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ORIGINAL ARTICLE

Effect of oxidation treatment of multi-walled carbon nanotubes on the adsorption of pentachlorophenol from aqueous solution: Kinetics study

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Abstract Multi-walled carbon nanotubes (MWCNTs) were oxidized using different oxidizing agents and the produced oxidized MWCNTs were characterized using different techniques. IR measurements showed the presence of carboxylic acid function groups especially for the MWCNTs oxidized with nitric acid and hydrogen peroxide. The adsorption of pentachlorophenol (PCP) to pristine and oxidized multi-walled carbon nanotubes (MWCNTs) has been studied. The results showed that the oxidation of the MWCNTs decreased their abilities to adsorb PCP compared with the pristine MWCNTs. The adsorption was studied kinetically and the results showed that the adsorption process occurs in two different steps. The first step involves the transfer of PCP to the surface of the oxidized MWCNTs, which was very fast due to the diffusion of PCP from the liquid phase to the solid phase. This step followed by a second slower step of adsorption could be due to intra-particle diffusion.

1. Introduction

Chlorophenolic compounds, such as pentachlorophenol (PCP), are generally generated from a number of industrial manufacturing processes. Those compounds comprise the bulk of the environmental pollutants. The adsorption over solid adsorbent is a well-established technique used for the removal of different pollutants, especially chlorophenols, from domestic and industrial effluents. Many research studies have showed the ability of carbon nanotubes (CNTs) to adsorb different pollutants from various aqueous samples (Abdel Salam and Burk, 2008a,b, 2009a,b; Abdel Salam et al., 2010a,b; Liang et al., 2004, 2005; Cai et al., 2003a, 2005; Lu et al., 2005a,b; Ambrosi et al., 2005; Pan et al., 2005; Liu et al., 2004; Stafiej and Pyrzynska, 2007; Ye et al., 2007; Xie et al., 2007; Tuzen and Soylak, 2007). This ability of CNTs is due to strong interaction between the CNTs surface and the pollutants due to the unique structure of CNTs as a result of the delocalized π-electrons on the hexagonal arrays of carbon atoms in grapheme sheets of CNTs surface. Although, there is only one study

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exploring the potentiality of using pristine MWCNTs (Cai et al., 2005) for the removal of chlorophenols (CPs) from aqueous solution, but there is no study on the adsorption behavior of PCP, as an example of CPs, on oxidized MWCNTs. Oxidized MWCNTs were used previously for the adsorption of lead, copper and cadmium ions (Li et al., 2003a,b), and trihalomethanes (Lu et al., 2005a) from aqueous solutions. These research studies concluded that the oxidized CNTs show higher adsorption capacities for the target pollutants compared with other adsorbents such as activated carbon. Investigations of the adsorption kinetics and the uptake of different analytes by CNTs are very limited. More work is needed to understand the adsorption behavior of various analytes on MWCNTs. Kinetic studies are very important factor for understanding the mass transfer of different analytes for the bulk phase to the adsorbent and to determine the different conditions affecting this transfer. In this paper, multi-walled carbon nanotubes were oxidized with different oxidizing agents; namely nitric acid, hydrogen peroxide, and potassium permanganate were studied first to find the most suitable oxidizing agent. The adsorption of PCP to different oxidized form of multi-walled carbon nanotubes (MWCNTs) was studied to determine the effect of introducing oxygen-containing functional group on the adsorption of PCP on the MWCNTs. The study focuses on the kinetics of the adsorption.

2. Experimental section

2.1. Oxidation and characterization of MWCNTs

MWCNTs were purchased from Sun Nanotech (China) and were used as received. The MWCNTs (2.0 g) were added to 200 mL solutions of 8 M HNO_3, 18% H_2O_2 and 1 M $KMnO_4$ (acidified), separately. The HNO_3 suspension was refluxed at 140 °C for 4 h. The H_2O_2 and $KMnO_4$ suspensions were heated at 80 °C for 4 h. MWCNTs oxidized with $KMnO_4$ were further treated with H_2SO_4 in order to remove MnO_2 from the product. The three suspensions were then washed with distilled water until the wash water was pH neutral and a centrifuge (3500 rpm for 30 min) was used for solid-liquid separation. The solid products were dried in air at 50 °C. Infrared spectra measurements of the oxidized MWCNTs were performed on an ABB Bomem MB Series FTIR spectrometer using KBr pellets. The specific surface area of the pristine and oxidized MWCNTs were measured by nitrogen adsorption at 77 K using the single point BET method with a Micromeretics model 2200 surface area analyzer. All the chemicals used were purchased from Sigma–Aldrich.

2.2. Analytical procedure

For the determination of PCP concentration in aqueous using gas chromatograph with an electron capture detector (GC–ECD) an acetylation step is required. For acetylation of PCP, 3.0 mL of 5% K_2CO_3 solution was added to the aqueous solution of PCP and stirred for 1 min, followed by the addition of 2.0 mL of acetic anhydride and stirred vigorously for 2 min to ensure complete acetylation of the PCP. The acetylated PCP was extracted into 4.0 mL *n*-hexane with 2 min of shaking. The acetylated PCP was analyzed using a Varian 3600 GC-ECD and a 30 m × 0.25 μm id × 0.25 μm film thickness DB-5 silica capillary column (Varian). The GC conditions were as follows: helium as the carrier gas at a flow rate of 1 mL/min; nitrogen as the make-up gas at a flow rate of 27 mL/min; injector temperature, 300 °C; column initial temperature, 70 °C; and final temperature, 280 °C; with heating rate, 15 °C/min; detector temperature, 300 °C. The sample size injected was 1 μL.

2.3. Solid phase extraction (SPE) of PCP

Stock solutions were prepared by dissolving pentachlorophenol (Aldrich 99.0 + %) in deionized water followed by serial dilutions. Three milliliters inert glass tubes (Supelco) were used as the packing cartridges for SPE and 5.0 mg of the MWCNTs were packed into the cartridge. Teflon upper and lower frits (20 μm pore size) at each end of the cartridge were used to hold the MWCNTs packing in place. The PCP solution (1.0 mg/L) was passed through the cartridge at a flow rate of 2 mL/min using suction. The aqueous solution was then analyzed for the remaining PCP.

2.4. Kinetics study

The kinetics study was done using 14 mL glass vials with Teflon-lined caps. In each vial, 10 mL of the 1.0 mg/L PCP solution was added to 5.0 mg of MWCNTs. The vials were mounted on a shaker and shaken at 23 ± 2 °C for different intervals of time. Each suspension was centrifuged at 3000 rpm and the supernatant solution was analyzed for PCP.

3. Results and discussion

3.1. Infrared spectroscopy

The IR measurements were performed in order to confirm the formation of acidic functional groups upon oxidation and the spectra are presented in Fig. 1. The figure shows that the oxidized MWCNTs spectra have absorption peaks at 1730 cm^{-1} corresponding to the stretching vibration of C=O from the carboxylic acid groups (–COOH) (Sun et al., 2003; Chen et al., 1998, 2001; Shaffer et al., 1998; Hamon et al., 2001; Koshio et al., 2001). This absorption peak was very clear and strong for the MWCNTs oxidized with HNO_3, whereas it was much weaker with the MWCNTs oxidized with H_2O_2 and $KMnO_4$. A broad peak at approximately 3500 cm^{-1} was

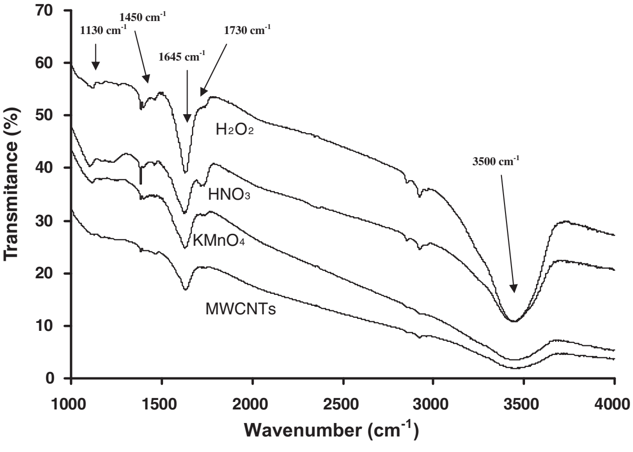

Figure 1 IR spectra of MWCNTs before and after oxidation.

much stronger in the H_2O_2 and HNO_3-treated MWCNTs. This peak is characteristic of an O–H stretch. It is due to either moisture, alcohol or phenol, OH^- or carboxylic groups in these samples. The aromatic C=C stretch is observed at 1645 cm^{-1} and at 1450 cm^{-1} for all of the spectra with comparable intensities. The peak at 1450 cm^{-1} is more intense for the MWCNTs oxidized with HNO_3 and H_2O_2 than in the pristine and MWCNTs oxidized with $KMnO_4$. The weak, broad peaks at 1130 cm^{-1} are found in all the oxidized MWCNTs which could be due to a C–O stretch from either a phenol or lactone. These results show clearly that the MWCNTs can be oxidized and that carboxylic, phenolic and lactone groups are likely obtained.

3.1.1. *Specific surface area measurements*

The specific surface areas of the pristine and oxidized MWCNTs were measured using the BET method by nitrogen adsorption at 77 K. The specific surface area for the pristine MWCNTs was found to be 148 ± 5 m^2/g, which was in good agreement with the other reported values (Cai et al., 2003a,b, Li et al., 2003b, Li and Yuan, 2003). The specific surface area of the oxidized MWCNTs was slightly lower; 144 ± 6 and 140 ± 3 m^2/g for the MWCNTs oxidized with H_2O_2 and HNO_3, respectively. For the MWCNTs oxidized with $KMnO_4$ the specific surface area of the oxidized tube was smaller; 106 ± 3 m^2/g. This may suggest that $KMnO_4$ as an oxidizing agent was very strong and effective enough to remove most of the amorphous carbon as well as damaging some of the MWCNTs.

3.2. *Adsorption of PCP on pristine and oxidized MWCNTs*

The adsorption of PCP to pristine and oxidized MWCNTs was studied after packing the CNTs in a solid-phase extraction (SPE) cartridge and the results are shown in Fig. 2. It is clear from the figure that oxidation of the MWCNTs decreased the adsorption of PCP in general. The amount of PCP adsorbed was found to be 91.9% for the pristine MWCNTs, and 76.4%, 77.8%, and 43.4% for the MWCNTs oxidized with HNO_3, H_2O_2, and $KMnO_4$, respectively. It was expected that PCP, with its phenolic group would strongly interact with the oxygen-containing acidic functional groups; carboxylic, phenolic and lactonic, that were created upon oxidation of the MWCNTs, and consequently the adsorption of PCP would be increased. However, the adsorption of PCP on the oxidized MWCNTs actually decreased compared with the pristine MWCNTs. This may be explained as follows. Generally, in the case of adsorption from the liquid phase to solid phase, the adsorption capacity of any carbonueous adsorbent for aromatic compounds, such as PCP, depends on the following factors (Haghseresh et al., 2002):

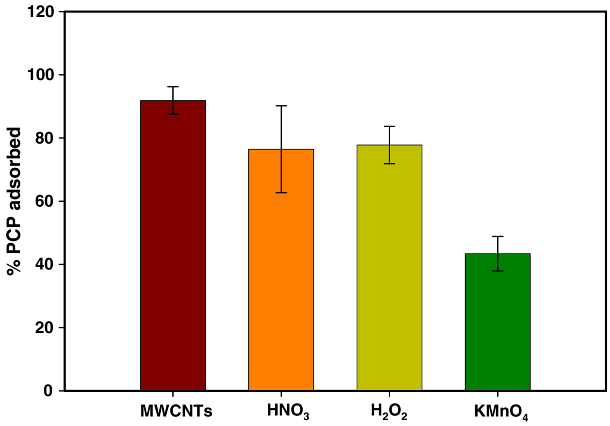

Figure 2 Adsorption of PCP by pristine MWCNTs and different oxidized MWCNTs. The error bars represent three standard deviations, 5 mg of the adsorbent, 2 mL/min flow rate, PCP concentration is 20 ng/mL.

- the physical nature of the adsorbent, such as pore structure, and the presence of surface functional groups,
- the nature of the adsorbate, such as its pK_a, functional groups present, polarity, molecular weight, and size,
- the conditions of the solution, such as pH, ionic strength as well as the adsorbate concentration.

From the physical nature of the adsorbent point of view, one of the important features for most of carbon adsorbent is their aromaticity due to the presence of the delocalized π-electrons. Pristine MWCNTs, mostly, have a uniform surface with many delocalized π-electrons, which increases their adsorption capability in comparison with other carbonueous materials. It was found that the adsorptive properties of carbonueous material are determined mostly by its surface chemical composition. The presence of oxygen and hydrogen within the surface groups crucially affected the adsorptive properties of the adsorbent. Functional groups and delocalized electrons of the MWCNTs structure determine the chemical character of the MWCNTs surface (León and Radovic, 1994). It was reported that the presence of the carboxyl and hydroxyl groups inhibited the adsorption of phenolic compounds. This effect can be explained by PCP adsorption that is governed by "π–π dispersion interaction" between the MWCNTs and the aromatic ring of the PCP. Oxygen-containing functional groups can localize the π-electrons, and consequently, remove them from the π-electron system of the MWCNTs (Couglin and Ezra, 1968), lowering the dispersive forces with PCP π-electron. Another explanation is the adsorption of water molecules to the oxygen-containing function groups on the surface of the oxidized MWCNTs by the means of hydrogen bonding (Dabrowski et al., 2005) which is the "solvent effect".

It can be concluded here that the oxidation of MWCNTs and the introduction of oxygen-containing functional groups inhibit the adsorption of PCP due to the localization of the π-electron of the MWCNTs as well as the solvent effect.

3.3. *Kinetics study of the adsorption of PCP on pristine and oxidized MWCNTs*

The kinetics of PCP adsorption in aqueous solution on pristine or oxidized MWCNTs were studied and the results are presented in Fig. 3. Within the first 5 min 88% of the PCP was adsorbed by the pristine MWCNTs whereas 88.9%, 87.1%, and 75.0% by the oxidized MWCNTs; H_2O_2, HNO_3, and $KMnO_4$, respectively. The adsorption capacity reached 1.76 mg/g for the pristine MWCNTs and 1.78, 1.74, and 1.50 mg/g for the MWCNTs oxidized with H_2O_2, HNO_3, and $KMnO_4$, respectively. The adsorption of PCP by pristine MWCNTs and the oxidized MWCNTs (HNO_3, H_2O_2) achieved equilibrium gradually at about 60 min and the final adsorption capacity

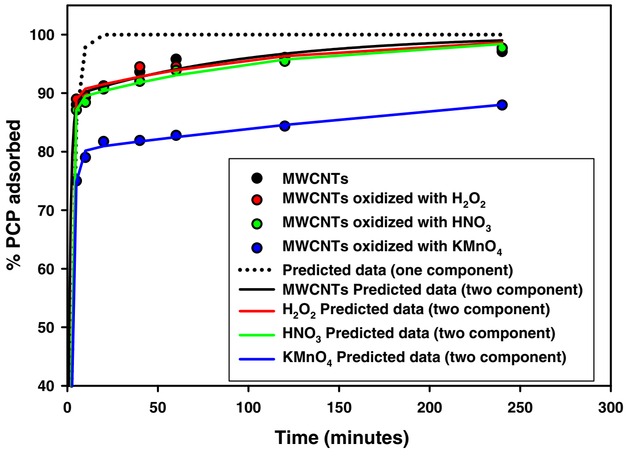

Figure 3 The kinetic model for the adsorption of PCP to pristine and oxidized MWCNTs and the comparison between the experimental data and the predicted data (one component, two component).

reached 1.92, 1.88, and 1.89 mg/g, respectively. For the other oxidized MWCNTs it took 120 min to reach the equilibrium concentration of 1.69 mg/g and for the oxidized MWCNTs $KMnO_4$. Comparing the kinetics results of the %PCP adsorbed by the oxidized MWCNTs with the SPE application (Fig. 2) showed that the oxidized MWCNTs adsorbed less if they are packed inside the SPE cartridge. The explanation for that may be attributed to the fact that the diffusion of the PCP molecules during the kinetics experiment was fast due to the shaking of the solution during the experiment. This shaking enhanced the transfer of the PCP from the liquid phase to the solid phase as well as exposed most of the MWCNTs to the PCP molecules.

3.4. Kinetic modelling

The transport of the PCP from the aqueous solution to the surface of the adsorbent may occur in several steps (Srivastava et al., 2006). The overall adsorption process may be controlled by one or more steps, e.g. film or external diffusion, pores diffusion, surface diffusion and adsorption on the pore surface, or a combination of more than one step.

In order to distinguish between the different adsorption steps, the following kinetic model was adopted.

The adsorption of PCP molecules from aqueous solution to an active site S, on the adsorbent (MWCNTs or AC) can be represented as:

$$\text{Analyte}_{(\text{aq.})} + \text{Absorbent}_{(\text{aq.})} \underset{\text{desorption}}{\overset{\text{adsorption}}{\rightleftarrows}} \text{Analyte-adsorbent complex}_{(\text{aq.})}$$

or

$$\text{PCP} + \text{S} \underset{k_{-1}}{\overset{k_1}{\rightleftarrows}} \text{PCP} - \text{S} \tag{1}$$

where PCP − S is the PCP adsorbed and k1 and k−1 are the adsorption and desorption rate constants.

The rate of formation of the PCP − S is determined by the following equation:

$$\frac{d[\text{PCP} - \text{S}]}{dt} = -\frac{d[PCP]}{dt} = k_1[\text{PCP}][\text{S}] \tag{2}$$

In the case where [S] ⋙ [PCP] and before significant adsorption has occurred, the adsorption is considered a pseudo-first-order, and the rate of adsorption can be simplified as:

$$\frac{d[\text{PCP} - \text{S}]}{dt} = -\frac{d[\text{PCP}]}{dt} = k_1[\text{PCP}] \tag{3}$$

By assuming that the adsorption of PCP from the aqueous phase to the solid phase occurs in different steps; film or external diffusion, pores diffusion, surface diffusion and adsorption on the pore surface, and if PCP molecules undergo a first-order or pseudo-first-order adsorption to the solid adsorbent, the sum of the concentrations of all PCP adsorbed at the solid adsorbent at time t, $C_{\text{PCP-S}}(t)$ increases exponentially as:

$$C_{\text{PCP-S}}\ (t) = \sum_{i=1}^{n} C_i^0[1 - \exp(-k_i t)] \tag{4}$$

where C_i^0 is the initial concentration of PCP_i, the ith component. The concentration of PCP − S adsorbed to the adsorbent, $C_{\text{PCP-S}}\ (t)$, is determined as a function of time by measuring the concentration of PCP remaining in solution. The percentage of PCP remaining in the aqueous solution and the percentage adsorbed to the solid adsorbent can be calculated using the following equation:

$$\%\ \text{removed} = \frac{100(C_0 - C_e)}{C_0} \tag{5}$$

and

$$\%\ \text{remaining} = 100 - \%\ \text{removed} \tag{6}$$

where C_0 is the initial solution phase PCP concentration (mg/L) and C_e the equilibrium solution phase PCP concentration (mg/L).

The experimental data were analyzed for discrete values of the adsorption rate constants by nonlinear regression. One of the important conditions for the application of the kinetic model is the speed convergence of the regressions, and there should be a reasonable fit (as measured by correlation coefficient, R^2) with the kinetic data. The kinetic adsorption curves of PCP may suggest that adsorption onto the surface occurs quickly, followed by a slower diffusion step. The short time needed to reach equilibrium also suggests that both the pristine and oxidized MWCNTs, excluding the tubes oxidized with $KMnO_4$, are superior adsorbents to remove PCP from aqueous solution. The experimental data were analyzed for discrete values of the adsorption rate constants by a nonlinear regression. Fig. 3 shows the relation between the % of PCP adsorbed from the aqueous solution to the adsorbent and time for pristine and oxidized MWCNTs. These data were fitted using the one component model and the values for kinetic parameters of the PCP adsorption are presented in Table 1. From this table, it is clear that applying the one component kinetic model for the adsorption of PCP to MWCNTs converges well and the value of the R^2 was acceptable (more than 0.95), whereas in the case of the CNTs oxidized with $KMnO_4$, the R^2 was not acceptable with a value of 0.75. This may be due to the fact that $KMnO_4$ was a harsh oxidizing agent, which destroyed the crystalline structure of the MWCNTs and led to the formation of amorphous carbon. Comparing the one component predicted data (dotted line) with the experimental data of the PCP adsorption to the pristine and the oxidized tubes shows significant differences. Again, the fitted data did not match

Table 1 Kinetic parameters of the adsorption of PCP to pristine and oxidized MWCNTs using different kinetic models (one component and two components).

	Kinetic parameters				
	$\%C_1$	$k_1 \times 10^{-1}(\text{min}^{-1})$	$\%C_2$	$k_2 \times 10^{-3}(\text{min}^{-1})$	R^2
One component model					
MWCNTs	100.0 ± 2.7	3.84 ± 0.9			0.967
H_2O_2	100.0 ± 2.7	4.02 ± 1.0			0.969
HNO_3	100.0 ± 3.0	3.67 ± 1.0			0.960
$KMnO_4$	100.0 ± 7.1	2.15 ± 0.8			0.753
Two component model					
MWCNTs	89.2 ± 1.7	7.85 ± 2.4	10.8 ± 2.6	9.9 ± 7.5	0.998
H_2O_2	90.0 ± 1.3	8.30 ± 2.4	10.0 ± 2.8	8.4 ± 6.4	0.999
HNO_3	88.7 ± 0.9	7.53 ± 1.2	11.4 ± 1.9	8.2 ± 4.0	0.999
$KMnO_4$	80.1 ± 0.7	5.34 ± 0.4	19.9 ± 2.8	2.1 ± 3.9	0.999

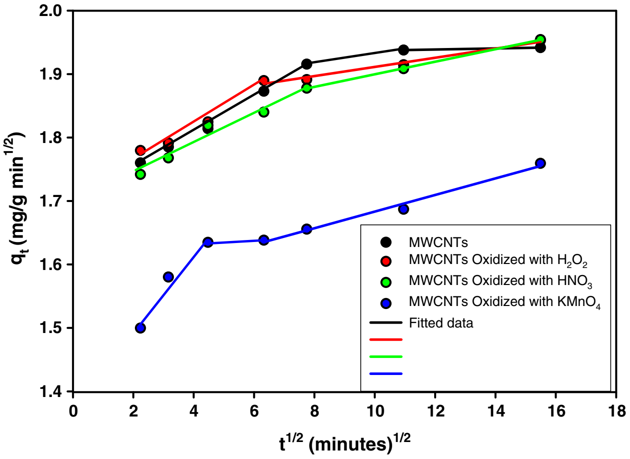

Figure 4 Weber–Morris intra-particle diffusion plots for the adsorption of PCP to pristine and oxidized MWCNTs.

the experimental data at all. This may imply that the adsorption of PCP mainly in more than one step. The two component kinetic model was used to fit the experimental data and the results are presented in Table 1 and Fig. 3. It was obvious from the graph and the table that the two component kinetic model fits much better than the one component kinetic model as the predicted data fitted well with the experimental data with very good correlation coefficients (R^2 = 0.998 and 0.999). In general, the %PCP adsorbed during the first step ($\%C_1$) was significantly higher than the second step (($\%C_2$) in the case of pristine MWCNTs; 89.2%, and the oxidized MWCNTs; 90.0% and 88.7%, for the MWCNTs oxidized with H_2O_2 and HNO_3, respectively. The differences between the first rate constant for the pristine and the oxidized MWCNTs were not significant. The first component may be attributed to the fast diffusion of the PCP from the aqueous phase to the surface of the MWCNTs. The fact that the difference between the pristine and the oxidized MWCNTs for the first component was not significant may indicate that the oxidation of the MWCNTs with H_2O_2 and HNO_3 was successful. This oxidation increases the hydrophilic character of the MWCNTs through the introduction of carboxylic and phenolic groups on the surface of the tubes without destroying the tube like what happened in the case of $KMnO_4$. Analysis of the second component shows that nearly 10% of the PCP was adsorbed on the pristine and the oxidized MWCNTs. This step may be attributed to intra-particle diffusion in aggregates of MWCNTs. The probability of intra-particle diffusion was explored by using the intra-particle diffusion model (Weber and Morris, 1963).

$$q_t = k_{id}t^{1/2} + C \tag{7}$$

where, q_t is adsorption capacity at any time (t), k_{id} is the intra-particle diffusion rate constant (mg/g min$^{1/2}$) and C (mg/g) is a constant proportional to the thickness of the boundary layer. Plotting q_t versus $t^{1/2}$ a "Weber–Morris plot" (Weber and Morris, 1963) can give an indication of the dependency of adsorption on the intra-particle diffusion. If the plot gives a straight line, then the adsorption (or sorption) process is controlled by intra-particle diffusion only, and if it exhibits multi-linear plots, then there are two or more steps affecting the adsorption process. Fig. 4 shows the Weber–Morris plot of the PCP adsorption to pristine and oxidized MWCNTs. It is clear from the figure that the adsorption of PCP to the pristine and the oxidized MWCNTs two different straight lines. This may indicate that the adsorption of PCP to MWCNTs, both pristine and oxidized MWCNTs, is controlled by two steps. The first part of the straight line may be due to the adsorption to the surface of the MWCNTs due to the fast diffusion from the aqueous phase to the solid phase of the MWCNTs, whereas the second part could be due to the intra-particle diffusion. The slope of the q_t versus $t^{1/2}$ in the region where the intra-particle diffusion control the adsorption is defined as the k_{id} is the intra-particle diffusion rate constant (mg/g min$^{1/2}$). The values for k_{id} and C (mg/g) (a constant proportional to the thickness of the boundary layer) are tabulated in Table 2. It is clear from this table that diffusion constant value

Table 2 Intra-particle diffusion parameters for the adsorption of PCP to pristine and oxidized MWCNTs.

	Intra-particle diffusion parameters		
	k_{id} (mg/g min$^{1/2}$)	C (mg/g)	R^2
MWCNTs	$(8.38 \pm 0.0) \times 10^{-4}$	1.93 ± 0.00	1.000
H_2O_2	$(7.21 \pm 0.8) \times 10^{-3}$	1.84 ± 0.01	0.979
HNO_3	$(9.90 \pm 0.1) \times 10^{-3}$	1.80 ± 0.00	0.999
$KMnO_4$	$(1.31 \pm 0.1) \times 10^{-2}$	1.55 ± 0.00	0.987

for the pristine MWCNTs is the lowest compared with the oxidized tubes, which may indicate the less dependency of the pristine MWCNTs on the intra-particle diffusion. For the oxidized MWCNTs the difference between the k_{id} values was not significant between the MWCNTs oxidized with HNO_3 and H_2O_2, whereas this value was the highest in the case of the tubes oxidized with $KMnO_4$. There was not a clear explanation for this criterion.

4. Conclusions

The adsorption of PCP to pristine and oxidized MWCNTs was studied and the results showed that decreased the adsorption of PCP on oxidized MWCNTs. This mainly was due to the introduction of the oxygen-containing functional groups on the MWCNTs surface upon oxidation. Although the adsorption of PCP onto the oxidized MWCNTs was less than the pristine MWCNTs, but they can be considered good adsorbents for the removal of PCP from aqueous solutions. The kinetics study showed that the adsorption process occurs in two steps. The first step involves the transfer of PCP to the surface of the oxidized MWCNTs, which was very fast due to the diffusion of PCP from the liquid phase to the solid phase. This step followed by a second slower step of adsorption which could be due to intra-particle diffusion.

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