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Evaluation of Carbon Nanotubes (CNTs) In Management of a Full Scale Centralized WastewaterTreatment Plant.

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ABSTRACT

In the present study, Carbon nanotubes were preparedlocally, characterized and investigated for wastewater treatment. In the batch mode, experiments were carried out to investigate the effect of contact time, wastewater strength and adsorbent dosage on the removal of COD, BOD, TSS, and pathogens from a real municipal wastewater. The as prepared nanotubes exhibit effective removal efficiencies over the different concentration ranges.The results show that the equilibrium time was 10 min for COD and TSS, and the maximum removal percentage for all COD, BOD and TSS was 90, 91 and 97, respectively.
Keywords: CNTs,COD,BOD, full scale plant, wastewater management**.**

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INTRODUCTION

Wastewaterdischarge from agricultural, domestic or industrial sourceshave a negative effects on the water, the health and the environment [1].The disposal of untreated wastewater into water bodies cause potential or severe pollution [2]. The demand is driven up continuously due to the fast growing of the global population and the elaboration of living standard.However, current wastewater treatment technologies are reaching their end for achieving appropriate water quality to attain human and environmental requirements[3]. Nanotechnology based treatment has offered very efficient and ecofriendly access. Thebenefitsof nanotechnologyin wastewater treatment technology focused in specific areas: sensing and detection, pollution control and treatment [4], this achieving through nano-filtration technique, adsorption of pollutants on nano-adsorbents and the contaminants breakdown by nano-catalyst [5].

A rapid increase in interest in the use of nano-materials, well developed internal pore structures of nano-particles and their tendency to functionalize with various chemical surface groups increase their affinity towards target contaminants [6]. Recent studies suggest that many of the recent wastewater problems could be solved using nanomaterials,[7],[2],[9]

Nanoparticles have greatinteracting, absorbing and reacting abilities due to theirvery small size andelevated proportion of atoms at their surface, they can achieve energy conservation due to their small size which may lead in the end to cost saving.The unique properties of CNTs have attracted the attention of many researchers, their high strength, resistance to acidic and basic media [8], high surface area [9],good thermal, electrical, and conductive properties brought up the possibility of a novel structure with extraordinary properties [10]. The CNTs consist of very thin honeycomb structures of graphene sheets rolled up in cylindrical shape with a few nanometer diameter andmany micron or even centimeters length [9], including single walled CNTs (SWCNTs) and multi-walled CNTs (MWCNTs) distinguished by the layersnumbers[10]. Due to the hexagonal network of carbon atoms [11] and strong van der Waals interaction forces between the nanotubes, they form tight bundles[9]

The CNTs have presentedexcellent adsorption efficiencies for various organic pollutants [13], heavy metals,i.e. lead, cadmium [14], Application of CNTs in wastewater treatment is not restricted to adsorption and filtration; CNTs have strong antimicrobial properties that control microbial pathogens. They are not strong oxidants and relatively inert in water resulting in avoiding the formation of carcogenic disinfection byproducts (DBPs)[15]

The effect of metal and metal oxide nanoparticles, such as nano silver[16]nano copper [17]nano gold[18] titanium dioxide [19], aluminum oxide [20], silicon dioxide[21] and zinc oxide [19]on wastewater treatment plants (WWTPs) have been widelyinvestigated. However, few studies have investigated the effectof CNTs on WWTPs. The aim of this study is to investigate the effect of CNTs on chemical oxygen demand (COD),biochemical oxygen demand (BOD), total suspended solids (TSS), nutrients and pathogens removal from real municipal wastewater. CNTs, as a new approach, were prepared locally, purified, functionalized, and investigated as adsorbent to treat real wastewater under different conditions.

MATERIALS AND METHODS

The raw wastewater used in this study was obtained from the inlet of the different wastewater treatment plants of Fayoum government (Egypt).

Experimental Methods

This section presents in detail, the description of equipment and the experimental methodologies used.

Synthesis, purification and fictionalization of carbon nanotubes

Carbon nanotubes were synthesized locally by a CVD according to the procedure described by [22] using cobalt (12 wt%) and iron (6 wt%) catalyst supported on calcium carbonate. The catalyst was prepared by impregnation of carbonate from metallic salts of $Co(NO_3)_2.6H_2O$ and $Fe(NO_3)_3.9H_2O$ solution.The chemical deposition reaction was carried out at 750ºC for 30 min. Ethyleneas a source of carbon was mixed with nitrogen at a ratio of 3:1 ethylene/nitrogen with a constant flow rate of nitrogen (400 mL/min). The product was treated with the 30 wt% nitric acid solution for 72 hr to remove the impurities, and to reduce the amorphous carbon by-products. Subsequently, the resulting product washed thoroughly using distilled water and finally dried at 125ºC for 24 hr[23].

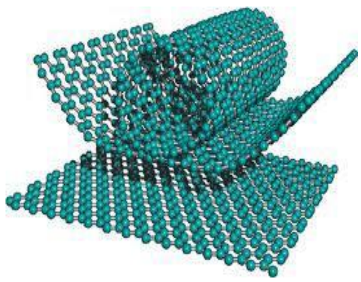
The phase structure of CNT was investigated using X-ray phase analysis (PW 1730) with nickel filtered Cu radiation at 40 kV and 30 mA. The wall thickness, tubes length and inner cavity of the produced CNTs were investigated using TEM (JEOL JEM-2100).

Adsorption experiments

Adsorption activity was evaluated by the effect of the CNTs on the adsorption of some pollutants from the raw wastewater. Exactly 1 L of raw wastewater with different initial pollutants concentrations placed in a beaker and 0.8 g CNTs was added and left on an isothermal reciprocating shaker at 400 rpm at 298 K. At specified interval times, 5 ml of the wastewater sample was withdrawn and filtered. The residual concentrations of the filtrate were determined for COD, NO_3^-, NO_2^-, and P-PO_3^- using an UV–visible spectrophotometer (Shimadzu –UV 1800, Japan), BOD using probe (Hach, HQ3OD, Chaina).The percent removal at a specific time was computed by the difference between the initial and the final concentration at that time.

Chemical oxygen demand (COD), nitrite, nitrate, ammonia, ortho-phosphate, TSSand volatile suspended solids (VSS) analyses were routinely assayed and these were performed in accordance with standard methods for the Examination of Water and Wastewater American Public Health Association (APHA 2005, 20th edition). All samples were analyzed after filtration through 0.45 mm filter paper.

Figure (1): Schematic diagram of graphene sheet layers of CNT [12]

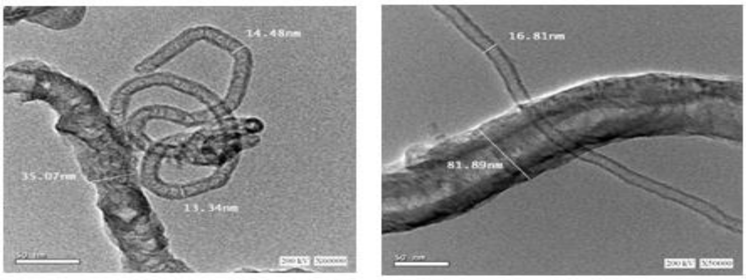


RESULTS AND DISCUSSION

Morphology and structures of carbon nanotubes

The internal structure of sample of carbon nanotubes prepared was identified by transmission electron microscope (TEM). Fig.(2) displays the morphology of CNTs prepared. The TEM figure shows that the CNTs are cylindrical and that the range of external diameters is 13.34 -81.89 nm. The TEM analysis confirms the growth of MWCNTs structure.Structurally, hollow, layered structures.

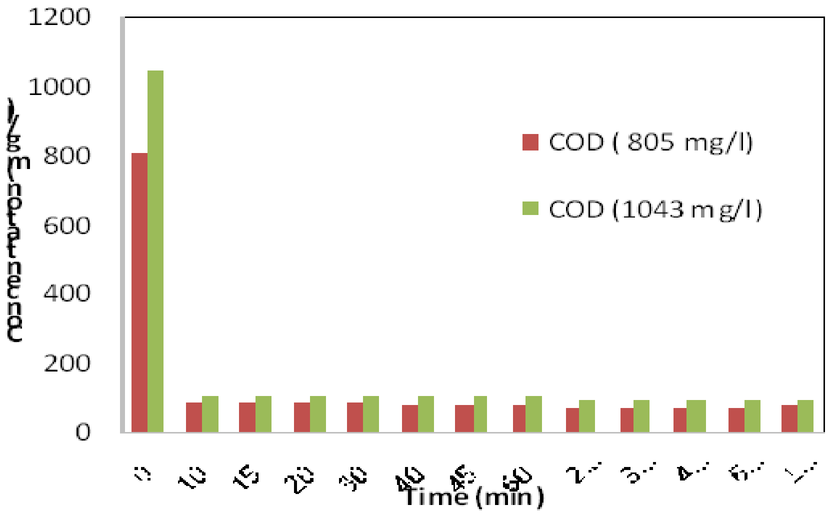
Fig.2: TEM images of the prepared samples for CNT



Effect of the contact time on COD removal

For determining the removal efficiency of CNTs, the COD, BOD, and TSS was chosen as a study parameters because its unit of mg/L was more convenient. To understand the behavior of all compounds in the wastewater, each compound was analyzed alone and is presented in Figures (3:5). Approximately 0.8 g of CNTs were added to 1000 mL of raw wastewater with different concentrations of COD ranges (805 and 1043mg/L). The effect of contact time on the sorption of COD is shown in Figure (3).Adsorption was the principal COD removal mechanism that was represented by COD remaining in solution after the treatment process.

Fig.3: Concentration profiles of COD during 24 hr.



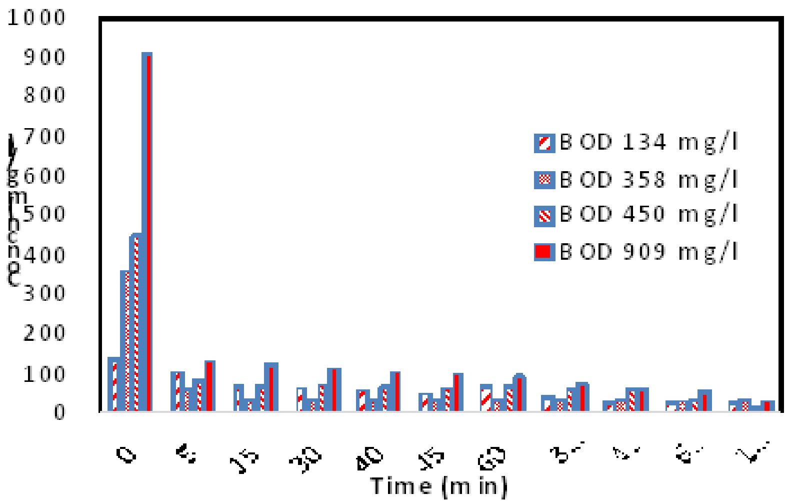
As can be seen from Fig. 3, It is remarkable note that there is a sharp decrease in COD concentration, after 10 min about 90% of the initial concentrations 805mg/l and 1043mg/l was removed, then no significant difference was noticed until the end of 24 hrs for all the concentration range under study, It is clearly obvious that the time of 10 minutes is sufficient for COD sorption to reach equilibrium. However this rapid equilibrium rates, and high sorption capacity suggest CNTs a competitorforactivated carbon (AC), the most famous adsorbentin water treatment.This result was agree with that CNTs can sorb soluble COD because of their large surface area to volume ratio. However, according to [24], there are four possible adsorption sites present in CNT bundles: 1. Internal sites: the hollow interior of individual nanotubes accessible only if the caps are removed and the open ends are unblocked. 2. Interstitial channels between individual nanotubes in the bundles. 3. Grooves present on the periphery of a nanotube bundle and the exterior surface of the outermost nanotubes, where two adjacent parallel tubes meet. 4. The outside surface: the curved surface of individual nanotubes on the outside of the nanotube bundles. Yang and Xing explained that the external surface of CNTs are the major area for the adsorption of organic contaminants, while the inner pores of tubes are not available sites of sorption due to large size of organic molecules compared to the entrance of that area. The interaction between organic contaminants and CNTs is attributed to π-π electron donor- acceptor (EDA) interaction

[25]Many of polar organic compounds is strongly adsorbed due to the various contaminants/CNT interactions including covalent bonding, hydrophobic effect, electrostatic interactions and hydrogen bonding,[26]The results indicate that CNTs have been proven to possess great potential as superior adsorbents for removing many kinds of organic and inorganic pollutants which may be attributed the high surface area of CNTs which mainly attributed to the outer-exposed surfaces [27] in contrast to AC whose surface area resulted from its porous structure [28]. Thus, the availability of surfaces for adsorption of organic contaminants on CNTs would be higher than that of AC, due to effects of pore blocking. There are enhanced interactions between functional groups of CNTs and contaminants in wastewater due to hydrogen bonds [29].

Effect of the contact time on BOD removal

Fig. 4 shows the BOD concentration profile for initial concentration ranges C1(134mg/l), C2 (358 mg/l), C3 (450 mg/l) and C4 (909 mg/l), after the sorption reach equilibrium at 15 min, The percentage removal up to 51% , 91% , 85% and 89 % for C1, C2, C3 and C4, respectively,CNTs show short equilibrium times, higher availability and adsorption capacity for BOD due to their highly porous and hollow structure, large specific surface area[30]. However, this short equilibrium time help in speedy treatment of wastewater.

Fig. 4: Concentration Profiles of BOD_5, during 24 hr



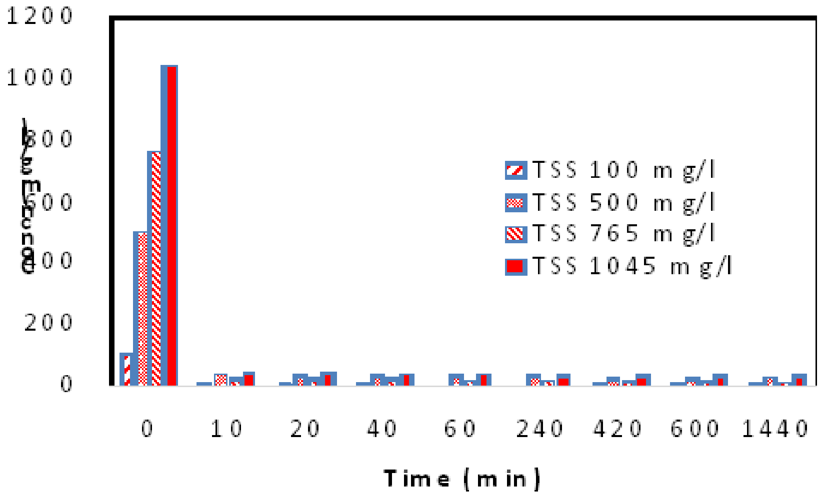
In the case of BOD, as the react period increases, there is more time for the biological reaction, organic matters (OM) present in wastewater improve CNTs colloidal stability which increase the removal of other biological contaminants[31]. BOD removal efficiency is higher in the case that has more reaction time. The characteristics of CNTs suggest that they have strong interaction with organic molecules through non-covalent forces, hydrogen bonds, for instance, polycyclic aromatic hydrocarbons (PAHs) electrostatic forces and van der Waals forces. On the other hand, the main interactions responsible for the adsorptions of organic compounds by CNTs are: hydrophobic effect, π-electron donor–acceptor (π-EDA) bonds and hydrogen bonds. Multilayer adsorption may also occur when organic chemicals are adsorbed on the surface of CNTs**.** The efficiency of sorption of BOD is lower than COD , this may be attributed to cytotoxicity of CNTs of different sizes on Escherichia coli, researchers demonstrated that CNT are toxic to bacteria [32], [53] also demonstrated that SWCNTs are more toxic to bacterial pathogens.

Effect of the contact time on TSS removal

Total suspended solids (TSS)found in the water column are particles that are larger than 2 microns, while particles smaller than 2 microns are considered dissolved solids. Most suspended solids are made up of inorganic materials, bacteria and algae can also contribute to the total solids concentration. The decomposition of algae, plants and animals decay, allows small organic particles to break down and enter the water stream as suspended solids[33]The concentration profiles of initial TSS concentration of 100, 500, 765 and 1045 mg/l are shown in Fig. (5), the results show high TSS percentage of removal. There is a sharp decrease in the case of TSS, after the first 10 min. The adsorption of TSS on CNTs is very fast as the equilibrium is reached in 10 min. Coagulation implies the step where coagulant destabilizedparticles, lead to the formation of small aggregates by the Brownian motion, then flocculation occur when larger aggregates are formed by shearing[34]. The phenomena of heterogeneous coagulationand flocculation in addition to sorption was appliedin this study, since the colloidal particles found in municipal wastewater are negatively charged while CNTs are positively charged, The repulsive potential of electrical double layer of colloids is reduced so that micro-particles can be produced in the first stage. Subsequently, the micro-particles collide with each other and form large flocs in the subsequent stage [35], resulting in a percentage removal up to 95, 93, 97 and 96 % for initial TSS concentration of 100, 500, 765 and 1045 mg/l, respectively.

The results detected a sudden decrease in COD, $BOD_5$and TSS as shown in Fig. (3:5),this can be the consequence of the fact that a large number of free surface sites are available for sorption during the initial stage and, afterwards, the remaining free surface sites are difficult to be occupied, because of repulsive forces between the phases[36].Also it indicates that the adsorption mechanism of CNTs is completely different from that of activated carbon. The adsorption by activated carbon depends mainly on the porous structure, so it takes time for adsorbents to diffuse through pores [37] while the spaces available for adsorption in CNTs are mainly due to the surface site of external wall of cylindrical shape and do not depend on the inner cavities and inner wall spacing [38],Tiehmet at 1999reported a percentage removal of 45-90% of COD and 35-80% of phosphorusassociated with suspended solids.In this study, the high removal percent of COD may resulting in thehigh removal percent of TSS.

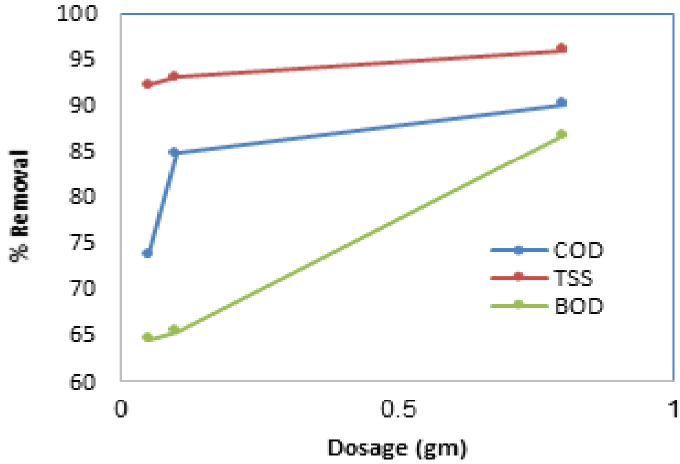
Fig. 5: Concentration Profiles of TSS during 24 hr.



Effect of CNTS dosage on COD, BOD and TSS removal

To investigate the effect of dosage of CNTs, three doses of CNTs were investigated. The results of the effect of varying the dosage on COD, BOD and TSS removal are shown in Fig. (6), where 0.05, 0.1 and 0.8 g of CNTs was mixed with 1000 ml of raw wastewater. The tests were performed at contact time of 20 min. As shown in Fig. (6), there is a significant reduction in COD, BOD and TSS up to dosages of about 0.05 to 0.8 g. Beyond this range, the rate of COD removal is less than 75 %, and BOD removal is less than 65 %. An increase in adsorption rate with adsorbent dosage can be attributed to increased surface area and the availability of more adsorption sites.This is evidence to the fact that with more CNTs, more charged sites are available for increasing energy adsorption sites such as CNT defect, functional groups.The adsorption efficiency increased with increasing the amount of nanoparticle, this is due to increase in surface area where the adsorption takes places. There is a sharp increase in the case of BOD, a possible explanation is that CNTs may act as a supporting media for the microorganisms which facilitate the intake of substrate in addition to adsorptive characteristic of CNTs.

Fig. 6: Effect of CNTs dosage on COD, BOD and TSS % removal (contact time = 20 mi



Effect of CNTs on nutrients removal

Effective treatment is needed for N removal beforedischarging of wastewater to the environment.Adsorption occurs when the attraction between the nutrients and CNTs is greater than the cohesive energy of the nutrients itself. In order to investigate the effect of CNTs on the adsorption of nutrients, approximately 0.8 g of CNTs were added to 1000 mL of raw wastewater with different concentrations of NO_3^-, NO_2^-,and P –PO_4 respectively as presented in Figures (7:9).

As can be seen from Fig. 7, It is remarkable note that there is a sharp decrease in NO_3^- concentration , after 60 min about 67% ,65%, 65% and 62% of the high initial concentrations 80.7 ,70, 62.5 and 25 mg/l respectively was removed, and reached above 80% after 600 min, then no significant difference was noticed until the end of 24 hrs for the concentration range under study, It is clearly obvious that the time of 10 hr is sufficient for NO_3^- sorption to reach equilibrium in high NO_3^- concentration samples. The high expectations of using nanomaterials as special adsorbents to remove nutrients relies not only on the highsurface area and the high affinity to organic compoundsbut also greatly on the fact that nanomaterials can be engineered or modified specifically toenhance selectivity to specific target pollutants such as nitrogen.

As can be seen from Fig. 8, nitrite exhibited decrease sharply after 60 min, removal efficiency of 81% was reached in the case of initial concentrations 0.18mg/L, a small difference in nitrite concentration profile was noticed until the end of 24 hrs for the concentration range under study , It is clearly obvious that the time of 8 hr is sufficient for NO_2^- sorption to reach.

As can be seen from Fig. 9, there is a sharp decrease in P-PO_4^- concentration, after 60 min about 40% removal of the highest initial concentrations (35 mg/l) was attained, the efficiency of removal reached above 93% after 600 min. It is clearly obvious that the time of 10 hr is sufficient for P-PO_4^- sorption to reach equilibrium, while conventional treatment process removes a maximum of 20 to 30% of the influent phosphorous [4].Adsorptioncharacteristic is a function of both, average size and chemical composition of OM in wastewater and the physical properties of the CNTs. [39]. The average size of OM varies between 0.5and 5 nm, thus the removal efficiency of OM largely varies. On the other hand, due to the disperse nature, different parts of OM interact with the CNTs surface differently. This will affect its removal by the adsorbent significantly. Adsorption of nutrients on CNTs may occur at four regions, at the CNTs external surface, at groves of the boundary ofnanotube bundles, athollow open ended interiors of nanotubes, or at interstitial spaces between the tube bundles.Because of the external diameter and large mesoporous volume of CNTs[40], CNTs symmetrical structure and surface defects [41],CNTs show stronginteractions with these compounds, in addition to that, nutrients in wastewater, i.e. nitrate, nitrite, phosphate are negatively chargedresulting in strong electrostatic attraction to the positively charged CNTs.

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Fig. 7: Concentration Profiles of NO_3^-, during 24 hr.

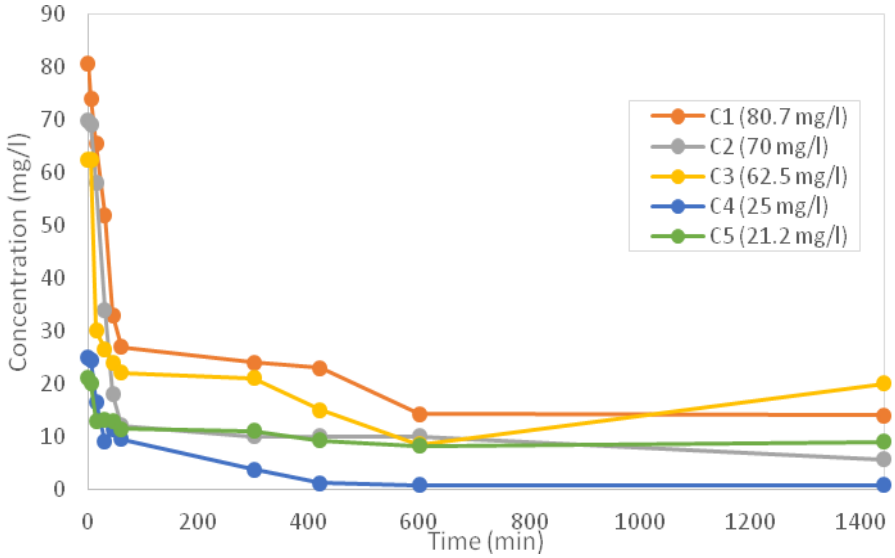


Fig. 8: Concentration Profiles of NO_2, during 24 hr.

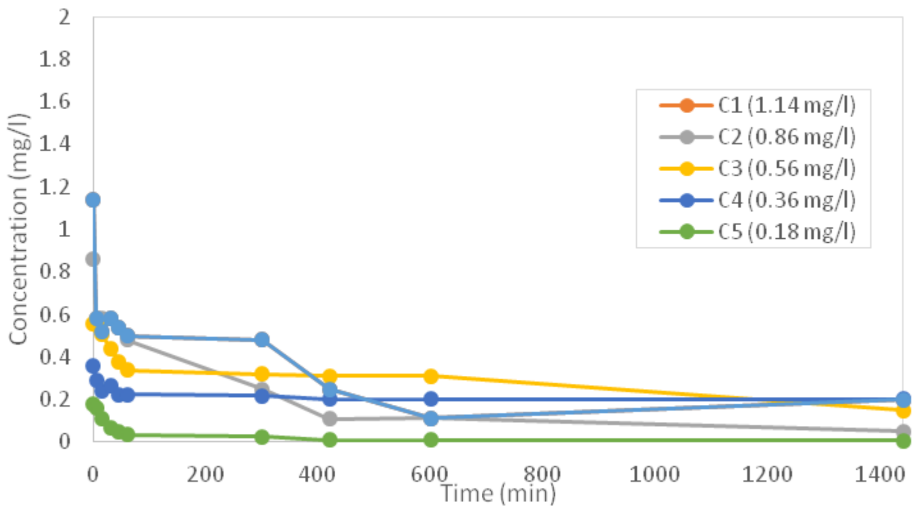
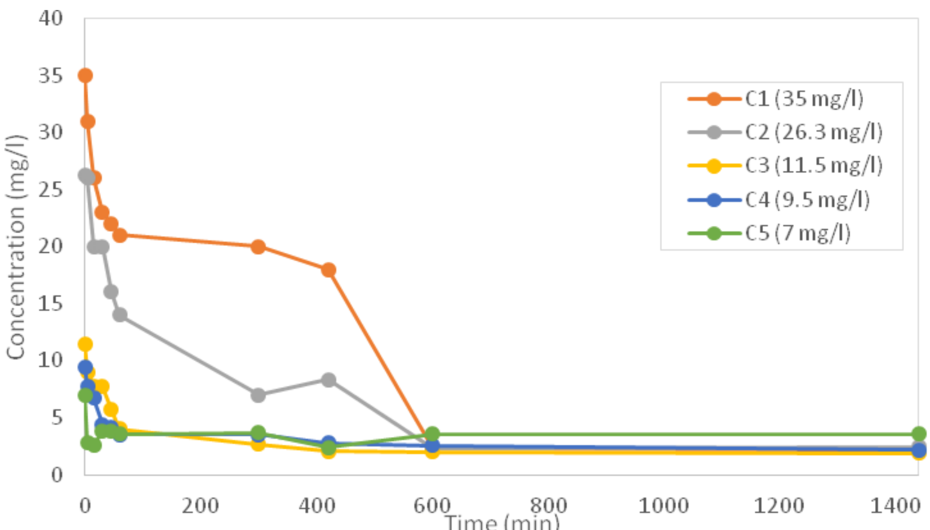


Fig. 9: Concentration Profiles of P-PO_4, during 24 hr



Biological experiments

ConventionalWWTPs are not designed tohandle pathogen [42], also a slight variationin influent raw wastewater quality effect on pathogen removal efficiency[43]. The use of traditional disinfectants such as Chlorine, potassium permanganate, ozone, and hydrogen peroxide had limitation in disinfection due to high sensitivity to changes in pH or temperature of wastewater[44].Another problem is the reaction between chlorineand organics present in waterwill lead to formation ofdisinfection byproducts (DBP's) which areclassified aspossible human carcinogens.[15]observed strong antimicrobial characteristic of CNTs.Highly purified CNTs exhibit strong antimicrobial activity toward bacteria, This can be attributed to impairment of pathogen cellular function by destruction of major constituents (e.g., cell wall), interference with the pathogen cellular metabolicprocesses, and inhibition of pathogen growth by blockage of the synthesis of key cellular constituents (e.g., DNA, coenzymes and cell wall proteins).

The percent removals of Logconcentration.MPN/100ml as a function of time are shown in Figures (10&11). Biological results show percent removal above 99% for total and fecal bacteria through one hour only in spite that the concentration of those bacteria in raw wastewater reached millions, represent rapid kinetics and high sorption capacities over a wide range of bacteria. This may be attributed to the unique physical andcytotoxic properties of CNTs. This very good result due to pathogenic effect of that bacteria,this might be because the long-term accumulation of CNTs appears to lead to an increase in antimicrobial properties.CNTs kill bacteria by causing disturbance of the cell membrane, oxidative stress, or disruption of a definite microbial process[45]. CNTs have been established to be poisonous to different kinds of organisms[46]. CNTs exhibit antimicrobial characteristics without strong oxidation, and hence have lower tendency to form disinfection by product like chlorine [47].Another suggestion of disinfection mechanism of CNTs is that the CNTs thin layer successfully remove bacteria and viruses by size exclusion and depth filtration [48] in which CNTs would allow molecules of water to pass through the cylinders interior while microbe could not, inactivation of the retained bacteria occur within hours.Most of biological substances (e.g. bacteria and virus) have sizes larger than the pore size of micro porous adsorbents [49], resulting in limiting removal of that substance. In case of CNTs, external surface area and presence of pores with volumes greater than mesopore increase the adsorption of biological substances. The thin fibrous shape of CNTs impinge bacterial cell surface, then,oxidative stress disrupt the intracellular metabolic pathways and subsequently releasing of the internal contents [32].

In agreement with previous studies, CNTs exhibited antimicrobial activities over Streptococcus mutans[50], Micrococcus lysodeikticus[51], E. coli [32] and bacteria endospores [52], Salmonella [53], protozoa species [54], and viruses [55].

Fig.10: Total coliform group (Log.MPN/100ml) in the raw wastewater samples (sample 1=34×107,sample 2=35×107,sample 3=3.5×109 ,and sample 4=3.5×109)

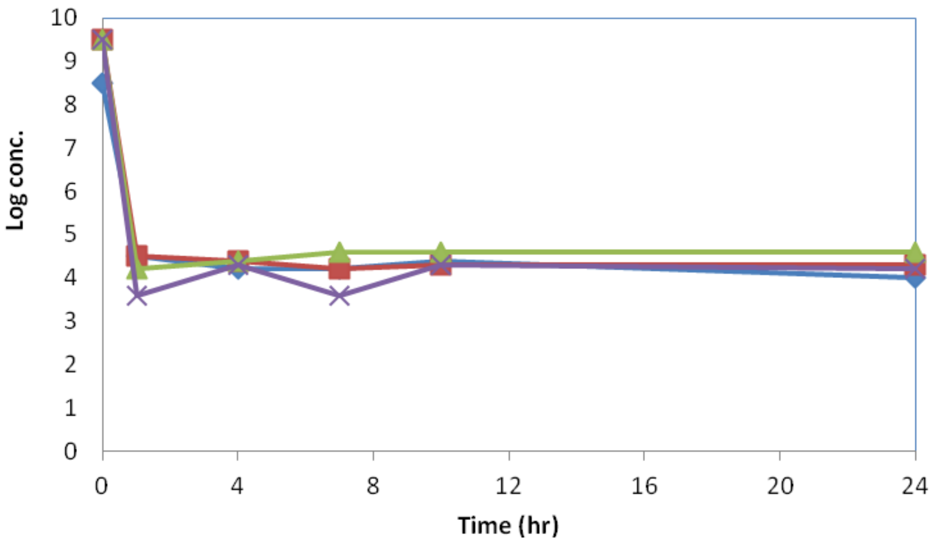
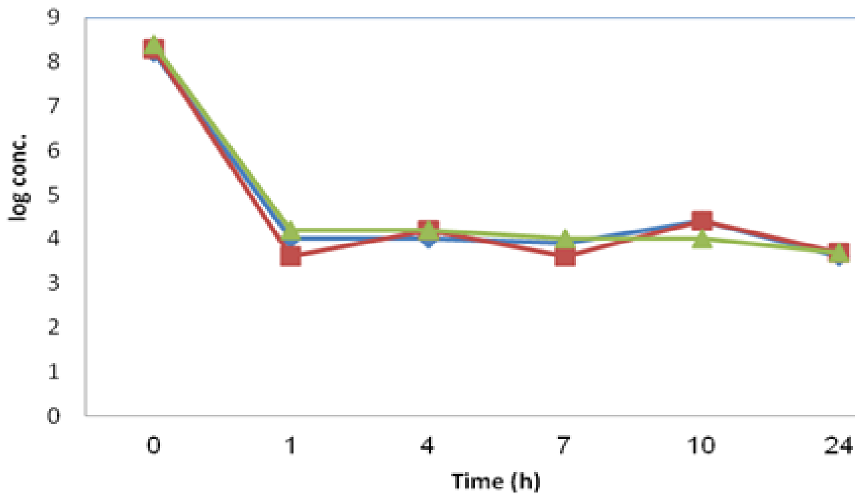


Fig.11: Fecal coli form group (Log.MPN/100ml) in the raw wastewater samples (sample 1=17×107 , sample 2=2×109and ,sample3 =23×107)



CONCLUSIONS

The CNTs were successfullyprepared, purified, and used in treatment of raw wastewater. Such process may be considered as a promising method for removal of COD, BOD, TSS and pathogens from wastewater. The CNTs identified by TEM clearly possessed multiwalled, and single tubular structures with diameters between (13.34 -81.89 nm). The carbon nanotubes which were prepared in this study are found to be efficient for the adsorption of biodegradable and non-biodegradable pollutants. It was found that removal percentage of pollutants increases with increase in the contact time and CNTs dosage. The CNTs exhibit strong antimicrobial activity toward coliform group.Based on this study, CNTs pose a great potential as a promising material for application in wastewater management.

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Thanks for god, my parents, my husband, and my doctors.

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