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Oxygen Functionalized and Pristine Carbon Nanotubes Efficiency for Adsorption of Methyl Orange Dye

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Authors' contribution

The sole author designed, analysed, interpreted and prepared the manuscript.

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ABSTRACT

Pristine and oxygen functionalized multi-walled carbon nanotubes were used as adsorbent for removal of Methyl Orange (MO) dye. The adsorption was carried out under different pH values. The analysis of results indicated that the adsorption characteristics of the MO dye by pristine carbon nanotubes (P-CNTs) and oxygen functionalized carbon nanotubes (O2-CNTs) are highly influenced by the pH of the medium. The study report that the best pH medium of solution for the adsorption of MO on both CNTs was acid medium. The point of zero charge (pzc) of O2-CNTs and P-CNTs were determined as function of pH. The pzc of P-CNTs and O2-CNTs are found to be 4.7 and 3.9, respectively. The adsorption data have been analyzed using Langmuir and Freundlich. Fitting the equilibrium adsorption data by Langmuir and Freundlich models shows that experimental data well explained by the Langmuir equation.

Keywords: Functionalized carbon nanotubes; dye removal; organic pollutants; wastewater treatment.

1. INTRODUCTION

Effluents from industries contain numerous organic pollutants and discharging them has become a significant issue of environmental concern due to their toxicity and carcinogenicity. Polycyclic aromatic hydrocarbons, e.g. oils, plasticizer, phenols, pesticides, dyes and

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fertilizers, are one of the main groups of these organic pollutants. These organic contaminants may remain in the environment for long period of time because of their high degree of aromaticity and conjugation [1]. Dyes are widely used and extensively discharged by industries and considered the main type of pollutants and posing hazard to living organisms [2,3]. They have many applications in different fields such as dyeing and printing on fibers and fabrics of all kinds, food coloring, as well as for medicinal and cosmetic uses [4].

Several methods in wastewater treatment technology have been performed to degrade or remove organic contaminants from aqueous solutions including electrochemical treatment, ozonation, photo degradation, adsorption, ion exchange and evaporative recovery [5-14]. Adsorption method has been proved by researches to be an effective and one of the extensively used approaches for the removal of organic and inorganic pollutant from wastewater [15-17]. Different adsorbents have been developed and modified for this purpose. Among these absorbents, activated carbon is the most commonly due to its excellent capacity of adsorption for organic pollutants [18]. Recently, nano-structured materials have been extensively used to remove toxic substances. CNT scan served as excellent adsorbents and widely studied carbon nanomaterials due to their large specific surface area and hollow and layered structure [19-21].

CNTs have received great interest in water treatment as a new kind of adsorbent because of their small size, well-developed mesopores, large accessible specific surface area, high aspect ratio, as well as easily modified surfaces. Large surface area and high porosity provide enough adsorption sites for harmful contaminations present in wastewater. High aspect ratio of CNTs makes it a possible candidate for water purification. In recent years, great efforts have been made to remove various organic pollutants and metal ions in wastewater by CNTs [22]. To further improve their adsorption performance, various CNTs composites have been synthesized and widely used to remove dye pollutions from wastewater. However, the adsorption capabilities are not entirely satisfactory and it is still a challenge to explore novel CNTs composites adsorbents with high adsorption capacity, short adsorption time as well as low cost for practical utilization.

The functionalization of CNTs surfaces was envisioned by many researchers in order to enhance their chemical properties [23]. The surface modification of the CNTs can be performed by covalent and noncovalent (van der Waals bonds), or by creating defect, sidewall functionalization and exohedral and endohedral functionalization [24]. Generally, during CNTs modification, chemical functionalities such as OH, COOH, NH_2 groups are attached to the sp^2 carbon framework. This kind of functionalization promotes the CNTs' dispersion in a wide variety of solvents and polymers, enabling the use of nanotubes in a wide range of applications [25]. The most prominent interactions are between aliphatic C-H donors and aromatic pi-acceptors and interactions between aromatic C-H donors and aromatic pi-acceptors. In the case of covalent modification, the desired functional group is attached to the sidewall or to the ends of the carbon nanotubes [26], while in the case of non-covalent modifications, van der Waals force and π-π interactions take place. It is worth mentioning that the non-covalent tuning of CNTs is preferable for the enhancement of the interfacial properties of the CNTs as it avoids the destruction of CNT structure [27].

The MO is a representative azo dye which is widely used for colorization in paper, textile and chemical industries. The MO dye and its degradation products threaten to human health and aquatic life safety Due to their toxicity and carcinogenicity [28]. Since CNT shave emerged as a good adsorbent for dye removal from wastewater, therefore, the main aim of this study is to use to different type of multiwalled CNTs pristine CNTs (P-CNTs) and oxygen functionalized CNTs (O-CNTs) for MO dye removal and investigate the ability of these two CNTs as adsorbents. The effect of pH and point of zero charge for each CNTs on adsorption were studied. We also interested to study the two well-known isotherms, Langmuir and Freundlich on adsorption process.

2. MATERIALS AND METHODS

2.1 Materials

The MO dye was purchased from Labpak Chemicals Ltd, UK, and used as received to prepare solutions that used in this paper. The characteristics of MO are shown in Table 1. The methyl orange concentrations used in this study were 10, 20, 30 and 40 mg/L. The stock solution of MO (1000 mg/L) was covered and stored in a

dark place. Deionised water was used to prepare all solutions used in this research. Multi-walled carbon nanotubes functionalised by oxygen (O2-CNTs) and pristine multi-walled carbon nanotubes (P-CNTs) were purchased from Haydale Ltd., UK and used as catalyst in this study. The characteristics of both CNTs are shown in Table 2. Nitric acid (HNO_3) and sodium hydroxide (NaOH) were purchased from Fisher-Scientific, UK. Various molarities of HNO_3 and NaOH were used to adjust the pH value of solutions between 2 and 10 using pH meter.

2.2 Adsorption Isotherm

P-CNTs and O2-CNTs each with 10 mg were used as adsorbents and four solutions of MO with initial concentration 10, 20, 30 and 40 mg/L were used. This involved using 200 mL of MO in a reactor and placed on a stirrer for better mixing for 60 min. Samples were taken at a specific schedule, 2 mL of sample, using a glass syringe with 10 mL. For separating adsorbents from the taking sample before analysis, a centrifuge was used. The samples were analysed using spectrophotometer with maximum absorbance 508 nm.

The following equation was used to calculate the percentage of MO removal:

$$\% \, Adsorption = \frac{C_o - C_e}{C_o} * 100 \, \% \quad (1)$$

where C_o is the concentration of MO dye at initial and C_e is the concentration at equilibrium.

The amount of dye adsorbed per unit mass of adsorbent was calculated as follows:

$$q_e = \frac{C_o - C_e}{\mathrm{m}} * \mathrm{V} \quad (2)$$

Langmuir and Freundlich models were applied for describing the adsorption of MO on P-CNTs and O2-CNTs. The Langmuir model can be written in a linear form as in below equation In order to obtain Q_m and b values [29,30]:

$$\frac{C_e}{q_e} = \frac{1}{bQ_m} + \frac{C_e}{Q_m} \quad (3)$$

Where C_e is the equilibrium concentration of sorbent (mg/L), Q_m is the maximum sorption capacity (mg/g), q_e is the mass of MO adsorbed per unit mass of adsorbent at equilibrium (mg/g), b is the Langmuir adsorption constant (L/mg).

On the other hand, the Freundlich isotherm can be obtained by the equation 3. For non-ideal adsorption and when the surface is heterogeneous the Freunlidh isotherm is used [31]. Where n_f is the Freundlich coefficient and K_f is the Freundlich constant. A linear equation is used to determine K_f and n_f:

$$lnq_e = lnK_f + \frac{1}{n_f} lnC_e \quad (4)$$

2.3 Characterisation of Adsorbents

Identifying point of zero charge (pzc) in aqueous media is very important for adsorption and desorption of contaminants. The particle size of CNTs was measured using a High Performance ParticleSizer 3.3 (Malvern Instrument, UK) at room temperature, pH 5.13 and a 30-min presonication. The zeta potential of CNTs was measured by a Malvern Zetasizer 2000 at room temperature. Five CNT solutions at a concentration of 10 mg/L and pH values of approximately 2, 3, 4, 5, 6, 7 and 8 were used for measuring the CNTs point of zero charge (pzc). The pH values of these solutions were adjusted using 0.1M NaOH or 0.1M HNO_3.

Table 1. Methyl orange dye characteristics

Property	Value
C.I. index	C.I. Acid Orange 52
Molecular Formula	$C_{14}H_{14}N_3NaO_3S$
Molar Mass	327.34 g/mole
IUPAC Name	Sodium 4-([4-(dimethylamino) phenyl] diazenyl) benzenesulfonate
Colour	Blue
λ_{max}	508 nm at pH< 4 464 nm at pH ≥ 4
pKa	3.47

Table 2. Properties of the P-CNTs and O-CNTs used in this research work

P-CNTs		O-CNTs	
Property	**Value**	**Property**	**Value**
Carbon Content (%)	96	Carbon Content (%)	95
Outer Diameter (nm)	~ 13-16	Outer Diameter (nm)	~ 13-16
Length (μm)	~ 1	Length (μm)	~ 1
Aspect ratio (length/diameter)	~ 69	Aspect ratio (length/diameter)	~ 72
Bulk Density (g/cm^3)	~ 0.19	Bulk Density (g/cm^3)	~ 0.21
BET surface area (m^2/g)	~ 250	BET surface area (m^2/g)	~ 258
Ash	~ 1.5	Oxygen content	3.5-4

3. RESULTS AND DISCUSSION

3.1 Effect of pH

The oxidation states and hence the properties of CNTs are mainly rely on the pH of the solution [32]. The surface charge of the adsorbents and the degree of ionization of dye are influenced by the pH of the solution due to the protonation and deprotonation of the functional groups [33]. The adsorption of other ions is affected by the pH of the solution because the hydrogen ion (H^+) and hydroxyl ion (OH^-) are strongly adsorbed to the surface of adsorbent. The effect of pH was studied and experiments were carried out at different pH levels (2 to 10) and the results for MO in the presence of different adsorbents are given in Fig. 1. The figure shows the increasing of adsorption efficiency of P-CNTs and O2-CNTs as pH increased from 2 to 4 because the surface of both adsorbents may contain a large active sites number. The concentration of MO used for the determination of pH effect was 10 mg/L. The dye adsorption on O2-CNTs and P-CNTs was decreased from pH 4 to pH 10. In this range of pH, the surface charge of P-CNTs and O2-CNTs is negative and more [A-] is formed, which also has a negative charge, when MO dissociate; thus the adsorption of MO decreased. Fig. 2 shows the speciation curve of MO and the formation of [A-] in the pH range of 4 and 10.

3.2 Point of Zero Charge for the Absorbents

The point of zero charge (pzc) of O2-CNTs and P-CNTs were determined by Zetasizer 2000. Fig. 3 shows the zeta potential values as a function of pH. The solutions of different pH were obtained by adding 0.1 M NaOH and 0.1 M HNO_3. In high acidic medium, pH< 4, O2-CNTs present positive zeta potential. At pH> 4, the zeta potential became negative. It was measured that the pzc of O2-CNTs is assumed to be 3.9. However, pzc of P-CNTs is assumed to be 4.7. The zeta potential values were calculated using Henry's equation:

$$U_E = \frac{2\,\varepsilon_o\,\varepsilon_r\,\zeta}{3\,\eta} f(ka) \quad (5)$$

3.3 Adsorption Isotherm Models

Various concentration range (10, 20, 30 and 40 mg/L was used for investigating the effect of MO initial concentration on the adsorption process. However, other parameters like pH, volume and adsorbent dose were kept constant. Fig. 4 shows the effect of MO initial concentration on removal percentage. It was found that the removal percentage of MO decreased as the initial MO concentration increased. The adsorption capacity of O2-CNTs and P-CNTs was increased from 129.69 to 171.48 mg/g and 127.85 to 159.32, respectively, as the initial MO concentration increased from 10 to 40 mg/L (see Fig. 5). This can be explained that the active sites number remain constant, while the initial concentration of MO increases.

The experimental results, as shown in Figs. 6 and 7 for O2-CNTs and P-CNTs respectively, were more fitted to the Langmuir isotherm than Freundlich isotherm. This suggest that, for the present system, the Freundlich isotherm is a less appropriate. The parameters values of both models for O2-CNTs and P-CNTs are shown in Table 3. Freundlich constants, K_F and $1/n_f$, indicate the adsorption capacity and adsorption intensity, respectively. Higher the value of 1/nf, the higher will be the affinity between the adsorbate and the adsorbent, and the heterogeneity of the adsorbent sites. The $1/n_f$ value indicates the relative distribution of energy sites and depends on the nature and strength of the adsorption process. The $1/n_f$ value for O2-CNTs and P-CNTs was found to be 0.192 and 0.188, respectively, and these refer to adsorption process is favorable. The adsorption process is

favorable when the value of n_f is in the range between 1–10 and this is confirmed for both type of studied carbon nanotubes. The K_F value can be taken as a relative indicator of the adsorption capacity and the magnitude of K_F also showed the higher uptake of MO dye at higher temperature and endothermic nature of adsorption process. The b value between 0 and 1 indicates favorable adsorption of MO dye onto CNTs used. The comparison of the values in Table 3 for understudy isotherm models shows that the R^2 values for Langmuir model are closer to unity that shows its applicability for explanation of MO dye adsorption on this adsorbent.

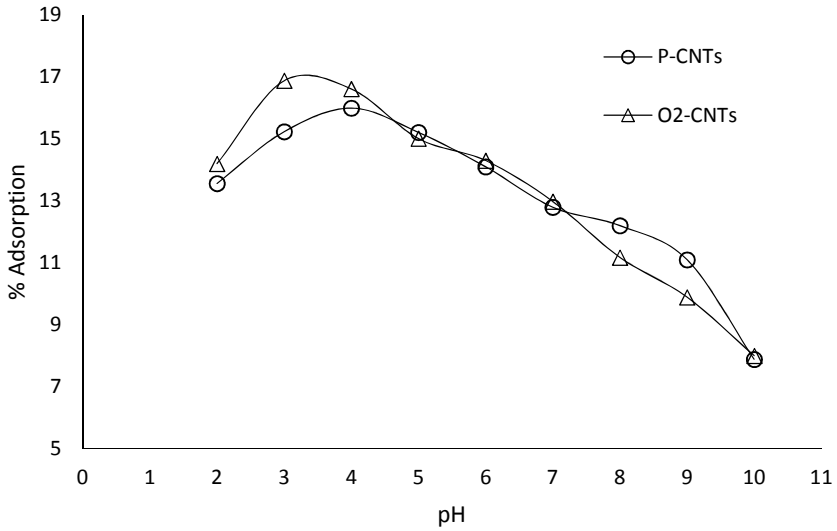

Fig. 1. Effect of pH on adsorption of MO on P-CNTs and O2-CNTs, time= 40 min, C_o= 10 mg/L

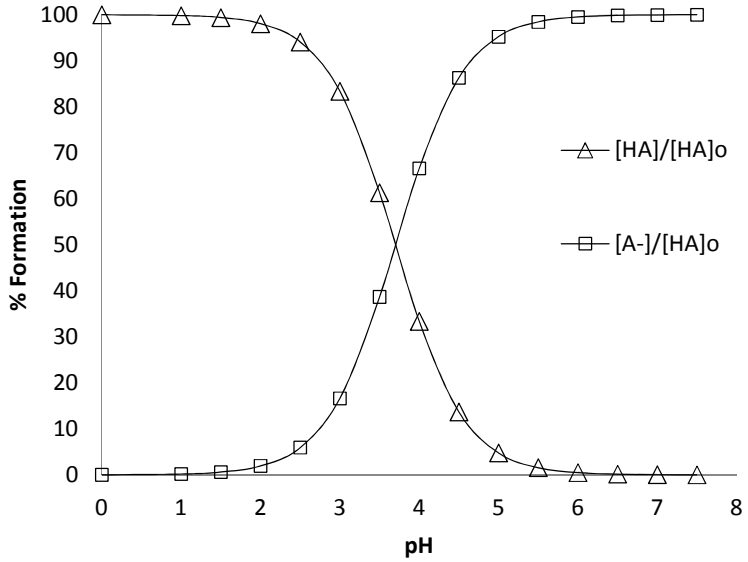

Fig. 2. The speciation curve of methyl orange

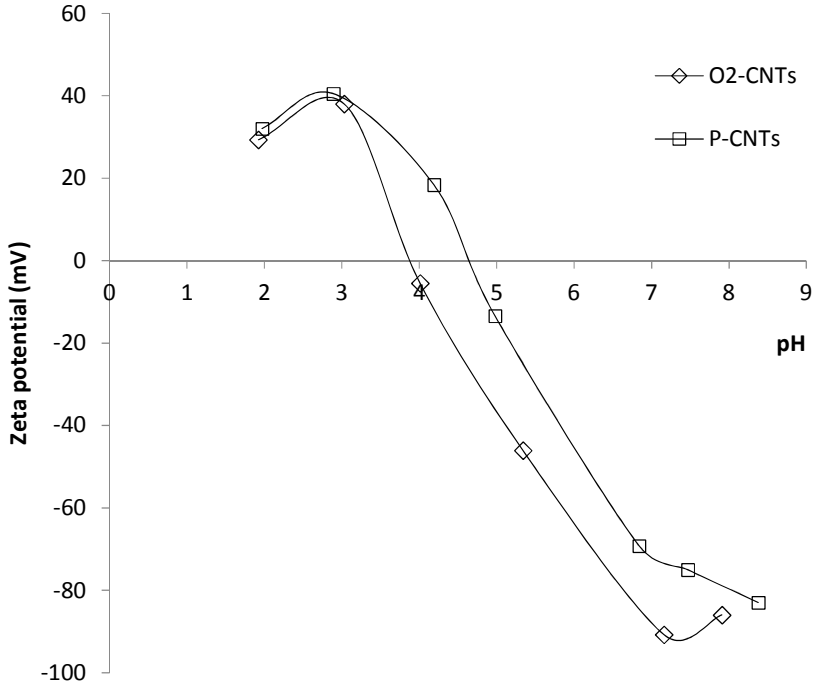

Fig. 3. Variation of zeta potential for O2-CNTs and P-CNTs as a function of pH and determination of pzc

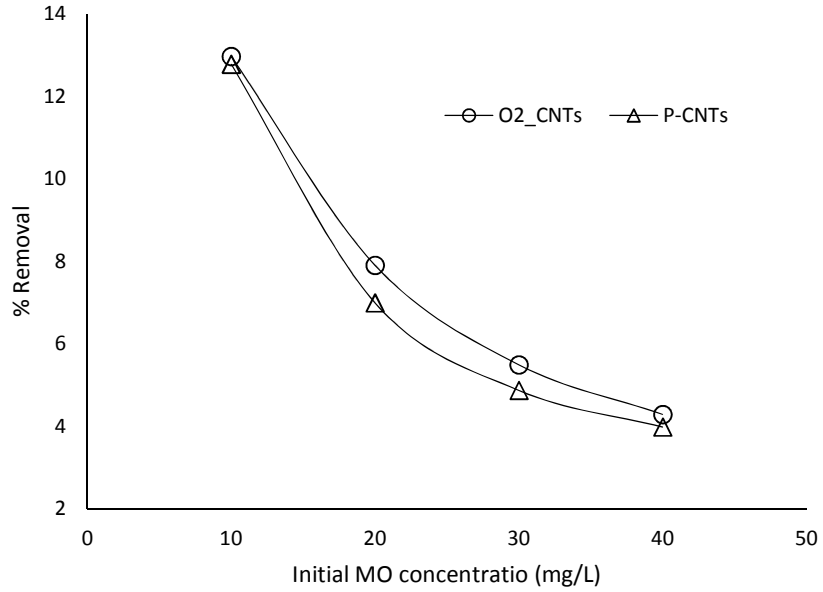

Fig. 4. The effect of initial concentration of MO on removal percentage at time = 40 min

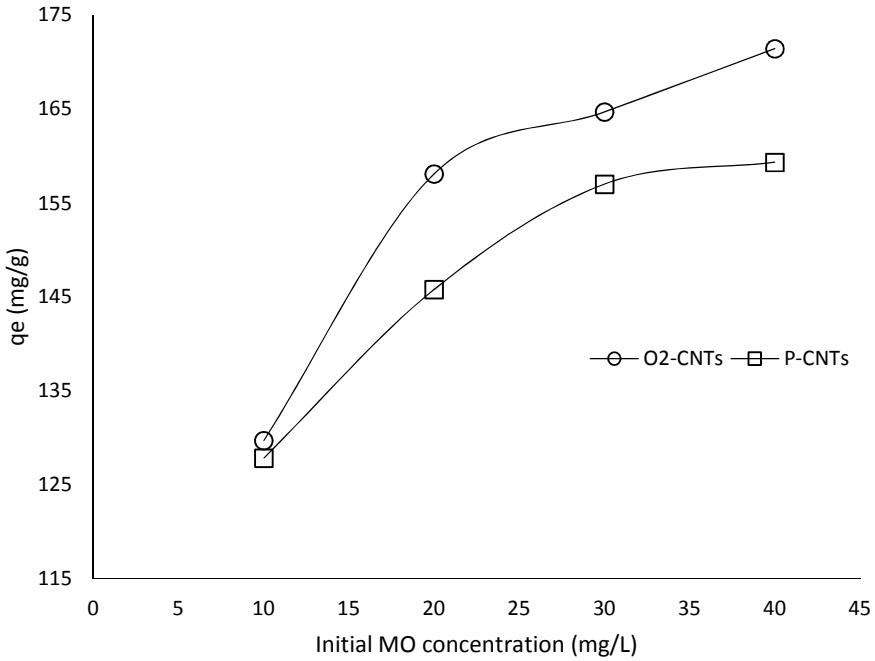

Fig. 5. The effect of initial concentration of MO on q_e at time = 40 min

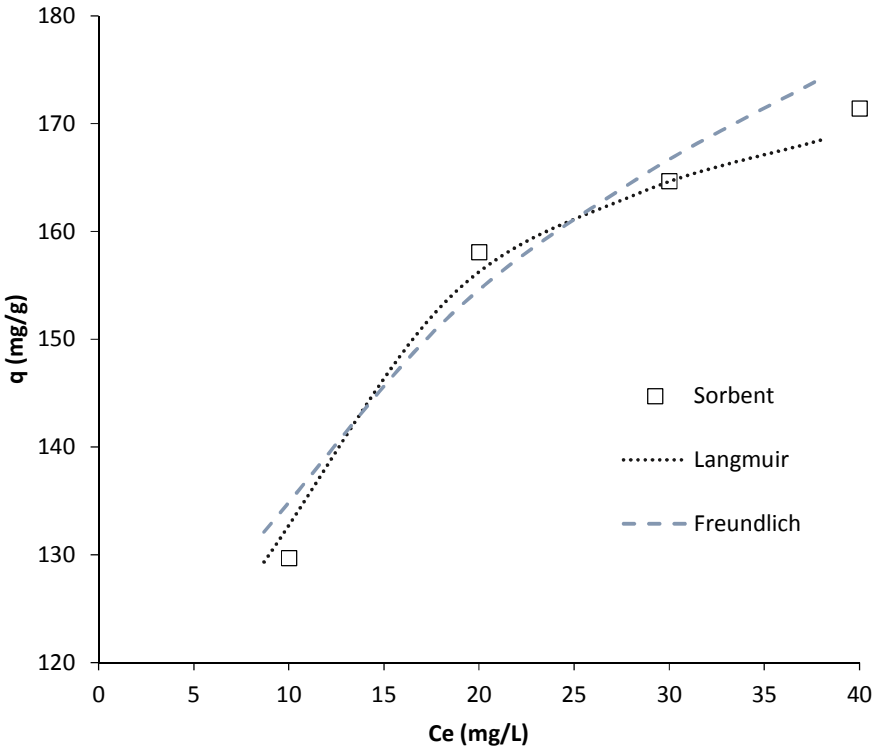

Fig. 6. Langmuir and Freundlich isotherms in the presence of O2-CNTs

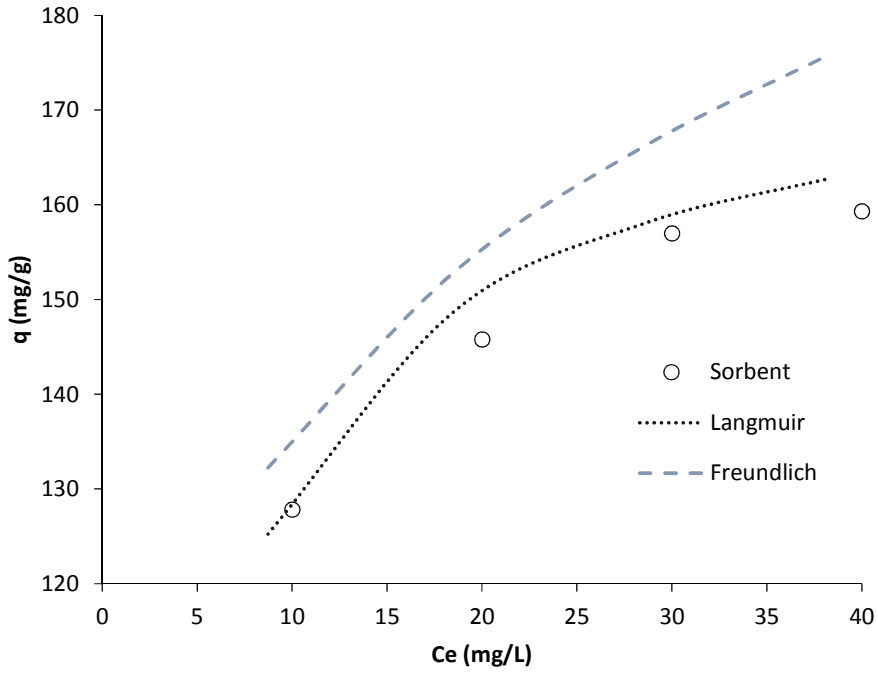

Fig. 7. Langmuir and Freundlich isotherms in the presence of P-CNTs

Table 3. Isotherm parameters in the presence of O2-CNTs and P-CNTs

Models	O2-CNTs		P-CNTs	
	Langmuir	**Freundlich**	**Langmuir**	**Freundlich**
Isotherm parameters	Q_m= 185.18 b = 0.266	K_f= 87.16 n_f= 5.19	Q_m= 178.57 b = 0.269	K_f= 88.03 n_f= 5.33
R^2	0.9997	0.9531	0.9942	0.981

4. CONCLUSION

The efficiency of oxygen functionalized and pristine multi-walled carbon nanotubes were investigated for adsorption of MO dye. The analysis of results indicate that the adsorption process was highly affected by the pH of the solution. The adsorption efficiency of MO dye on P-CNTs and O2-CNTs increased as pH goes from 2 to 4 and decreased during the pH 4 and pH 12. The point of zero charge (pzc) of O2-CNTs and P-CNTs were determined as function of pH and were recorded 4.7 and 3.9, respectively. Comparing O2-CNTs to CNTs, it was found that at the same dose, O2-CNTs enhanced the decolorization rates more than P-CNTs. Langmuir and Freundlich isotherms were studied to analyze the removal of MO dye on both CNTs. The Langmuir adsorption model gives very satisfactory fitting to the adsorption isotherms.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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