dosage of 0.1 g/L and 0.2 g/L, respectively. Two studies have been recently conducted on the efficiency of removal of selected PAHs by activated carbon in water and oil matrixes, both studies reported that at least 90% of compounds such as phenanthrene were successfully removed from both media [23, 44].

Fig. 5 shows that 65% of the PAH was removed on SW-A2 adsorbent compared to 52% on the commercial car-

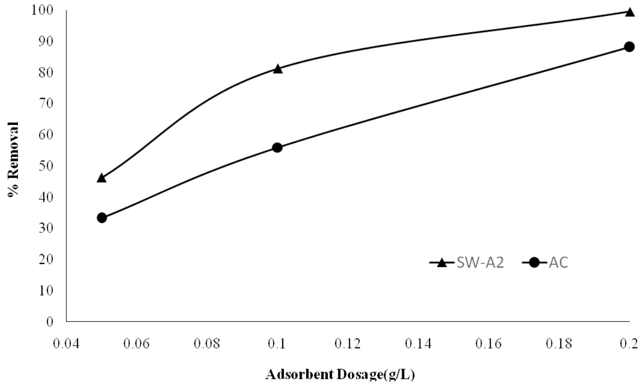

Fig. 4. Effect of adsorbent dosage on the adsorption capacity of Benzo(a)anthracene on SW-A2 and AC at 25°C, 2 h contact time, shaking speed of 150 rpm, and adsorbents dosages of 0.050 g/L, 0.10 g/L and 0.20 g/L.

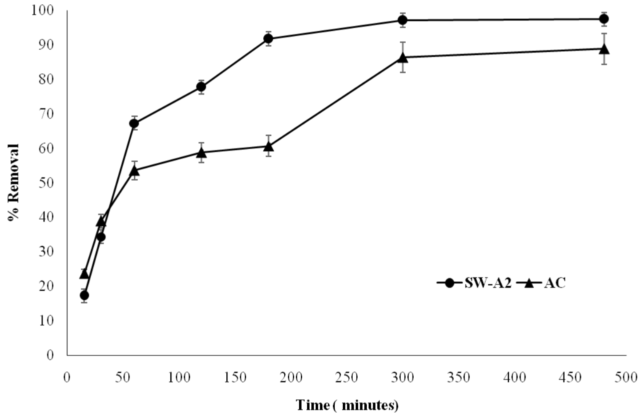

Fig. 5. Effect of contact time on adsorption capacity of Benzo (a) anthracene on SW-A2 and AC at 25°C, initial concentration = 10 ppm, shaking speed of 150 rpm and adsorbent dosage 0.1 g/L.

bon (AC) after one hour. At a contact time of 300 min both adsorbents reached their maximum equilibrium efficiencies under these conditions, with SW-A2 achieving a higher percent removal of over 90% compared to 80% for the AC. The results of this study are comparable to other studies using immature coal (leonardite) where 70% removal of PAH was reported after two hours [45]. Furthermore, similar studies in the literature on the removal of phenanthrene by activated carbon [23] revealed similar efficiencies to our work, however, the time needed to reach equilibrium was 11 h compared to 5 h in our study. These results render the newly developed adsorbent to be efficient with faster kinetics for the removal of PAH as modeled by benzo (a) anthracene, moreover, it has a potentially better removal efficiency even at very low adsorbent dosage.

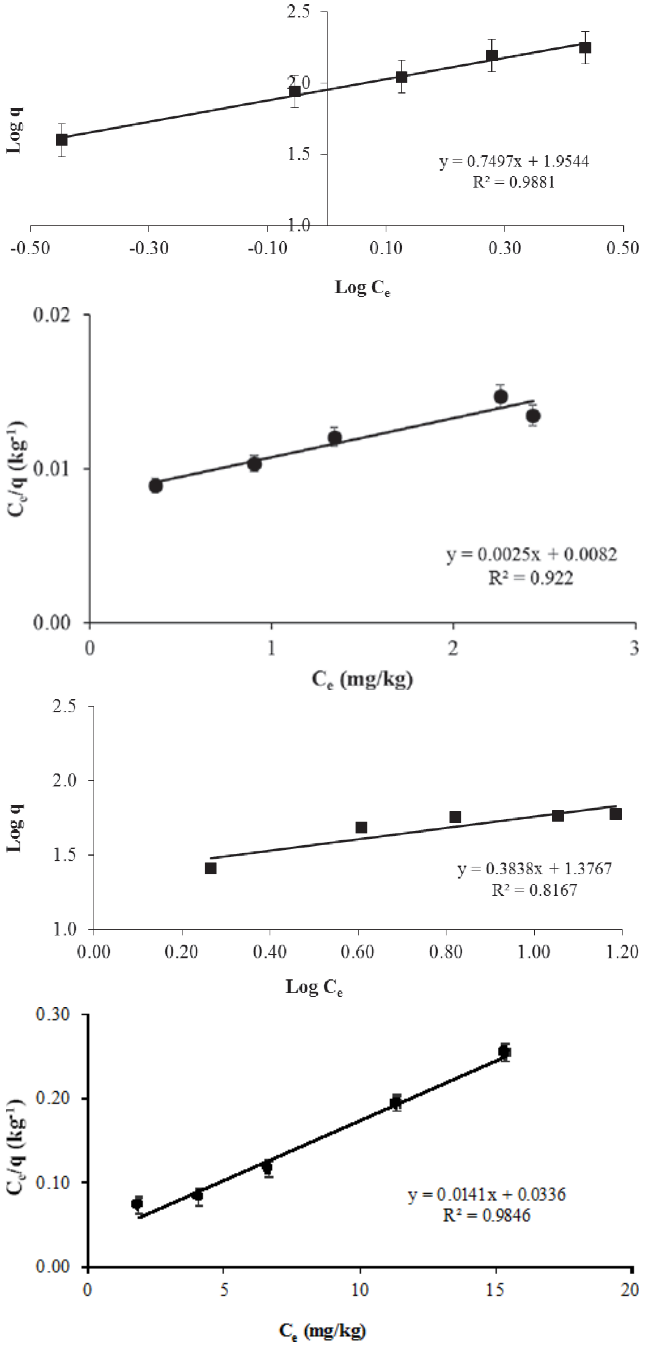

Fig. 6. Adsorption models of the adsorption of benzo(a) anthracene on the sewage sludge-based activated adsorbent (SW-A2) and on the commercially available activated carbon (AC): (a) and (b) Freundlich and Langmuir models for SW-A2, (c) and (d) Freundlich and Langmuir for AC at 25°C, 3 h contact time, shaking speed of 150 rpm, and adsorbents dosage of 0.10 g/L.

3.2.3. Adsorption isotherms

The experimental adsorption isotherm data for benzo (a) anthracene on SW-A2 and AC were fitted to both Langmuir and Freundlich isotherms models as shown in Figs. 6a–d. The fitting parameters and adsorption isotherms constants for both adsorbents are summarized in Table 2. Inspection of these figures reveals that the data for AC fits Langmuir isotherm better than Freundlich isotherm as evident from the R^2 values (Table 2). Where the data for SW-A2 fits Freundlich isotherm better than Langmuir isotherm

Table 2
Isotherms constants and fitting parameters for Langmuir and Freundlich models for the adsorption of benzo (a) anthracene on SW-A2 and AC at 25°C, 3 h contact time, shaking speed of 150 rpm, and adsorbents dosage of 0.1 g/L

Adsorbent	Isotherm model	Equation	Linear equation	Parameters	Values	Removal efficiency
SW-A2	Freundlich	$q_e = K_f C_e^{1/n}$	$\log(q_e) = \log K_f + 1/n \log C_e$	K_f(mg/g (L/mol)$^{-1.31}$)	$1.01 \times 10^2 \pm 8.9$	89.0%
				n	1.31 ± 0.03	
				R^2	0.990 ± 0.01	
	Langmuir	$q_e = (q_m b\ C_e)/(1 + bC_e)$	$C_e/q_e = (C_e/q_m) + (1/q_m b)$	K' (L/mg)	0.300 ± 0.02	
				q_m (mg/g)	$4.11 \times 10^2 \pm 65.7$	
				R^2	0.920 ± 0.01	
AC	Freundlich	$q_e = K_f C_e^{1/n}$	$\log(q_e) = \log K_f + 1/\mathrm{n} \log C_e$	K_f(mg/g (L/mol)$^{-1.31}$)	33.8 ± 1.2	30.5%
				n	2.60 ± 0.2	
				R^2	0.820 ± 0.01	
	Langmuir	$q_e = (q_m b\ C_e)/(1 + bC_e)$	$C_e/q_e = (C_e/q_m) + (1/q_m b)$	K (L/mg)	0.430 ± 0.04	
				q_m (mg/g)	70.5 ± 2.9	
				R^2	0.985 ± 0.006	

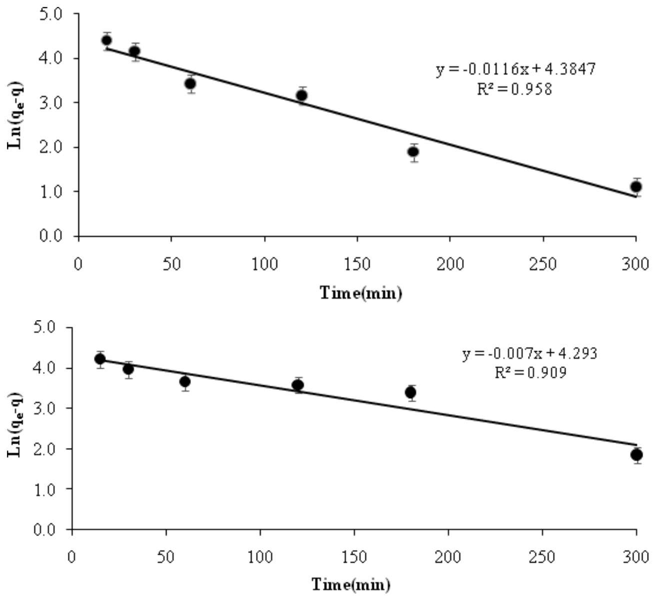

Fig. 7. Pseudo first order fitting of the adsorption of benzo (a) anthracene on (a) sewage sludge-based activated adsorbent (SW-A2) (b) commercially available adsorbent (AC) at 25°C, shaking speed of 150 rpm, initial concentration = 10 ppm, and adsorbents dosage of 0.10 g/L.

with remarkably higher R^2 values. The fact that the adsorption isotherm for SW-A2 fits Freundlich model indicate that the adsorption sites on SW-A2 are not equivalent whereas those for AC are equivalent as evident from their adherence to Langmuir model. This finding is consistent with the pore size distribution obtained in Fig. 2 for both adsorbents as discussed in section 3.1.

3.2.4. Adsorption kinetics

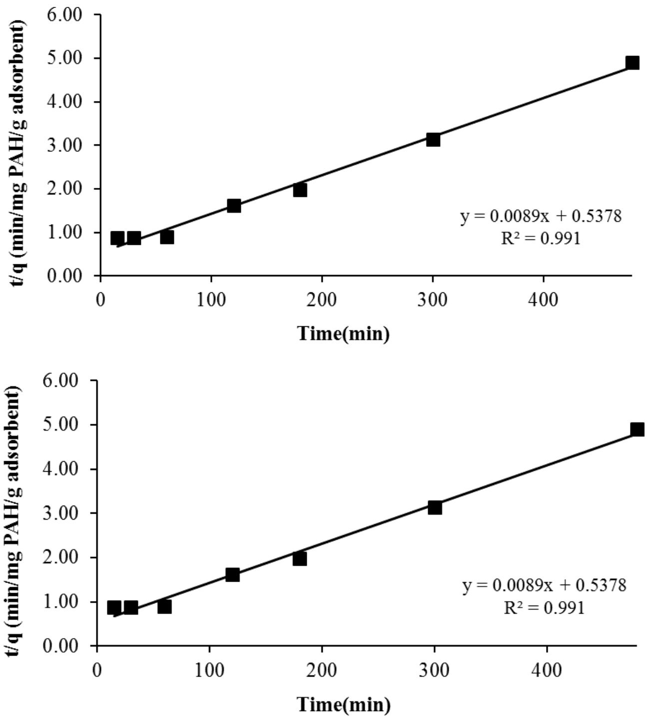

Fig. 8. Pseudo second order fitting of the adsorption of benzo (a) anthracene on (a) sewage sludge-based activated adsorbent (SW-A2) (b) commercially available adsorbent (AC) at 25°C, shaking speed of 150 rpm, initial concentration = 10 ppm, and adsorbents dosage of 0.10 g/L.

Experimental data of the adsorption of benzo (a) anthracene on SW-A2 and AC were fitted using pseudo first order and pseudo second-order models. Figs. 7 and 8 show the plot of the linearized forms of the two models for both adsorbents. Inspections of these figures reveals that both adsorbents fits the pseudo second order model bitter than the pseudo first order model as evident from R^2 values. Analysis of the fitting parameters for both adsorbents reveal that the pseudo second order rate constants are 1.55 $\times 10^{-4}$ and 1.44 $\times 10^{-4}$ mg g^{-1} min^{-1} for SW-A2 and AC, respectively. Whereas q_e are 112 and 101 mg/g for SW-A2 and AC, respectively. These results reveal that and based on solely the chemical kinetics that SW-A2 is more efficient adsorbent than AC with faster kinetics and larger adsorption capacity. Hence the results of this work renders the newly prepared adsorbent as a novel, efficient, and highly promising material for the removal of the carcinogenic PAHs from waterbodies.

4. Conclusions

The results presented in this paper proved the successful development and applicability of a new adsorbent material using sewage sludge. Several adsorbent materials were prepared and compared for the removal of benzo (a) anthracene from water. These included activation of unwashed and washed sludge samples at 680°C (S-A and SW-A, respectively), the activation of the washed sample at 750°C (SW-A2), and commercially available activated carbon (AC) for comparison purposes. The results showed that the adsorbent material prepared through activation at 750°C (SW-A2) had the best microporous structure and enhanced removal efficiency. The BET adsorption isotherm surface area (S_{BET}) for the commercial activated carbon testedwas 1236 m^2/g, compared to the selected adsorbent (SW-A2) which was 2189 m^2/g which is almost double of that of AC, indicating higher efficiency for the removal of pollutants. Adsorption isotherms for the removal of benzo (a) anthracene were fitted to both Langmuir and Freundlich models. Based on the regression coefficient, Freundlich model was found to better fit the adsorption data on the sludge-based adsorbent in comparison to Langmuir model with K_f and n of 100.8 mg/g (L/mol)$^{-0.763}$ and 1.31, respectively. On the other hand, Langmuir model was found to better fit the adsorption data for the commercial adsorbent with K and q_{max} of 0.43 L/mg and 70.5 mg/g, respectively. Kinetics analysis of the adsorption process reveals that both SW-A2 and AC follow second order kinetics with rate constants of 1.55 $\times 10^{-4}$ g mg^{-1} min^{-1} and 1.44 $\times 10^{-4}$ g mg^{-1} min^{-1} for SW-A2 and AC, respectively. Finally, the results of this study provided an efficient and environmentally friendly application of an adsorbent prepared from sewage sludge for the removal of PAHs in water.

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