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The influence of an electrolyte on the adsorption of 4-chlorophenol onto activated carbon and multi-walled carbon nanotubes

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ABSTRACT

The adsorption of 4-chlorophenol from deionized water and electrolyte solutions onto activated carbon and carbon nanotubes has been studied. The kinetics data were fitted well to the pseudo-second order model. It was found that inorganic salts enhanced the adsorption kinetics onto the carbon nanotubes but did not affect the adsorption kinetics onto the activated carbon. The adsorption was also analyzed as a function of the solution concentration at the equilibrium. The experimental data received were found to be well described by the Freundlich isotherm equation. The experimental data indicated that the presence of salts in the solution did not significantly affect the capacity of the adsorption of the MWCNTs. The adsorption capacity of the activated carbon for the 4-chlorophenol was higher in the electrolyte solutions than in the water, and further indicated that the removal of the 4-CP increased with the increase in the ionic strength of the solution.

Keywords: 4-Chlorophenol; Activated carbon; Carbon nanotubes; Adsorption; Electrolyte

1. Introduction

Chloro-organic compounds including chlorophenols are common water contaminants, which are generated by coal conversion, petrochemical, pharmaceutical, paper, and pesticide producing industries [1]. In drinking water, chlorophenols emit an unpleasant odor and flavor at the concentrations below 0.01 mg/L [2] and are poisonous to aquatic life, plants, and humans [1]. Therefore, they are classified as priority pollutants by the European Union [3] and US Environmental Protection Agency [4]. In view of the high toxicity, the wide prevalence and poor biodegradability of chlorophenols, it is necessary to remove them from wastewater, groundwater, surface water, and especially potable water.

Currently, many treatment techniques such as biological degradation [5], chemical oxidation [6], and adsorption are the most widely used methods for removing phenolic compounds from the water environment. Among the advanced chemical or physical treatments, adsorption is considered as more effective and less expensive than other technologies. The chlorophenols were adsorbed onto low-cost natural adsorbents [7,8], synthetic resins [8], silica [9], carbon nanotubes [10,11], carbon fibers [12], carbon-coated monolith [13], and mainly on activated carbons [14–20]. Among all the applied sorbents, the activated carbons are the most widely used adsorbents due to their excellent adsorption abilities for organic compounds [21]. The high adsorption capacities of the

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activated carbons are usually related to their high surface area, pore volume, and pore size distribution. The effectiveness of the adsorption of organic pollutants on the activated carbons depend also on the physicochemical properties of the substance adsorbed (e.g. molecular weight, solubility, polarity, type of functional groups), the solution chemistry (e.g. pH, ionic strength), and the adsorption temperature [21]. The effect of some of these factors on the kinetics and adsorption equilibrium of different organic pollutants, including chlorophenols, has already been described but some still require examination.

Dissolved salts in the aqueous environment have been found to affect the adsorption capacity of many organic compounds on the activated carbon [21,22]. The salts are involved in a variety of mechanisms including interactions with the adsorbates, both in the solution and on the carbon surface as well as in the modification in the charge on the carbon surface [22]. These interactions, between the adsorbent-ions and adsorbate-ions, as well as between the adsorbate-ion-adsorbent, may cause a substantially different adsorption behavior. This influence is particularly important in the applications where salts exist naturally in water, such as drinking water treatment systems [22]. Moreover, the presence of an electrolyte is necessary to operate the electrode during the removal of the chlorophenols by adsorption/electrosorption [23,24]. Therefore, the investigation of the salt effect on the adsorption isotherms and kinetics is necessary for effective water purification. It is also important because the majority of laboratory studies on the adsorption of chlorophenols on activated carbon is carried out in distilled/deionized water. As a consequence, the effect of salts on the adsorption of chlorophenols is not fully understood.

The present study, was undertaken to provide a clearer understanding of the influence of salts on the adsorption of 4-chlorophenol onto the granular activated carbon and multi-walled carbon nanotubes. Three salts of strong base and strong acids (Na_2SO_4, NaCl and $NaNO_3$) were chosen as electrolytes. The effect of the nature of the electrolyte and its concentration on the adsorption kinetics and equilibrium of 4-chlorophenol were examined.

2. Experimental

2.1. Materials

Electrolytes—sodium chloride, sodium nitrate, and sodium sulfate were purchased from POCh (Gliwice, Poland). 4-Chlorophenol (4-CP) was purchased from Sigma (St Louis, MO, USA). Acetic acid and HPLC-grade acetonitrile were purchased from Across Organics (Geel, Belgium).

As adsorbents the granular activated carbon Filtrasorb 400, F-400 (Chemviron, Feluy, Belgium), and multi-walled carbon nanotubes, MWCNTs (Chengdu Organic Chemical Co. Ltd, China) were chosen. Prior to use, the activated carbon was pretreated by ash removal by using HCl and HF concentrated acids and rinsed several times with deionized water. Both adsorbents were dried in an oven at 130°C to constant weight and stored in a desiccator until use. The BET surface area of the adsorbents were obtained on the basis determined low-temperature adsorption-desorption isotherms (ASAP 2020, Micromeritics, Norcross, USA).

2.2. Adsorption procedure

Adsorption experiments were carried out in a batch mode with the following procedure. For each time, 0.02 g of activated carbon and 40 mL of 4-CP or 0.03 g of MWCNTs and 20 mL of 4-chlorophenol solution were mixed in an Erlenmeyer flask, which was then shaken. The chlorophenol was adsorbed from the water and from 0.05 and/or 0.1 mol/L solutions of sodium chloride, sodium nitrate, and sodium sulfate. After mixing, the solutions were filtered and analyzed by high-performance liquid chromatography with UV detection.

The kinetic studies were conducted for an initial 4-CP concentration of 0.5 mmol/L at 25°C. The adsorption kinetics were studied from water and electrolyte solutions for 2 h onto the carbon nanotubes and 8 h onto the activated carbon, respectively. The amount of adsorption at time t, q_t (mmol/g), was calculated by the following equation:

$$q_t = V\frac{C_0 - C_t}{m} \tag{1}$$

where C_0 and C_t are the initial concentration and adsorbate concentration at time t (mmol/L), V is the volume of the solution (L), and m is the mass of the adsorbent (g).

In the adsorption isotherm studies, solutions of 4-CP with different initial concentrations (0.1, 0.2, 0.4, 0.6, 0.8, and 1.0 mmol/L) were added to an Erlenmeyer flask containing 0.02 g of activated carbon or 0.03 g of MWCNTs. The equilibrium time was set as 6 h for the activated carbon and 2 h for the carbon nanotubes. The chlorophenol was adsorbed from the distilled water and from the 0.05 or 0.1 mmol/L solutions of NaCl, $NaNO_3$, and Na_2SO_4. The uptake of

4-CP at equilibrium, q_e (mmol/g), was calculated by equation:

$$q_e = V\frac{C_0 - C_e}{m} \tag{2}$$

where C_e is equilibrium concentration of 4-CP (mmol/L) in solution.

2.3. Analytical method

For determination of 4-chlorophenol concentration, a high-performance liquid chromatography method with UV detection (Shimadzu LC-20, Kyoto, Japan) was used. Chromatographic conditions were as follows: mobile phase—acetonitrile/water adjusted to pH 3.0 with acetic acid (50/50, v/v), flow rate 0.25 mL/min, and analytical wavelength 281 nm. The chromatographic analysis were carried out on a Phenomenex Luna C18, 2.0 × 150 mm, 3 μm column (Torrance, CA, USA).

3. Results and discussion

3.1. Adsorbents properties

Nitrogen adsorption/desorption isotherm for F-400 activated carbon and MWCNTs were determined from N_2 adsorption isotherm measured at 77 K and the results are presented in Fig. 1. The specific surface area was calculated from the BET-equation and was found to be 997 and 181 m^2/g for activated carbon and MWCNTs, respectively. The total pore volume V_t for the carbon nanotubes were found to be 0.703 cm^3/g. The micropore volume V_{mi} and mesopore volume V_{me} for the activated carbon were 0.310 and 0.113 cm^3/g, respectively.

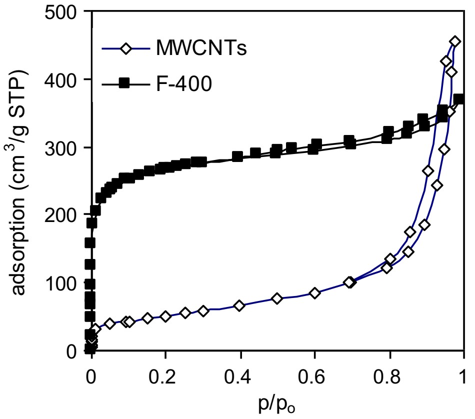

Fig. 1. Nitrogen adsorption-desorption isotherms of F-400 activated carbon and MWCNTs at 77.4 K.

3.2. Adsorption kinetics

The adsorption kinetic curves of the 4-chlorophenol are shown in Fig. 2. The adsorption equilibriums were achieved after about 5–6 h for the granular activated carbon and after 30–60 min for the carbon nanotubes. For the description of the curves $q_t = f(t)$, the equations of the pseudo-first [25] and pseudo-second order [26] were considered. The pseudo-first order model is expressed as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{3}$$

where k_1 is the rate constant of adsorption (1/min).

The pseudo-second order equation has the form:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{4}$$

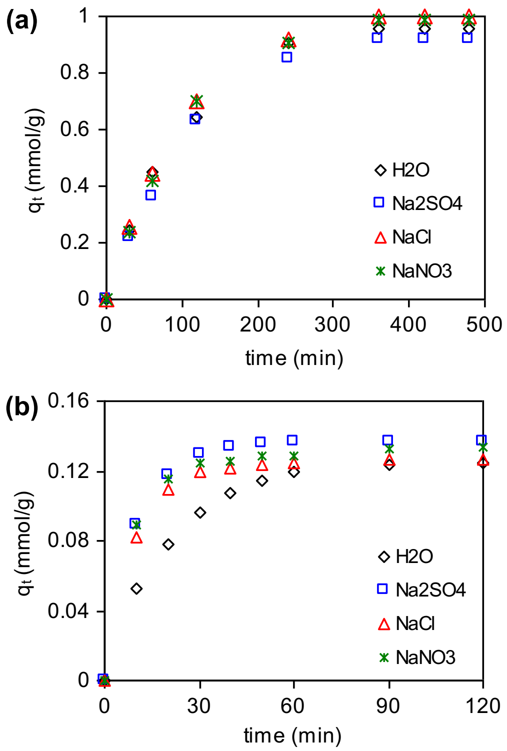

Fig. 2. Adsorption kinetics of 4-chlorophenol onto activated carbon (a) and MWCNTs (b) from water and 0.05 mol/L electrolyte solutions.

where k_2 is the rate constant of second order adsorption (g/mmol·min). The constants of both models and correlation coefficients are presented in Table 1.

The correlation coefficients for the pseudo-first order kinetic model were relatively low, whereas the pseudo-second order model gives a better fitting with the high R^2 values (>0.99). This indicates that the adsorption system belongs to the second-order kinetic model. The results show that the presence of electrolytes (and their concentrations) do not affect the adsorption kinetics of the 4-chlorophenol onto the activated carbon. The values of rate constant k_2 were very close to each other. In the case of the MWCNTs, the differences between the water and electrolyte solutions were quite pronounced. The pseudo-second order rate constants obtained for the 0.05 mol/L electrolytes are more than three times higher than that of deionized water, revealing that noticeable salt-enhanced adsorption kinetics occurs. The k_2 obtained for water, sodium sulfate, sodium chloride, and sodium nitrate were 0.492, 1.825, 1.639, and 1.655 g/mmol·min, respectively. The adsorption occurs fastest from a sodium sulfate solution. The differences observed between the sodium chloride and sodium nitrate are not significant. All of this suggests that the adsorption kinetics of the 4-chlorophenol onto multi-walled carbon nanotubes is associated with the ionic strength of the solution. The ionic strength of the 0.05 mol/L sodium sulfate solution is 0.15 mol/L and the 0.05 mol/L sodium chloride and sodium nitrate is 0.05 mol/L. The kinetics of adsorption increases with the increasing ionic strength.

The effect of salt on the adsorption of the organic compounds is studied mainly in the equilibrium conditions. The effect of the ionic strength on the adsorption kinetics has not been adequately studied. In one of the very few works, Chang et al. [27] reported that the adsorption kinetics of 2-ethyl-1-hexanol on a coal-based activated carbon increases with the salt concentration in the solution. To our best knowledge, there is no information on the effects of electrolytes and ionic strength on the adsorption kinetics on carbon nanotubes.

In order to investigate the mechanism of the adsorption, the intraparticle diffusion model (Weber–Morris model) [28] was also used. The intraparticle diffusion equation is described as:

$$q_t = k_i t^{1/2} + C_i \tag{5}$$

where k_i is the intraparticle diffusion rate constant [mmol/g·min$^{1/2}$] and C_i is the thickness of the boundary layer. Both constants were determined experimentally from the slope and intercept of plot q_t vs. $t^{1/2}$. Fig. 3 describes the plot of q_t vs. $t^{1/2}$ for 4-chlorophenol on activated carbon (left column) and MWCNTs (right column).

The slope of the Weber–Morris plot q_t vs. $t^{1/2}$ is defined as a rate parameter, a characteristic of the rate of adsorption. If intraparticle diffusion occurs, then plot q_t vs. $t^{1/2}$ will be linear and if the plot passes through the origin, then the rate limiting process is only due to the intraparticle diffusion. The multi-linearity indicates that several steps are involved. The first, sharper region is the instantaneous adsorption or the external surface adsorption. The second step corresponds to the gradual adsorption stage, where the

Table 1
Pseudo-first and pseudo-second order rate constants for adsorption of 4-chlorophenol onto activated carbon and multi-walled carbon nanotubes from water and electrolyte solutions

	Pseudo I order		Pseudo II order	
Electrolyte	k_1 [1/min]	R^2	k_2 [g/mmol·min]	R^2
F-400 activated carbon				
H_2O	0.016	0.963	0.008	0.993
Na_2SO_4 0.05 mol/L	0.017	0.925	0.007	0.991
Na_2SO_4 0.1 mol/L	0.015	0.932	0.007	0.995
NaCl 0.05 mol/L	0.016	0.932	0.008	0.994
NaCl 0.1 mol/L	0.013	0.976	0.008	0.998
$NaNO_3$ 0.05 mol/L	0.016	0.936	0.009	0.992
$NaNO_3$ 0.1 mol/L	0.017	0.907	0.009	0.991
MWCNTs				
H_2O	0.057	0.964	0.492	0.996
Na_2SO_4 0.05 mol/L	0.073	0.961	1.825	0.999
NaCl 0.05 mol/L	0.070	0.993	1.639	0.998
$NaNO_3$ 0.05 mol/L	0.063	0.952	1.655	0.999

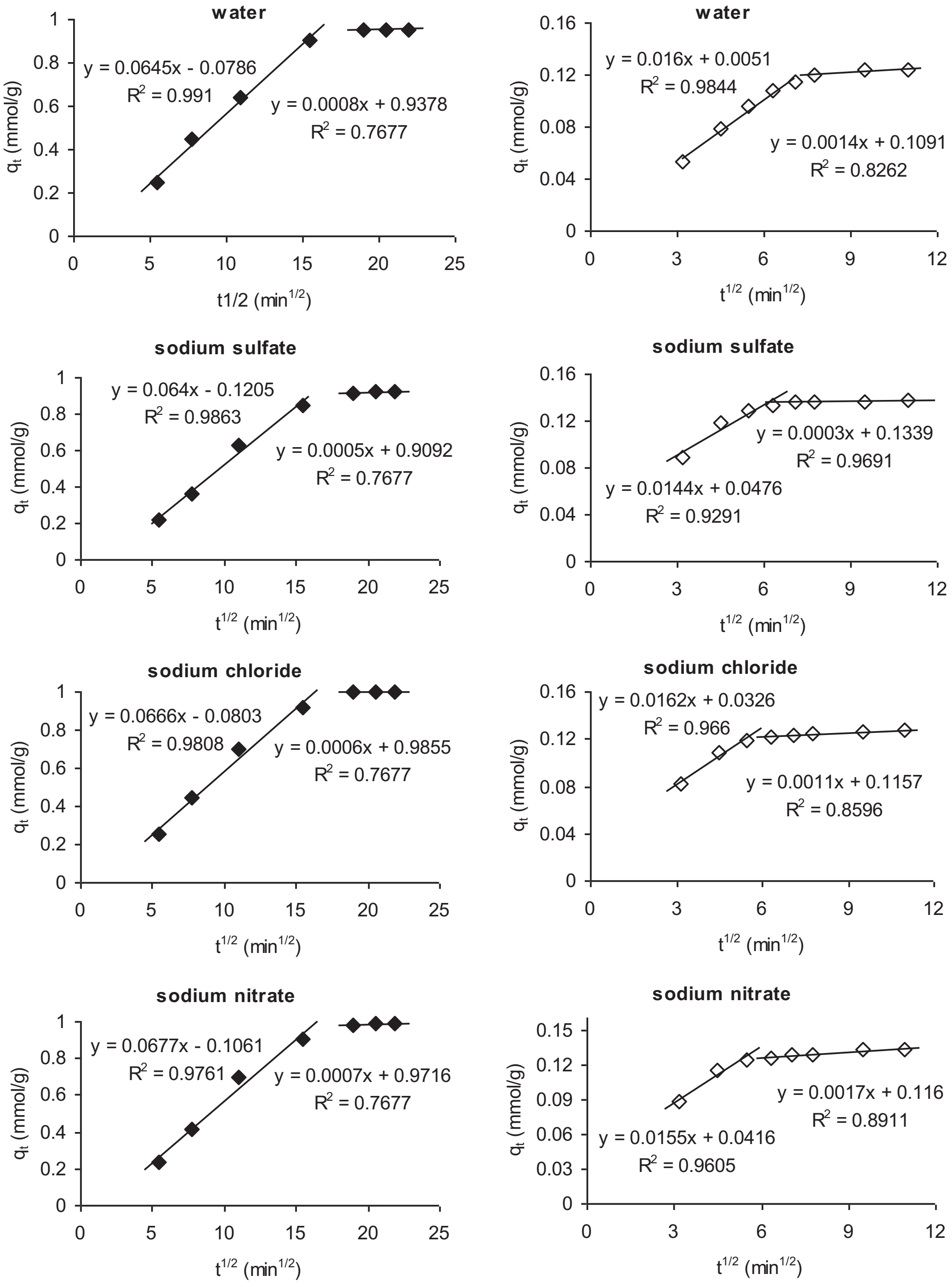

Fig. 3. Intraparticle diffusion model for the adsorption of 4-chlorophenol on activated carbon (left column) and MWCNTs (right column).

intraparticle diffusion is the rate-limiting step [29,30]. As shown in Fig. 3, none of the lines passed through the origin. This indicates that the intraparticle diffusion was not the only rate-controlling step. Moreover, the plots were not linear over the whole time range, suggesting that more than one process affected the adsorption. The slopes of the first part of the plot for the activated carbon are about four times greater than for the carbon nanotubes, however the slopes are practically the same for water and all electrolytes.

3.3. Adsorption equilibrium study

Fig. 4 shows the adsorption isotherms of 4-chlorophenol on the activated carbon from water and 0.05 and 0.1 mol/L sodium chloride, sodium nitrate and

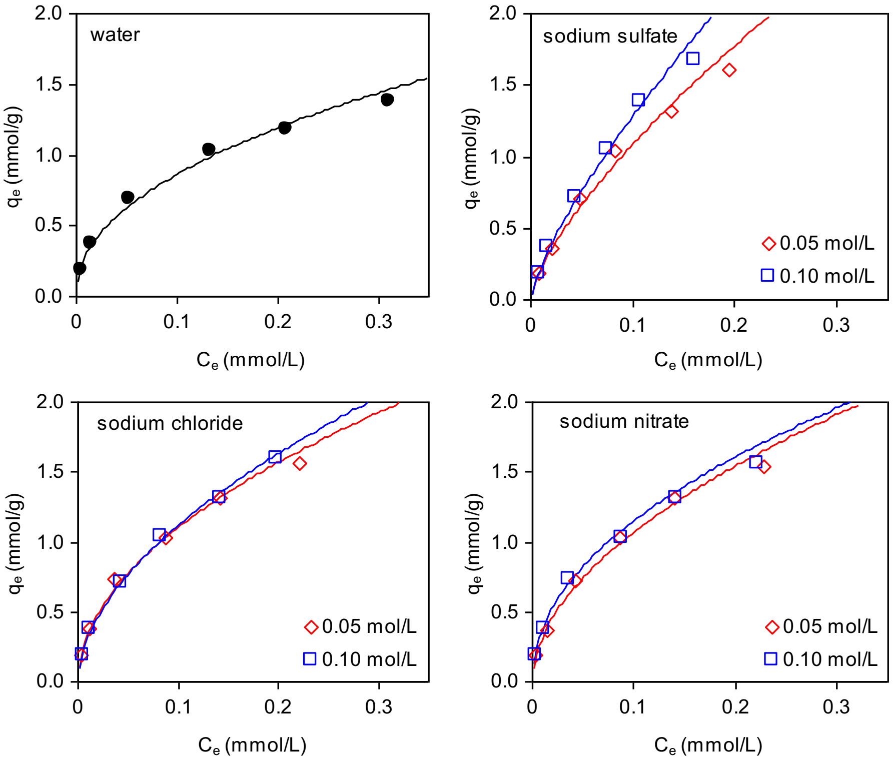

Fig. 4. Adsorption isotherms of 4-chlorophenol onto activated carbon from water and electrolyte solutions.

sodium sulfate solutions. The experimental isotherms of the 4-CP on the MWCNTs from water and 0.05 mol/L electrolytes are described in Fig. 5.

To test the fitting of the experimental data, the Langmuir and Freundlich isotherm models were used. The Langmuir isotherm equation is applied to the monolayer adsorption while the Freundlich isotherm is employed for the sorption surfaces with non-uniform energy distribution. The linear form of the Freundlich equation is as follows [31]:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{6}$$

where K_F [(mmol/g) (L/mmol)$^{1/n}$] and n are Freundlich equation constants which are calculated from the intercept and slope of ln q_e vs. ln C_e plot.

The linear form of the Langmuir isotherm is described by the formula [32]:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m} \tag{7}$$

where q_m [mmol/g] is the maximum adsorption capacity and b [L/mmol] is the constant related to the free energy of adsorption. In order to obtain the rate constants and correlation coefficients, the straight line plots of C_e/q_e vs. C_e have been tested.

The Freundlich and Langmuir adsorption isotherm model parameters and the correlation coefficients R^2 for the adsorption of the 4-chlorophenol onto the granular activated carbon and the MWCNTs from the water and electrolytes are presented in Table 2.

The R^2 values show that the equilibrium data obtained for both of the adsorbents were well represented by the Freundlich isotherm compared to the Langmuir equation. The K_F values obtained for the activated carbon are higher for the electrolyte containing solutions than for water. The equilibrium adsorption amount increased with the increasing salt content. The 4-chlorophenol was adsorbed on the activated carbon preferably from the sodium sulfate solution. The adsorption in the presence of sodium chloride and sodium nitrate was at the same level. That suggests that the adsorption does not affect the chemical composition of the solution (e.g. the type of ion present in the solution), but rather the ionic strength of the solution. The ionic strength of the NaCl and $NaNO_3$ solutions is identical, while the Na_2SO_4 is three times greater. The adsorption from the 0.0167 mol/L solution of Na_2SO_4 is practically the same as from the

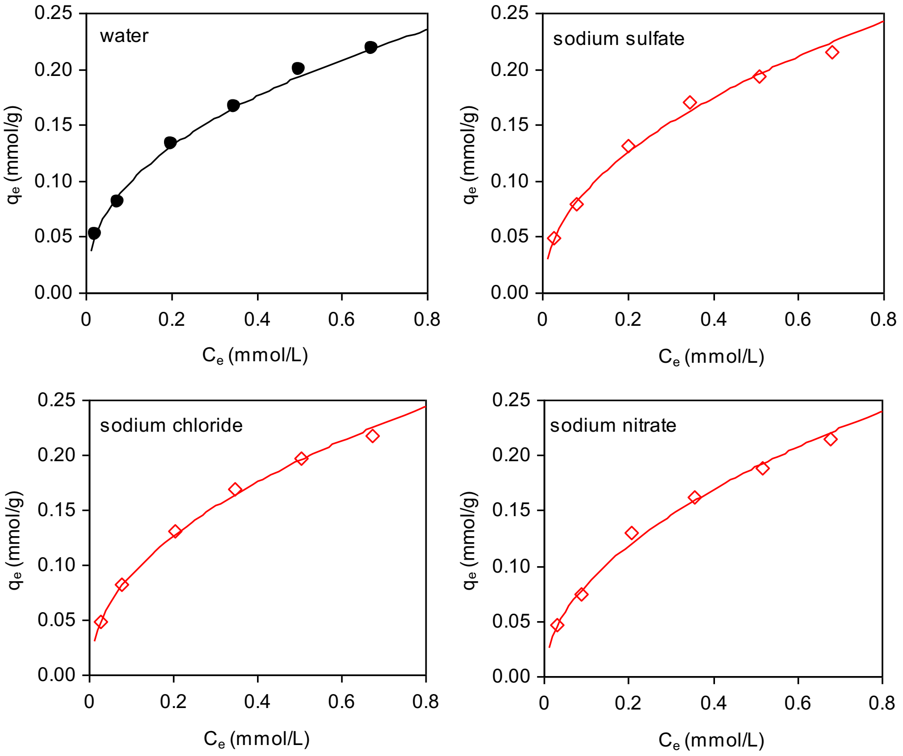

Fig. 5. Adsorption isotherms of 4-chlorophenol onto multi-walled carbon nanotubes from water and 0.05 mol/L electrolyte solutions.

Table 2
Freundlich and Langmuir adsorption isotherm model parameters and correlation coefficients R^2 for adsorption of 4-chlorophenol onto activated carbon and multi-walled carbon nanotubes from water and electrolyte solutions

	Freundlich model			Langmuir model		
Electrolyte	K_F	n	R^2	b	q_m	R^2
F-400 activated carbon						
H_2O	2.518	2.153	0.995	19.540	1.537	0.984
Na_2SO_4 0.0167 mol/L	3.513	1.624	0.998	8.356	2.088	0.917
Na_2SO_4 0.05 mol/L	5.570	1.408	0.994	7.876	2.612	0.990
Na_2SO_4 0.1 mol/L	7.306	1.323	0.990	3.095	7.310	0.976
NaCl 0.05 mol/L	3.551	1.969	0.994	21.315	1.804	0.975
NaCl 0.1 mol/L	3.934	1.830	0.998	17.265	1.944	0.960
$NaNO_3$ 0.05 mol/L	3.498	2.073	0.991	26.654	1.753	0.983
$NaNO_3$ 0.1 mol/L	3.610	1.895	0.995	14.245	1.895	0.985
MWCNTs						
H_2O	0.259	2.364	0.994	6.840	0.256	0.973
Na_2SO_4 0.05 mol/L	0.270	2.105	0.996	5.958	0.261	0.986
NaCl 0.05 mol/L	0.271	2.122	0.998	6.014	0.262	0.984
$NaNO_3$ 0.05 mol/L	0.269	1.974	0.995	4.930	0.268	0.980

0.05 mol/L solutions of sodium chloride and sodium nitrate, which is caused by the same ionic strength of the solutions (0.05 mol/L). The effect of the ionic strength on the adsorption of the 4-CP on the activated carbon is depicted in Fig. 6(a). Similar observations have been made for other organic compounds [27,33–41]. A review of the literature shows that, in general, increasing the salt concentration yields an increase in the capacity. The electrolytes were reported to increase the adsorption of the 2-ethyl-1-hexanol [27], natural organic matter [33,34], reactive black dyes [35,36], bisphenol A [37], phenol [38] on the activated carbon, 4-nitrophenol [39], pesticides diuron, and amitrole [40] on the carbon fibers and 2,4-dinitrophenol on the data seeds [41]. The presence of salt in the solution does not affect the adsorption of the 2,4-dinitrophenol [41] and the sodium dodecylbenzenesulfonate [42]. On the other hand, Arafat et al. [22] investigated the effect of salts on the adsorption of phenol, toluene, and benzene, and they observed the different trends of the salt effect for each compound. They concluded that the capacity as a function of the salt concentration may go through a maximum, as for phenol, or a minimum, as for benzene.

The presence of salts in the solution can modify the strength of the adsorbent-adsorbate electrostatic interactions. When the electrostatic interaction between the adsorbent surface and the adsorbate is attractive, and the surface concentration is sufficiently low, an increase in the ionic strength will decrease the adsorption. On the other hand, when the electrostatic interaction is repulsive, or the surface concentration is sufficiently high, the adsorption will increase with the increased ionic strength [21,33]. The adsorption of organic compounds on the activated carbon in the presence of salt may also increase due to the "salting-out" effect, which causes a reduction in the solubility of the adsorbate [37].

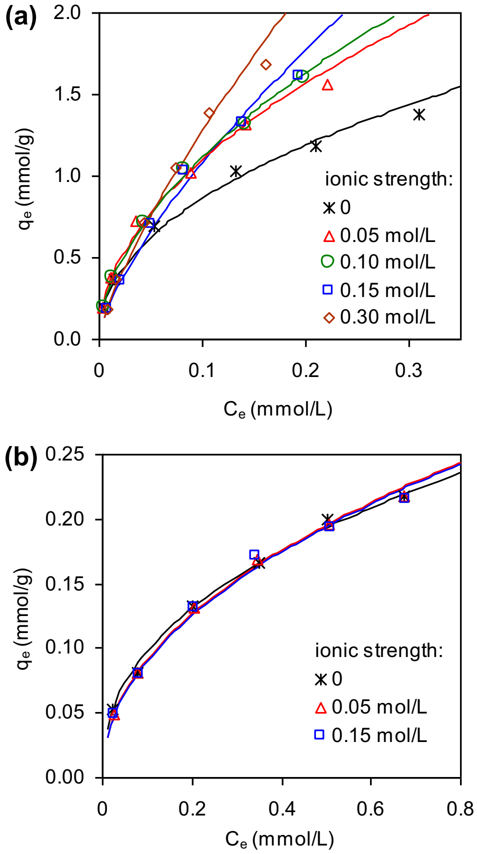

Fig. 6. Effect of ionic strength on the adsorption of 4-chlorophenol onto activated carbon (a) and MWCNTs (b).

A significant effect of the electrolytes on the carbon nanotubes adsorption characteristics has not been observed [43–45]. These reports are consistent with the results obtained in this experiment. The Freundlich constant K_F calculated for water, sodium sulfate, sodium chloride, and sodium nitrate were 0.259, 0.270, 0.271, and 0.269 (mmol/g) (L/mmol)$^{1/n}$, respectively. The adsorption of the 4-CP on the MWCNTs from solutions of different ionic strength is presented in Fig. 6(b). Zhang et al. [45] explain this phenomenon that an ionic strength may alter the aggregation state of the carbon nanotubes. The counter-ions added to the solution penetrate into the diffuse double layer surrounding the carbon nanotube particles, making them denser, and in consequence, thinner and smaller in volume. The repulsive energy between the CNTs would be reduced, and the aggregates of the nanotubes would be more compact which is detrimental to the adsorption of the organic compounds.

4. Conclusions

This study investigated the behavior and mechanism of the adsorption of 4-chlorophenol from deionized water and three electrolyte solutions onto activated carbon and multi-walled carbon nanotubes. The kinetic analysis indicated that the adsorption kinetics follows the pseudo-second order equation. The results show that the salt medium increased the adsorption kinetic of the 4-CP on the carbon nanotubes, but had no major effect on the 4-chlorophenol adsorption on the activated carbon. A study was also conducted on the effect of the presence of salts, dissolved in the medium, on the adsorption equilibrium. To describe the adsorption isotherms, the Langmuir and Freundlich equations were applied. The Freundlich model was better than the Langmuir model in describing the adsorption equilibrium data for both adsorbents. It was observed that altering the solution composition did not significantly affect the adsorption capacity of the carbon nanotubes. The regression constants were statistically similar between the salt and the water medium. On the other hand, it was found that electrolytes significantly enhanced the capacity of adsorption of the activated carbon. The removal of the 4-chlorophenol increased with the increase in the ionic strength of the solution.

The novelty of this research is the first comprehensive examination of the impact of salts on the adsorption of 4-chlorophenol on activated carbon and, above all, on the multi-walled nanotubes. This knowledge is essential for optimizing the use of activated carbon and/or carbon nanotubes in water treatment. The impact of the electrolyte on the adsorption kinetics of 4-chlorophenol on the MWCNTs has been described for the first time. To the best of our knowledge, the effect of salt on the adsorption kinetics of any other organic contaminants has not been described so far. The results showed that the adsorption of organic compounds is determined not by the chemical composition of the solution (the type of electrolyte) but by its ionic strength. The effect of salt on the adsorption of organics has been found to be more complex than previously reported and requires further studies.

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