



High Efficiency of Multiwalled Carbon Nanotubes Filters for Benzene Removal from Aqueous Solutions: Quantitative Analysis using Raman Spectroscopy

Emad M Elsehly^{1,3*}, Chechenin NG^{1,2}, Makunin AV¹, Motaweh HA³, Bukunov KA^{1,2} and Leksina EG^{1*}

Abstract

The environmental application of multiwalled carbon nanotubes (MWCNTs) as a new type of filters to remove benzene from aqueous solutions was investigated. The surface functionalization of MWCNTs enhances their performance for this application. Arrays of MWCNTs were synthesized via chemical vapor deposition (CVD) by spray-pyrolysis method. An aliquot of these MWCNTs array was oxidized by concentrated nitric acid. The Fourier transform infrared analysis showed that the peaks corresponded to the hydroxyl and carboxylic acid groups in oxidized MWCNTs (O-MWCNTs) are more intense than that in raw MWCNTs (R-MWCNTs). The images of scanning electron microscopy indicated that the O-MWCNTs have a lower degree of entanglement and decreased nanotubes diameters. Energy dispersive X-ray spectroscopy showed high oxygen content for O-MWCNTs compared to R-MWCNTs. In order to estimate the removal efficiency of MWCNTs filters, Raman spectroscopy as a quantitative technique to make concentration measurements of benzene in water solutions was conducted and used to estimate the purification efficiency. The removal efficiency of benzene by O-MWCNTs was found to reach 99% for concentration of 500 ppm, indicating the existence of specific π - π electronic interactions between benzene molecules and the surface of O-MWCNTs. Functionalized MWCNTs possess good potential applications to water and wastewater treatment, maintaining a high quality of water, and could be used for cleaning up environmental pollution.

Keywords

Benzene removal; Aqueous solutions; MWCNTs; Raman spectrometry; Functionalization; Adsorption

Introduction

Water pollution phenomena have rapidly become more and more frequent and acute in recent decades. Benzene is one of the most typical groundwater petroleum contaminants which have toxic and carcinogenic properties [1,2]. Benzene pollutes groundwater

through the leakage from underground storage, pipelines, improper waste disposal, in advertent spills and landfills [3]. There are concerns about adverse health effects of benzene including cancer, respiratory problems and disruption of liver and kidney [4]. Benzene can be detected by odor at levels of 2.5 ppm or greater in air, and in water it is detected by taste/odor at levels of 0.5-2.0 ppm [5]. Therefore, removal of benzene from water and wastewater is essential and has been widely studied. Several processes have been successfully applied, including bioremediation, volatilization (air stripping), chemical oxidation, as well as adsorption [6-8]. The utilization of these removal processes on a large scale presents certain advantages and disadvantages as far as applicability, site dependence, efficiency and cost parameters are concerned.

Nanomaterials are a new class of functional materials, which has attracted much attention due to its high adsorption capacity.

Carbon nanotubes (CNTs) have attracted great interest because of their unique chemical structure and intriguing physical properties [9]. Both single-walled and multiwalled CNTs have numerous potential applications in basic science and nanotechnology, such as nanoscale electronic devices, hydrogen storage devices, nanocomposite materials, and wastewater treatment filters [10-12]. MWCNTs are becoming more and more attractive from the practical standpoint [13]. Their relatively low production costs and availability in large quantities are two important advantages over SWCNTs. The adsorption mechanism of benzene on CNTs is mainly attributed to the π - π electron donor-acceptor interaction between the aromatic ring of benzene and the surface carboxylic groups of CNTs [14]. It has been found that CNTs are more effective for the removal of natural organic matter (NOM) than activated carbon [15]. However, an application of CNTs, especially in filtration of solutions, is lower than expected. A possible solution to this problem is to modify the surface of carbon nanotubes by oxidation [16]. Lu et al. used NaOCl-oxidized CNTs to enhance benzene and toluene adsorption, and showed superior adsorption performance compared with many types of carbon and silica adsorbents reported in the literature [17]. Advantages of using nitric acid in functionalization of MWCNT include high performance, short time to modification, high oxygen content represented in carboxylic groups. Also the amorphous carbon and catalyst particles introduced by the CVD preparation process were removed during the course of oxidation treatment [18].

The application of Raman spectroscopy for quantitative analysis has a number of advantages. Unlike infrared absorption spectroscopy, Raman spectra are easily acquired from aqueous solutions [19]. Another important advantage of Raman spectroscopy is flexibility of sampling. Raman spectroscopy is investigated as an alternative technique to determine benzene concentration in C_6H_6/H_2O solutions. As each molecular species has its own unique fingerprint, Raman spectroscopy can therefore be used for multi-species concentration measurements [20]. The OH-stretch in the water molecule gives rise to a broad peak at about 3400 cm^{-1} . The strength of the OH-peak is then proportional to the total number of water molecules. The modes of vibration for benzene molecule (C_6H_6) based upon the number of degree of freedom and the intensity of laser beam used. Using blue laser with wavelength 473 nm, the most vibration modes centered at 992 cm^{-1} and 3067 cm^{-1} for ring stretch and C-H stretch respectively. As

*Corresponding authors: Emad M Elsehly, Skobeltsyn Institute of Nuclear Physics, Lomonosov Moscow State University, Moscow, Leninskie Gory 1/2, 119234, Russian Federation, Tel: +79854904995; E-mail: elsehlyfigo@yahoo.com

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the ring stretch peak at 3067 cm^{-1} is combined with water spectrum and may be disappear at low concentrations of benzene solution, in our study we interest in the ring vibration peak, as its intensity is proportional to the number of benzene molecules. Follow these facts, the concentration of benzene in water can be estimated by the ratio of benzene ring peak and water OH- peak intensities.

An usage of functionalized MWCNT filters to increase the benzene removal efficiency and Raman technique to estimate this efficiency, to our knowledge, have not been reported in pervious publications. Our objectives were to evaluate the ability of functionalization process using nitric acid treatment in surface modification of MWCNTs and to demonstrate the utilization for adsorption of benzene from aqueous solutions. Also the significance and optimum condition (pH, MWCNT filter dose and initial benzene concentrations) for removing benzene were estimated. Raman spectroscopy was applied as a new quantitative technique to estimate the removal efficiency. Prepared functionalized MWCNTs filters possessed high efficiency for benzene removal from water and could be a promising kind of commercial filters for water purification.

Material and Methods

Synthesis and functionalization of MWCNTs

MWCNTs were synthesized by chemical vapour deposition (CVD) process as reported previously [21]. Briefly, The CVD reactor is $\phi 2.5\text{ cm} \times 100\text{ cm}$ long quartz tube placed in an automatically temperature controlled oven. A liquid solution of ferrocene in cyclohexane as well as the support nitrogen gas was supplied into the reactor. With the described method uniform MWCNTs arrays up to $20 \times 100\text{ mm}^2$ were systematically obtained on a Si-plate. MWCNTs were removed from the silicon substrate and crashed to obtain powder form. For acid treatment, 300 mg of the as-received MWCNTs powder was heated in air at 350°C for 20 min to remove amorphous carbon. After this thermal treatment, MWCNTs were dispersed in a flask containing 50 ml solution of 5 M HNO_3 (>69% purity, purchased from Sigma Aldrich), ultrasonicated at 80°C for 6 h in an ultrasonic bath (Fisher Scientific, FB15051, Germany) to obtain carboxyl functional groups. Upon cooling, the mixture was thoroughly washed with deionized water to remove traces of untreated acid until the pH value was 7, which signifies zero acidity. Then oxidized MWCNTs solution was filtered through a polycarbonate filter (pore size $0.2\ \mu\text{m}$) to separate from the liquid. Afterwards, the MWCNTs samples were dried at 120°C in a vacuum oven for 24 h.

Characterization of raw and oxidized MWCNTs

Physical morphology and structure analysis of raw and oxidized MWCNTs samples, prepared as described as pervious, was carried out using scanning electron microscope (SEM, TESCAN) with the Digimizer software which applied to detect the diameter of the MWCNTs. SEM energy dispersive X-ray spectrometry (EDS, Oxford Instruments Oxford Instruments INCA Energy 250/X-max 80 micro-analysis system) was used to verify the presence of different elements in the MWCNTs samples. Fourier-transform infrared (FTIR) spectra were recorded using IFS 66 V/S, Bruker FTIR spectrometer. For that the MWCNT samples were mixed with potassium bromide (KBr), pressed into pellets of 1 mm thickness and measured in the transmission mode.

Filter design and stock solution preparation

MWCNTs filters were prepared by sandwiching of compressed raw or functionalized MWCNTs between two pieces of glassy fiber membrane with a cotton layer as a filter substrate, to keep insuring

that not any MWCNTs passes through the filter, and putted into a syringe which act as the filtering system [12]. Figure 1 shows the steps for filter design for the adsorption process. A solution of benzene for 500, 1000, 1500, 1800 ppm (solubility limit) was prepared by dissolving appropriate amounts of benzene (98% purity, purchased from Sigma Aldrich) in distilled water (high purity produced in our lab, with resistivity not less than $18\ \text{M}\Omega\cdot\text{cm}$). The mixture was thoroughly dissolved using an ultrasonic bath for 60 min, and then, stirred continuously for another 60 min at 25°C . After shaking, the solution was put in ultrasonic bath again for 30 min. In order to avoid any losses due to volatilization during experimentation, benzene solution was filled completely in flasks and no headspace was left. To study the effect of pH on the filtration of benzene, pH of the solution was varied from 5 to 9, which is near the pH of tap water, by using 1 M solution of nitric acid and 1 M solution of sodium hydroxide.

Experimental condition and benzene concentration analysis

All of the batch experiments were performed in 50 ml syringe filtering system. In each experiment, various amounts of MWCNTs, from 0.2 to 0.6 g, were putted into the syringe and the 50 ml of benzene solution with initial concentrations of 500 ppm for low concentration and 1800 ppm for high one, were added. For each concentration, low or high, the efficiency was studied with different pH varying from 5 to 9 and mass of the filter, as indicated in Table 1. Before and after each experiment, benzene was determined using Raman spectroscopy, all of the experiments were repeated 3 times, and only the mean values were reported. A quartz glass was filled with benzene aqueous solution. A continuous wave frequency of blue laser radiating at 473 nm with a power of about 10 mW was used to illuminate the sample in Raman spectrometer (NT-MDT, INTEGRA SPECTRA). The Raman line-width (full width at half maximum intensity, FWHM) of benzene at 992 cm^{-1} assigned to the ring stretching vibration mode has been measured at a variety of concentrations in aqueous solution.

Table 1: Experimental parameters utilized in the study and their variation. Different concentrations of benzene solution are presented ranging from 500 to 1800 ppm. The pH of the solution is investigated for the range (5-9). These parameters are performed for various amounts of MWCNTs in the range (0.2-0.6) g.

Parameter	Variation		
	Low	Medium	High
1. Concentration of benzene in aqueous solution (ppm)	500	-	1800
2. pH of benzene aqueous solution	5	7	9
3. Raw and oxidized MWCNTs filter mass (in g per 50 ml of solution)	0.2	0.4	0.6

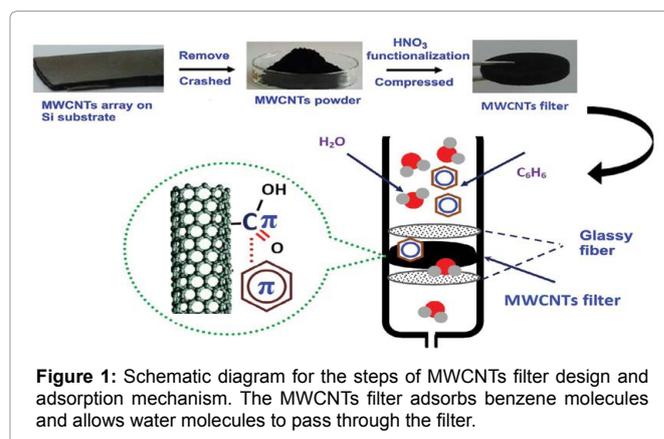


Figure 1: Schematic diagram for the steps of MWCNTs filter design and adsorption mechanism. The MWCNTs filter adsorbs benzene molecules and allows water molecules to pass through the filter.

The concentration of benzene in aqueous solution C was determined using the ratio between ring stretch peak of benzene I_B and OH-peak of distilled water I_{OH} intensities as:

$$C = \frac{I_B}{I_{OH}} \quad (1)$$

Normalizing the OH-peak of water to 1, the concentration of benzene in aqueous solution C is proportional to the ring intensity peak of benzene I_B . The removal efficiency (R) of benzene from aqueous solutions is defined as follow:

$$R(\%) = \frac{C_B^0 - C_B}{C_B^0} \times 100 \quad (2)$$

Where C_B^0 and C_B are the concentrations of benzene in the solution, initial and after adsorption. From equations (1) and (2) the removal efficiency (R) can be written as follow:

$$R(\%) = \frac{I_B^0 - I_B}{I_B^0} \times 100 \quad (3)$$

I_B^0 and I_B are the peak intensities of benzene before and after adsorption.

Results and Discussion

Characterization of raw and oxidized MWCNTs

Scanning electron microscopy and energy dispersive X-ray spectroscopy analysis: Morphology of the pristine and oxidized MWCNTs was characterized by SEM and EDS analysis. Figure 2A shows the raw MWCNTs with irregular distribution and some agglomerated parts, in addition to amorphous and metal impurities produced from the CVD method, which appear as white spots in the CNTs images in accordance with more detailed high resolution transmission electron microscopy [22]. Two different magnifications used to identify the surface morphology and outer diameter distribution for the raw MWCNTs (Figure 2A,2C) and after MWCNTs functionalization (Figure 2B,2D). The effect of nitric acid oxidation tends to functionalize the MWCNTs walls by oxygen groups. The oxidation process results in narrowing the MWCNTs diameter: high resolution SEM images showed a decrease in nanotube diameters from an average diameter 60 nm, (Figure 2C), to 25nm, (Figure 2D). Ling et al. [23] studied the influence of surface oxidation on the morphological structure of MWCNTs and found a clear change in diameter and surface roughness was occurred along the tube walls. The EDS analysis not only offers a quantitative study of the oxygen content of the modified MWCNTs, but also is a measure of metal impurities removal efficiency. Due to the synthesis procedure, MWCNTs as prepared contain a tiny oxygen content and a fraction of iron metal nanoparticles, (Figure 3A). It is clearly seen in (Figure 3B) that the iron impurity was removed after nitric acid treatment, while oxygen content increased as indicated by (Figure 4).

Fourier transform infrared spectra analysis: Fourier transform infrared spectrometry is mainly used as qualitative technique for evaluation of functional group. The FTIR spectrometry used in this study to account for the functional groups attached to MWCNTs by the oxidation process. Figure 4 shows the FTIR spectra of raw MWCNT (a) and oxidized MWCNT (b) from 500 to 4000 cm^{-1} . It is seen that the bands at 3436, 1629, and 1150 in oxidized MWCNTs are more intense than that in the raw MWCNTs spectra. The hydroxyl group peak in the spectra of the oxidized MWCNTs is more intense than that in the raw MWCNTs spectra. The band at 3436 cm^{-1} are assigned

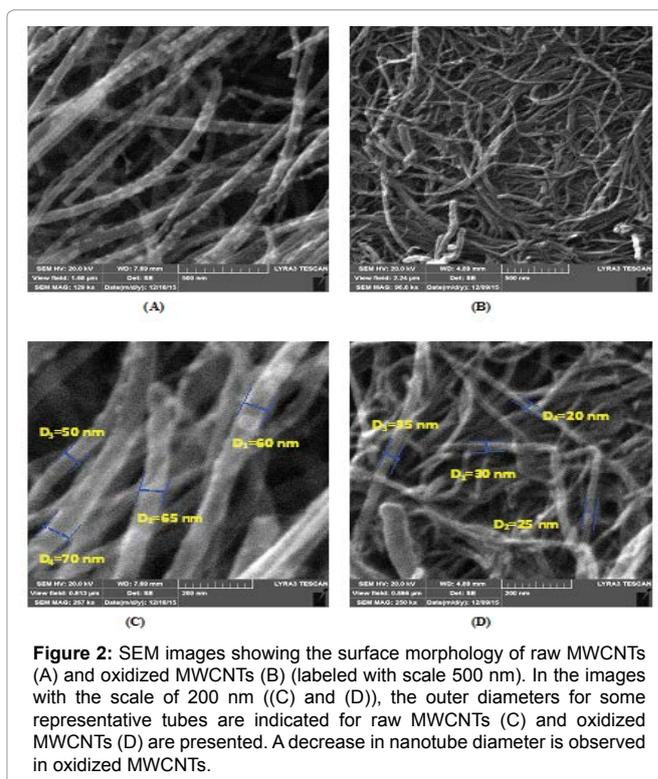


Figure 2: SEM images showing the surface morphology of raw MWCNTs (A) and oxidized MWCNTs (B) (labeled with scale 500 nm). In the images with the scale of 200 nm ((C) and (D)), the outer diameters for some representative tubes are indicated for raw MWCNTs (C) and oxidized MWCNTs (D) are presented. A decrease in nanotube diameter is observed in oxidized MWCNTs.

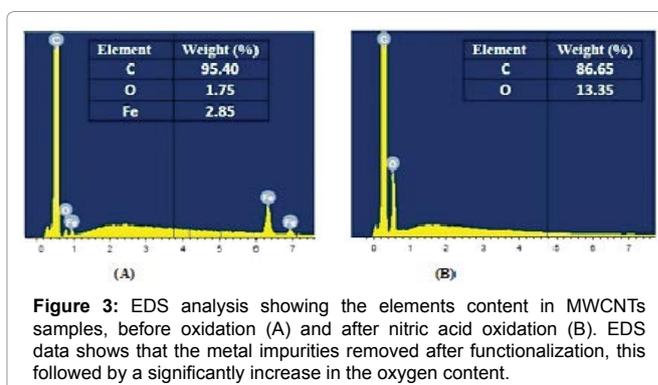


Figure 3: EDS analysis showing the elements content in MWCNTs samples, before oxidation (A) and after nitric acid oxidation (B). EDS data shows that the metal impurities removed after functionalization, this followed by a significantly increase in the oxygen content.

to -OH stretch from carboxylic groups (-COOH and -COH). In addition, band at 1629 cm^{-1} corresponding to -C=O and -COOH groups was reinforced after treatment. Peaks around 1150 cm^{-1} can be assigned to C-O stretching vibrations of the carboxylic acid group that increased after modification. Similar observations of the FTIR spectra for MWCNT-COOH were reported by Theodore et al. [24] and Wu et al. [25].

Benzene aqueous solutions analysis using Raman spectroscopy: To investigate the benzene concentration in aqueous solution, Raman spectra of four concentrations were recorded. Three measurements for each concentration were made and the mean values of these measurements for each concentration are plotted in Figure 5. It is clearly seen that, the ring stretch peak intensity of benzene at 992 cm^{-1} proportional to the concentration of benzene aqueous solutions. Raman linewidths (FWHM) of benzene peak at 992 cm^{-1} are shown as a function of benzene concentration aqueous solutions in the inset (Figure 5). In order to compare and calculate the linewidths (Γ) at different concentrations of benzene, Gaussian distribution function

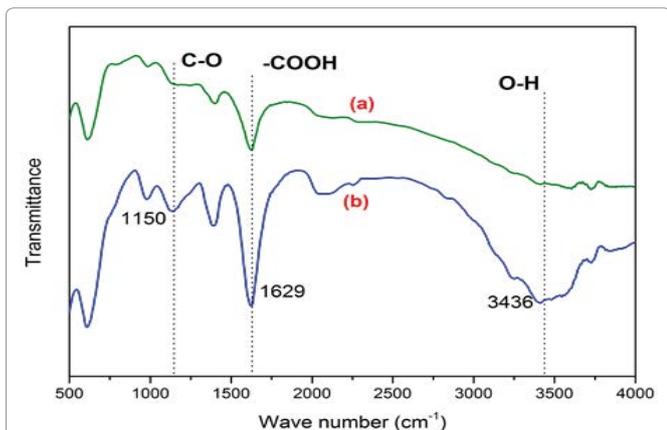


Figure 4: The FTIR spectra of raw MWCNTs (A) and oxidized MWCNTs (B) in the range from 500 to 4000 cm^{-1} showing the functional groups. The intensities of hydroxylic and carboxylic groups are increased after functionalization process.

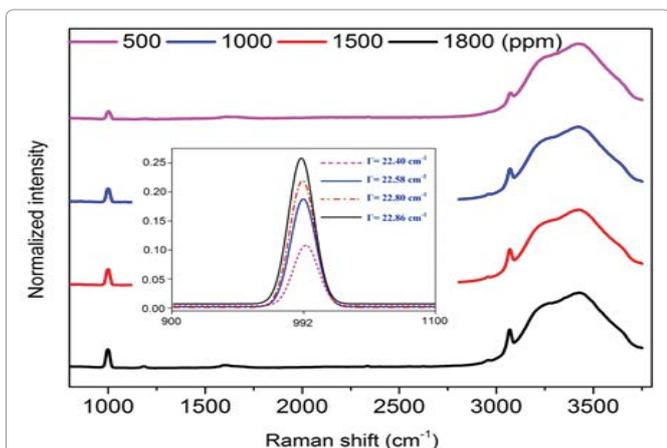


Figure 5: Raman spectra of various benzene/ H_2O solutions with concentrations ranging from 500 to 1800 ppm obtained using blue laser with wavelength 473 nm. The peak intensity of benzene at 992 cm^{-1} is multiplied by 10 for better clarity. The inset in figure shows the 992 cm^{-1} peak intensities as a function of benzene solution concentrations. The inset also shows the Raman linewidths (FWHM) of benzene peak at 992 cm^{-1} at different concentrations of benzene solution ranging from (500-1800) ppm. Gaussian function used to fit these peaks with fitting factor $R^2=0.998$.

was used to fit the 992 cm^{-1} peak for each concentration. The resulting linewidth showed a slight increase from $\Gamma=22.4 \text{ cm}^{-1}$ at benzene concentration of 500 ppm to $\Gamma=22.86 \text{ cm}^{-1}$ at high concentration of about saturation limit (1800 ppm). The increase is small indeed so we can consider the linewidth of 992 cm^{-1} peak as approximately the same, in contrast to the amplitude of the signal, indicating that there is no significant change in the ring vibration mode of benzene in aqueous solution in the concentration range studied.

The ratio of the benzene-to- H_2O peak intensities plotted against the concentration of benzene in aqueous solution in Figure 6. The plot shows well-defined linear proportionality between the ratio and the concentration of benzene as it was expected.

Effect of concentration on removal efficiency of benzene: A comparative study of the adsorption efficiency of benzene removal, R, using raw and oxidized MWCNT filters, was performed as a function of benzene concentration in aqueous solutions. The effect of high

concentration, 1800 ppm, and low concentration, 500 ppm, on the efficiency of benzene removal was investigated at $\text{pH}=7$. At 1800 ppm concentration, (Figure 7), R was 28% for 0.6 g raw MWCNTs (R-MWCNT), but increased to 60% for 0.4 g oxidized MWCNTs (O-MWCNT). At low concentration of 500 ppm, (Figure 8), R of O-MWCNT could reach as high as 99%. It is illustrated in Figure 8 where the C-H stretch peak of benzene at 3067 cm^{-1} practically disappeared after filtration. As follows from the comparison of Figures 7 and 8, the removal efficiency decreases when the concentration of benzene aqueous solution increases. High concentration limits benzene molecules transfer to O-MWCNT surfaces. Lu et al. [17] used NaOCl-oxidized CNTs for benzene and toluene removal and obtained qualitatively similar results though a quantitative figure of efficiency was not reported. To demonstrate the effect of benzene adsorption from aqueous solution on the linewidth of 992 cm^{-1} peak, the Raman linewidths (FWHM) of benzene peak at 992 cm^{-1} were measured before and after adsorption using R-MWCNT and O-MWCNT filters at two different concentrations 500 and 1800 ppm. From the inset in Figure 7 for 1800 ppm, the linewidth Γ before filtration, was 22.86 cm^{-1} and after filtration using R-MWCNT Γ was 22.76 cm^{-1} and using O-MWCNT Γ was 22.43 cm^{-1} . Whereas at low concentration of 500 ppm, the inset

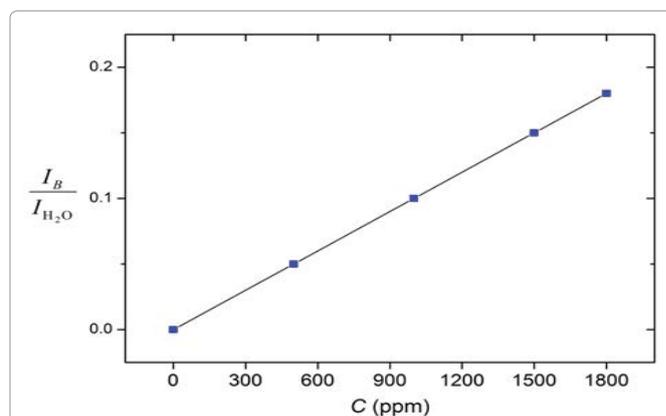


Figure 6: Ratio of the benzene and the H_2O peak intensities plotted against the concentration of benzene in aqueous solution. Each point in these data was obtained by measuring the ratio intensity corresponding to each concentration five times and taking the average.

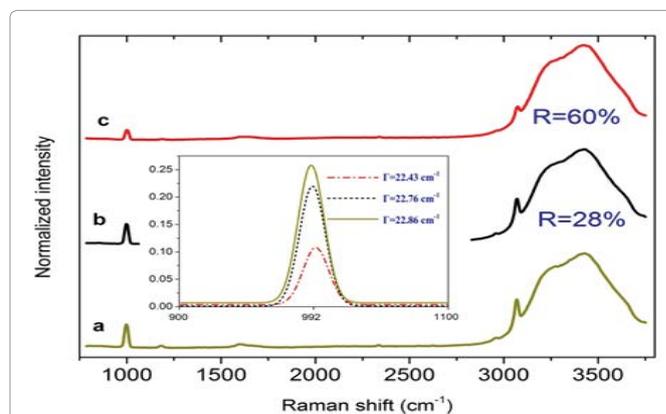
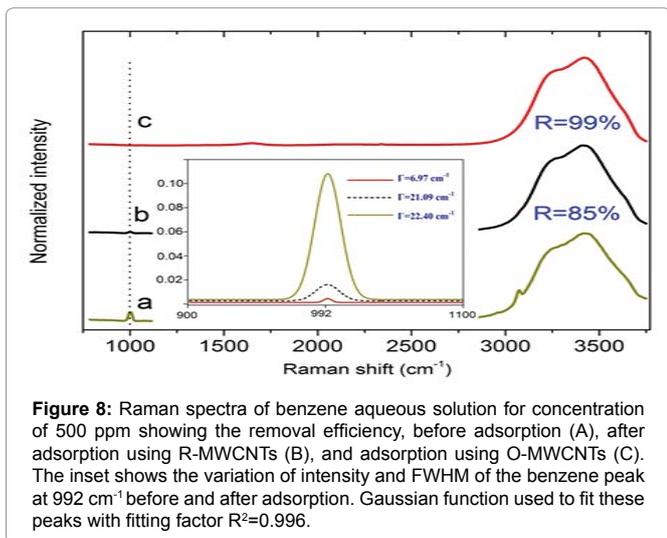


Figure 7: Raman spectra of benzene aqueous solution for concentration of 1800 ppm showing the removal efficiency, before adsorption (A), after adsorption using R-MWCNTs (B), and adsorption using O-MWCNTs (C). The inset in figure shows the variation of intensity and FWHM of 992 cm^{-1} peak before and after adsorption. Gaussian function was used to fit these peaks with fitting factor $R^2=0.997$.



in Figure 8, before adsorption, the linewidth Γ was 22.4 cm⁻¹, after adsorption using R-MWCNT Γ was 21.09 cm⁻¹ and using O-MWCNT was about 7 cm⁻¹. This high decrease in the linewidth reflects the high removal efficiency of O-MWCNT filters for low benzene concentration. These observations illustrate that the linewidth can be used to account for the removal efficiency of benzene from aqueous solutions using MWCNTs filters.

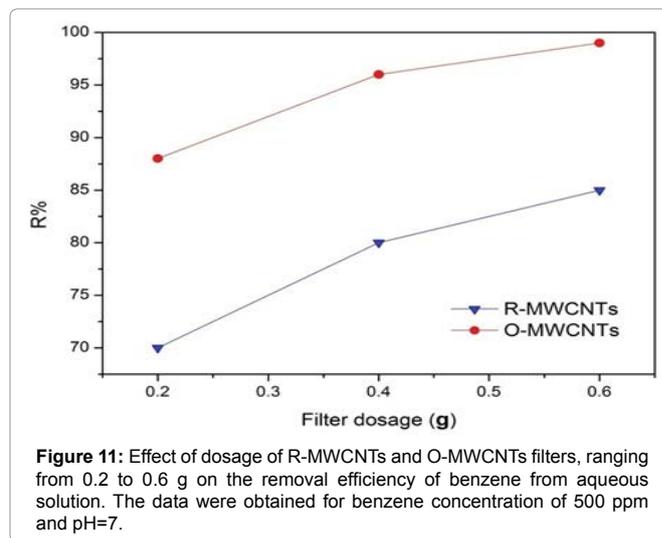
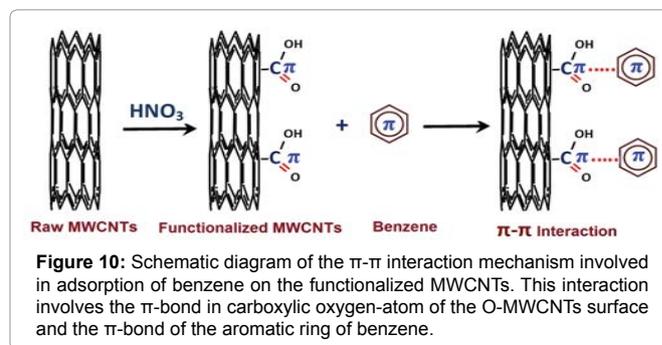
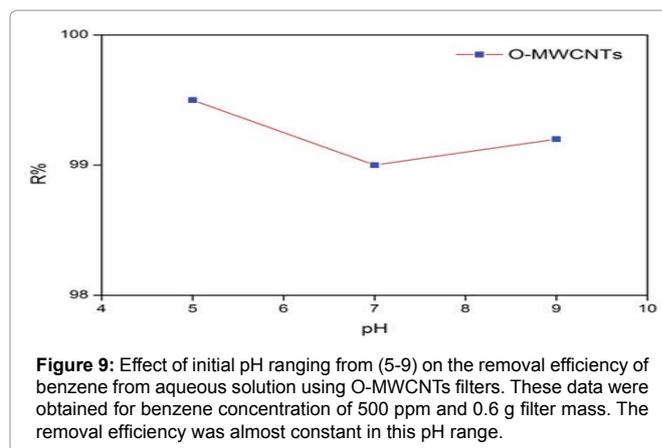
Effect of pH and adsorption mechanism: The solution pH is one of the dominant parameters controlling the adsorption efficiency. Benzene removal by O-MWCNT filters as a function of pH ranging from 5 to 9, which is a typical pH range for mostly industrial water, for 500 ppm concentration and 0.6 g filter mass, is given in Figure 9. It was observed that the removal efficiency was almost constant in this pH range. Since, benzene exists in the molecular form at whole range of pH. To investigate the adsorption mechanism of benzene on O-MWCNTs, the solution pH was measured at the beginning and at the end of each adsorption experiment [26]. Since no significant pH variation during adsorption process as well as direct connection between benzene adsorption and the presence of surface carboxylic groups of the MWCNTs was observed, it can be suggested that the π - π electron-donor -acceptor mechanism involving the carboxylic oxygen-atom of the MWCNTs surface as electron-donor and the aromatic ring of benzene as electron-acceptor is responsible for the uptake of benzene by O-MWCNTs. A diagram of the mechanism for adsorption of benzene on the MWCNTs is illustrated in Figure 10. These carboxylic groups are abundant on surface of oxidized MWCNTs, increasing the surface polarity and altering the surface charges in agreement with existing literature [27].

Effect of filter dosage on removal efficiency of benzene: The impact of filter dosage on adsorption capacity and removal efficiency was analyzed using different amount of adsorbents (0.2 to 0.6 g). Figure 11 represents the effect of filter dosage on removal efficiency and adsorption capacity of R-MWCNTs and O-MWCNTs. It was observed that with increase in the filter dosage, the adsorption efficiency has increased. The removal efficiency of benzene by O-MWCNT filter reaches the value as high as 99% for 0.6 g filter mass at pH=7 and concentration of 500 ppm. This phenomenon implied that the adsorption process depended on the availability of binding sites. The oxidation treatment had evident impact on the MWCNTs removal efficiency of benzene. It is also clear that the removal efficiency of O-MWCNTs

was higher than that of R-MWCNTs. The reason is that the O-MWCNT filters had larger specific surface area than R-MWCNT ones. This can be justified by availability of more active adsorption sites at higher filter dosage.

Conclusions

In this paper raw and oxidized MWCNTs filters were used as an effective media for benzene removal from aqueous solutions. Very high removal efficiency reaching 99% was observed for oxidized MWCNTs filters and somewhat less, about 85%, for raw MWCNTs filters for the solution with benzene concentration of 500 ppm and



filter mass 0.6 g at pH=7. Nitric acid was used to modify the surface characteristics of MWCNTs and enhance its adsorption property. The FTIR, SEM, and EDS analyses show that physicochemical properties of the MWCNTs such as structure and surface properties were greatly improved after oxidation, which resulted in its benzene adsorption capacity and adsorption efficiency to be increased by about 15%. We demonstrate that Raman spectroscopy as an excellent quantitative technique for concentration measurements of benzene in benzene/H₂O solutions, through the intensity and the linewidth of the characteristic benzene peak at 992 cm⁻¹, giving evidence that this technique can be successfully applied to water quality monitoring. It was also concluded that the adsorption using oxidized MWCNTs is effective method for the removal of volatile organic compounds such as benzene from water and wastewater. It was found that, the dominated interaction mechanism between benzene and oxidized MWCNT filters is π - π bond interaction. The removal efficiency of O-MWCNTs filters is higher than R-MWCNTs one, suggesting that functionalized MWCNTs filters are efficient for benzene removal, possessing good potential for toxic organic compounds removal from aqueous solutions and could be considered as a new kind of advanced filters for waste water treatment.

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Author Affiliation

Top

¹Skobel'syn Institute of Nuclear Physics, Lomonosov Moscow State University, Russia

²Faculty of Physics, Lomonosov Moscow State University, Russia

³Faculty of Science, Damanhour University, Egypt

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