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ORIGINAL ARTICLE

Removal of anionic and cationic dyes from wastewater by adsorption using multiwall carbon nanotubes

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KEYWORDS

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Dyes;
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Abstract This paper evaluated the efficiency and reusability of multiwall carbon nanotubes (MWNTs) on removal of cationic and anionic dyes under effect of pH, dose of MWNTs and concentration of dyes. The characterization of MWNTs is characterized by scanning electron microscope (SEM), transmission electron microscope (TEM), Raman spectra and BET (Brunauer, Emmett and Teller) surface area. SEM and TEM analyses showed that MWNTs had size within nano scale range of 10–50 nm. The experimental results indicated that the efficiency of removal of MWNTs increase under condition of normal pH, at contact time 60 min with agitation speed 240 rpm and initial concentration of dyes 10 mg/l. Under these optimal conditions, the removal reached 98.7% and 97.2% for anionic dyes and cationic dyes, respectively. For economic use, MWNTs can be used more than one time where the same experiments with the already used MWNTs was repeated and it was found that the percent removal is almost the same.

1. Introduction

By the humanity evolution, a revolution in the industry is already occurred. Industrial processes are coupled with the degradation of the global environment and resource depletion (Das et al., 2014). Contamination of surface and ground water with toxic organic substance as synthetic dyes for example is considered as consequential environmental problem. This is a threat not only to the human being but also to aquatic life and all living organisms were the demand of water increase as water pollution increase (Hossain et al., 2015). Synthetic dyes as acid scarlet 3R, auramine yellow and crystal violet

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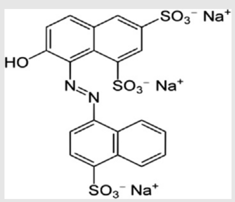
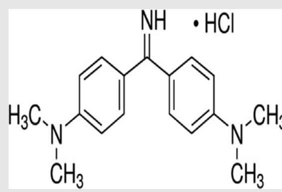
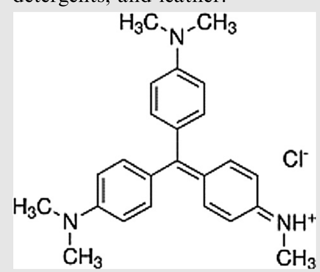
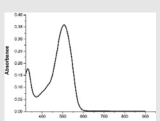
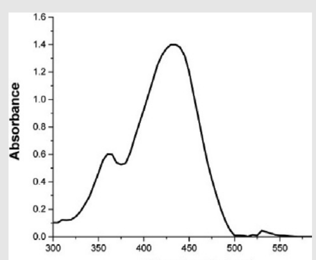
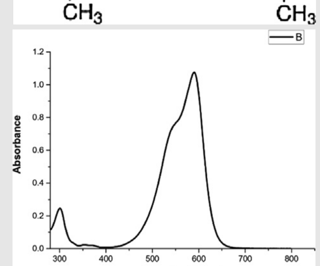
Table 1 Color, chemical formula, molar mass and maximum adsorption wavelengths of dyes.

Properties of dyes	Acid Scarlet 3 R	Auramine O	Basic Violet 2B crystal
Color index	16,255	41,000	42,535
Chemical formula	$C_{20}H_{11}N_2Na_3O_{10}S_3$	$C_{17}H_{21}N_3 \cdot HCl$	$C_{25}H_{30}CLN_3$
Molecular weight (g/mol)	604.47	303.83	407.89
Maximum wavelength	506 nm	432 nm	590 nm
Type of dyes	Acid red dyes(anionic dye)	Basic yellow dyes(cationic dye)	Basic violet(cationic dye)
Uses	coloring agents in the textile, paper, leather, gasoline, pharmaceutical, and food industries.	paper mills, textile mills, leather and carpet industry	Coloring agent for bacteria stain in medicine also Crystal Violet 2B is used as a textile dyes, dyes paper and as a component of navy blue and black inks for printing, ball-point pens, and inkjet printers. It is also used to colourizer diverse products such as fertilizers, antifreezes, detergents, and leather.
Molecular structure			
Absorption spectrum			

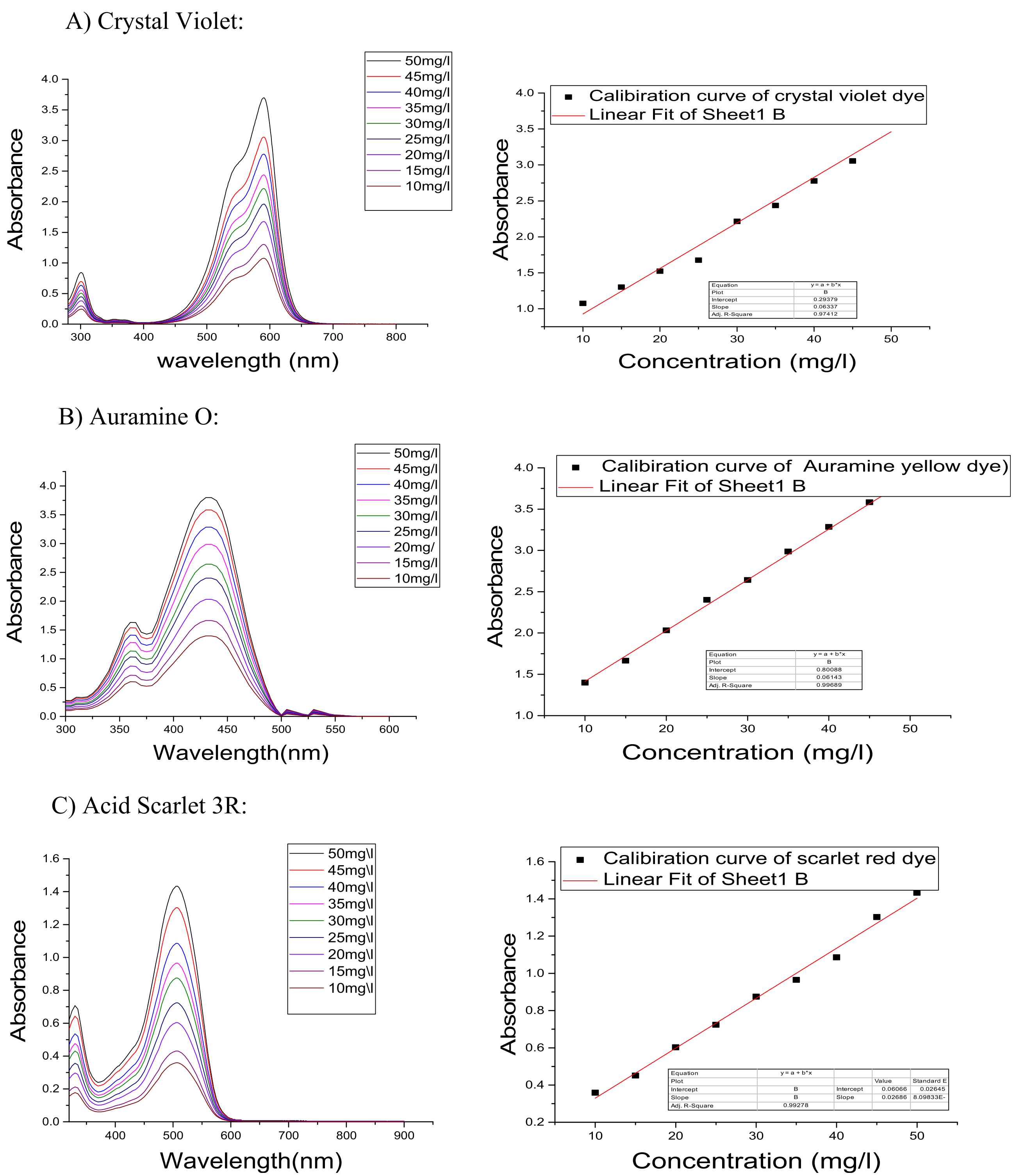


Fig. 1 Calibration curves of the three dyes (a) crystal violet, (b) auramine O and (c) acid scarlet 3R.

2B for example are boundless used as coloring agents in the textile, paper, leather, gasoline, pharmaceutical, and food industries. Their gushes into the aquatic territory have a significant source of pollution. Their degradation produced poisonous materials which absorb the oxygen of the water (Mahmoodi and Arami, 2009; Pirkarami et al., 2013). This has risen much as it threatens human life and the environment. Due to their nature and complexity of their organic structure, the dyes cause undesirable color to the water, resisting photochemical and biological attack, dropping sunlight penetration (Fu and Viraraghavan, 2001; Lazar, 2005).

Also, their degradation products like aromatic amine being toxic (Ivančev-Tumbas et al., 2008), even mutagenic, carcinogenic and rose COD and BOD levels of aquatic resources

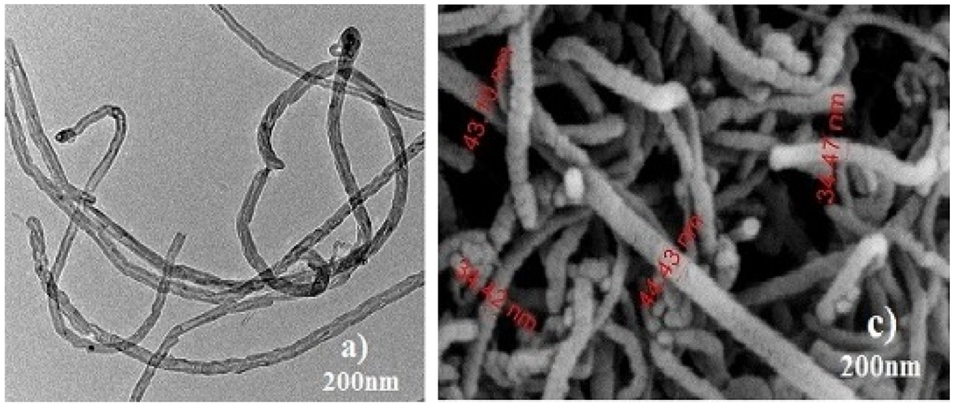


Fig. 2a and 2c . TEM and SEM images of MWNTs.

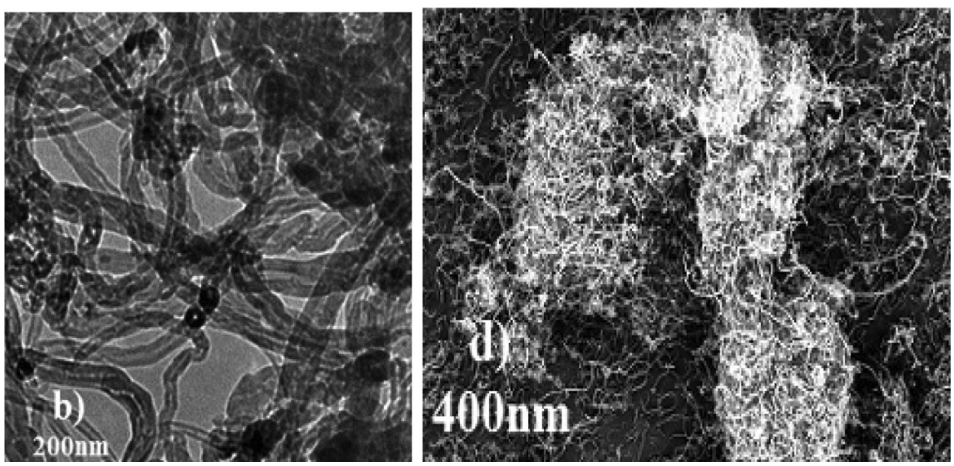


Fig. 2b and 2d TEM and SEM images of MWNTs after adsorption of dyes.

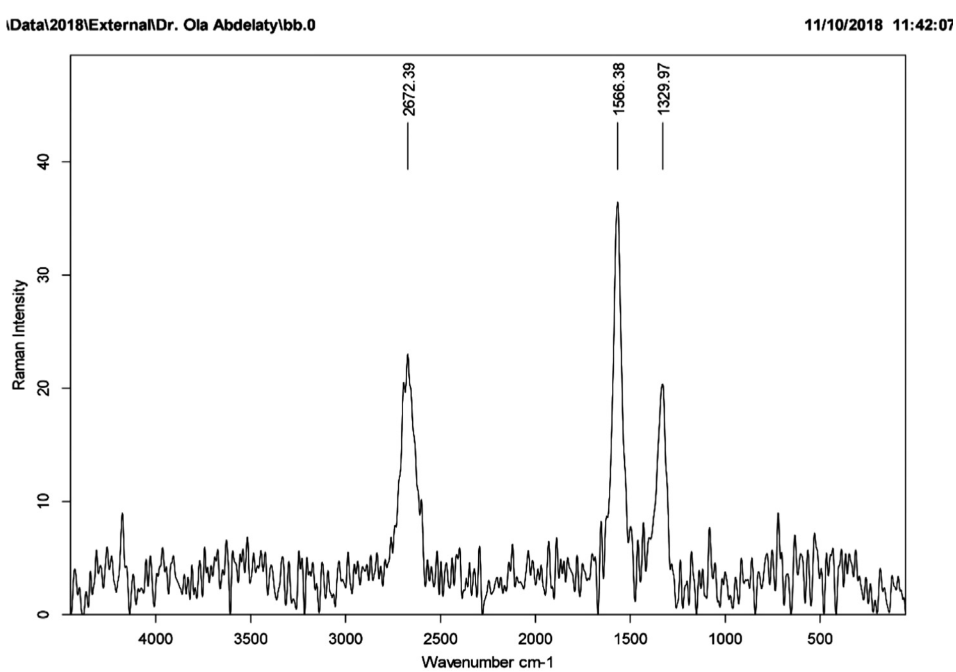


Fig. 3a Raman spectrum of MWNTs before adsorption of dyes**.**

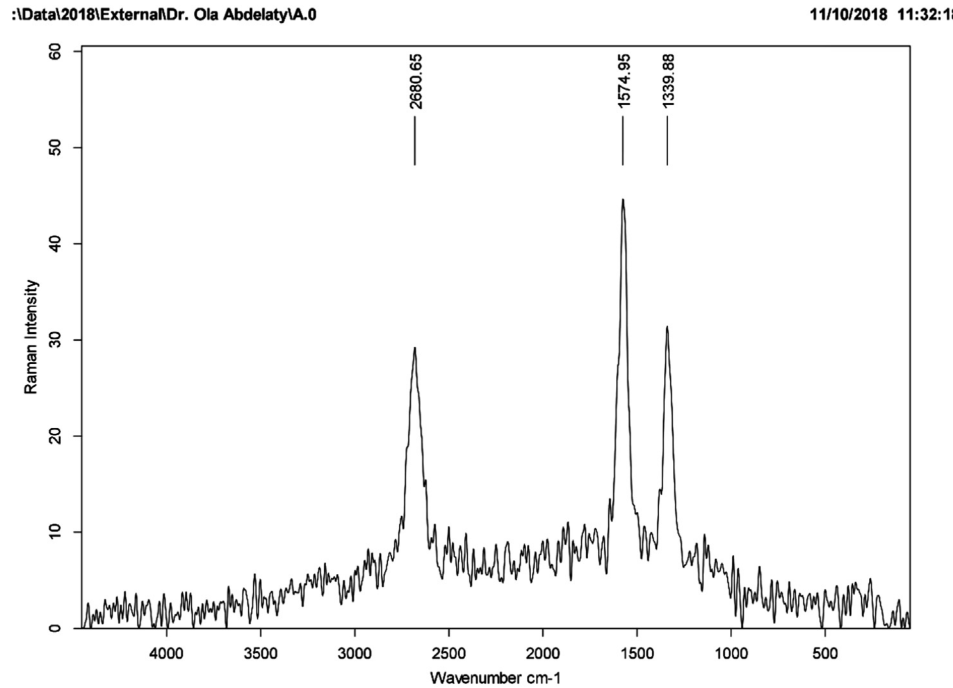


Fig. 3b Raman spectrum of MWNTs after adsorption of dyes.

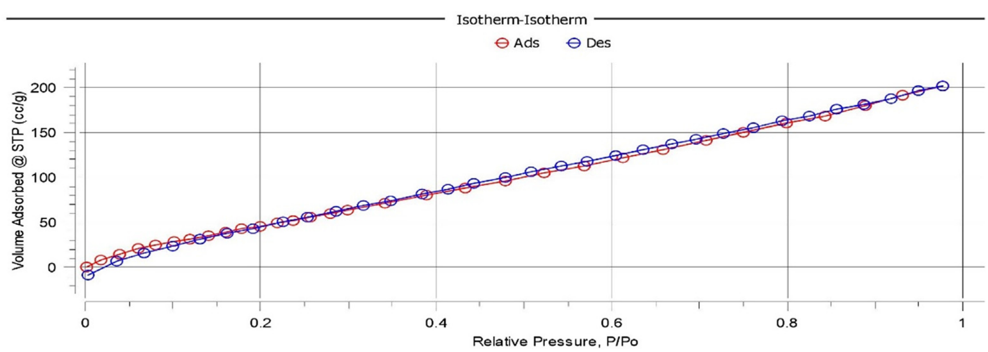


Fig. 4a BET surface area of MWNTs (a) N_2 adsorption–desorption isotherm based surface area measurement.

(Mu et al., 2009; Crini, 2006). Because of toxicity of dyes, it is obligatory to discard dyes from wastewater.

Due to complex aromatic structure and synthetic origin of the dyes, they make them stable to the oxidizing agents, biodegradation, and heat and photo degradation, (Mu et al., 2009; Banat et al., 1996; Wu, 2007). So many technologies as electrochemical techniques, degradation, chemical reduction (Qiu et al., 2012; Yao et al., 2014), membrane filtration (Das et al., 2014; Ismail et al., 2009), aerobic or anaerobic treatment, flocculation, coagulation (Das et al., 2014), fermentation of waste activated sludge using peroxymonosulfate, aerobic granular sludge (Yang et al., 2020,; Yang et al., 2019) and adsorption (Gupta et al., 2012; Mehrizad et al., 2012) have been developed and applied worldwide for removal of dyes. By taking the advantage of high simplicity, efficiency and low cost, the adsorption is the most widely technique used for removable of pollutants from aquatic resources. Although the adsorption capacity of an adsorbent is being conditional on its physicochemical characteristics and chemical composition. Example on physicochemical characteristics was dispersion, shape and size of particles.

The discovery of carbon nanotubes which is highly applicable since exceptional surface assimilation strength and advanced sorption efficiency with their controlled pore size and advanced distribution surface active site to volume ratio, compared to formulaic granules such as activated carbon (Ong et al., 2010); graphene oxide and nano particle size. CNTs showed adsorption capability and extremely adsorption efficiency for abstraction of organic pollutants like, trihalomethanes, 1,2-dichlorobenzene, carbon tetrachloride (CCl_4), and n-nonane (Peng et al., 2003; Ren et al., 2011) and heavy metals (e.g., Cu(II), Pb(II), Cd(II), and Zn(II)). Based on the mentioned above. this paper aims to investigate the efficiency and reusability of multiwall carbon nanotubes (MWNTs) in removable of hazard dyes such as cationic (auramine O and crystal violet) and anionic (acid scarlet 3R) dyes

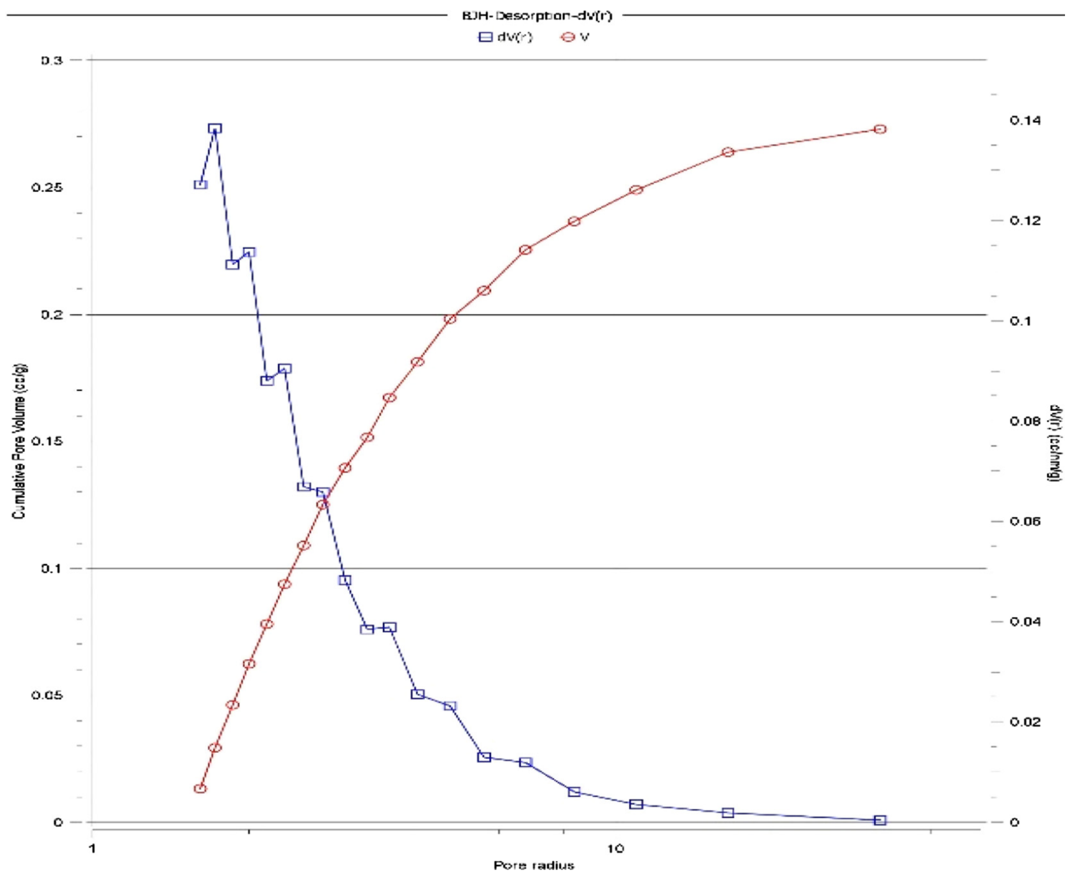


Fig. 4b Pore size measurements of MWNTs.

which are used in textile, paper, leather, gasoline, pharmaceutical, and food industries. This is done by estimating the efficiency of MWNTs at a different pH values for acid at pH = 3, for neutral pH = 7.56 (7–8) and for base pH = 9. This factor is applied for three dyes then the effect of initial concentration of dyes with different dose from 10 mg to 50 mg was carried out, and how dose of MWNTs affect its efficacy. Also, the effect of contact time on the adsorption processes was studied on both acidic and basic dyes. The removal of dyes was tracked by following the change in color using UV Spectrophotometer.

2. Experimental methodology

2.1. Chemicals and materials

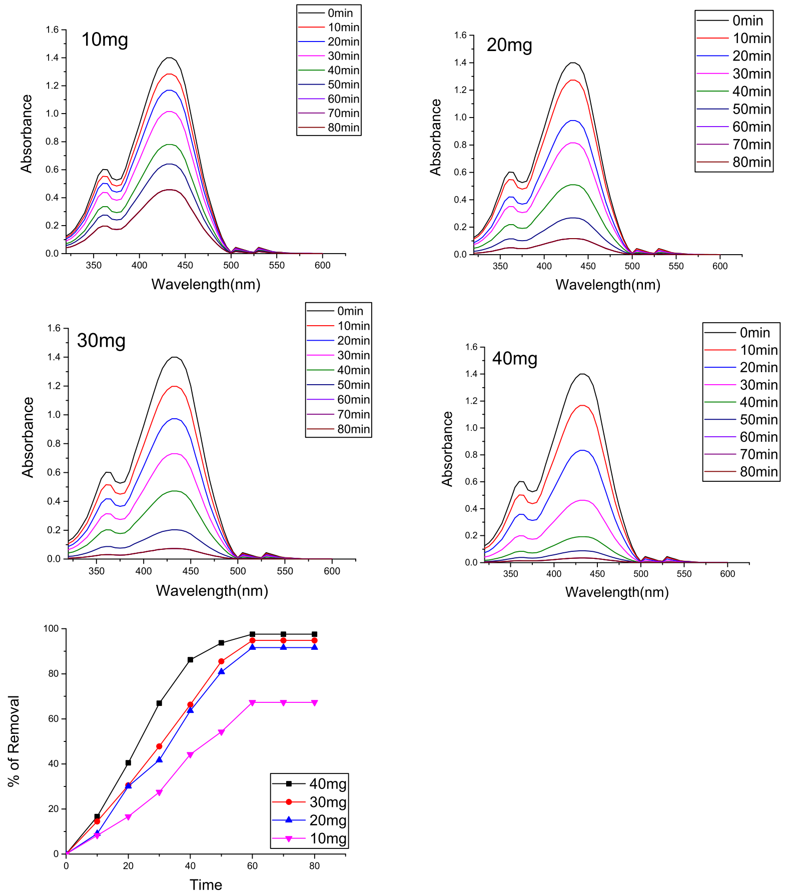
Laboratory experiments were carried out on three different color dye samples. They were used to study the efficiency of prepared MWNTs by Catalytic Chemical Vapor Deposition (CCVD). The three dyes under study were acid scarlet 3R dye (ponceau 2R; sample 1), auramine O yellow (sample 2) and crystal violet 2B (sample 3). These dyes have been chosen due to they enter in many industrial products. The color, chemical formula, molar mass, molecular structure, type of dyes, absorption spectrum and maximum adsorption wave length are mentioned in Table 1. By using HACH LANGE DR3900 laboratory UV–VIS spectrophotometer using silica cells of path length 1 in.

2.2. Methods

The Laboratory experiments were carried out at room temperature of 25 °C. The efficiency of MWNTs was studied under the effect of change of some physical parameters such as pH, contact time, MWNTs with different concentrations and different dosages of dyes on the adsorption. 50 mg of each dye was dissolved in one liter of double distilled water and 2 ml of ethanol as organic solvent, then it was diluted until 10 mg/l solution was obtained according to equation (M_bV_b = M_aV_a). Different quantities of MWNTs were weighed and taken in a standard glass beaker (250 ml) containing 100 ml of sample. The pH of the system was adjusted by using HCl (1 M) and NaOH (1 M) to the desired value in every experiment. Magnetic stirrer was used for mixing sample and adsorbent mixture for predetermined period at 240 rpm. The change of color in dyes was measured at maximum wave length corresponding to maximum absorbance of the sample.

Every experiment was performed such that the initial and final concentrations of dyes were analyzed using UV spectrophotometer. The percentage removal (%R) of dyes was calculated by using the equation $\%R = ((C_0 - C_e)/C_0) \times 100$. The amount of dyes adsorbed onto MWNTs (Q_e (mg/g) adsorption capacity) is calculated by applying the equation $Q_e = ((C_0 - C_e) \times V)/M$ where C_0 and C_e are the initial and final concentrations of the dyes (mg/L), respectively, V is the solution volume in liter and M is the MWNTs dosage (g). calibration curves of dyes.

A) Auramine O yellow Dye:



B) Crystal Violet 2B Dye:

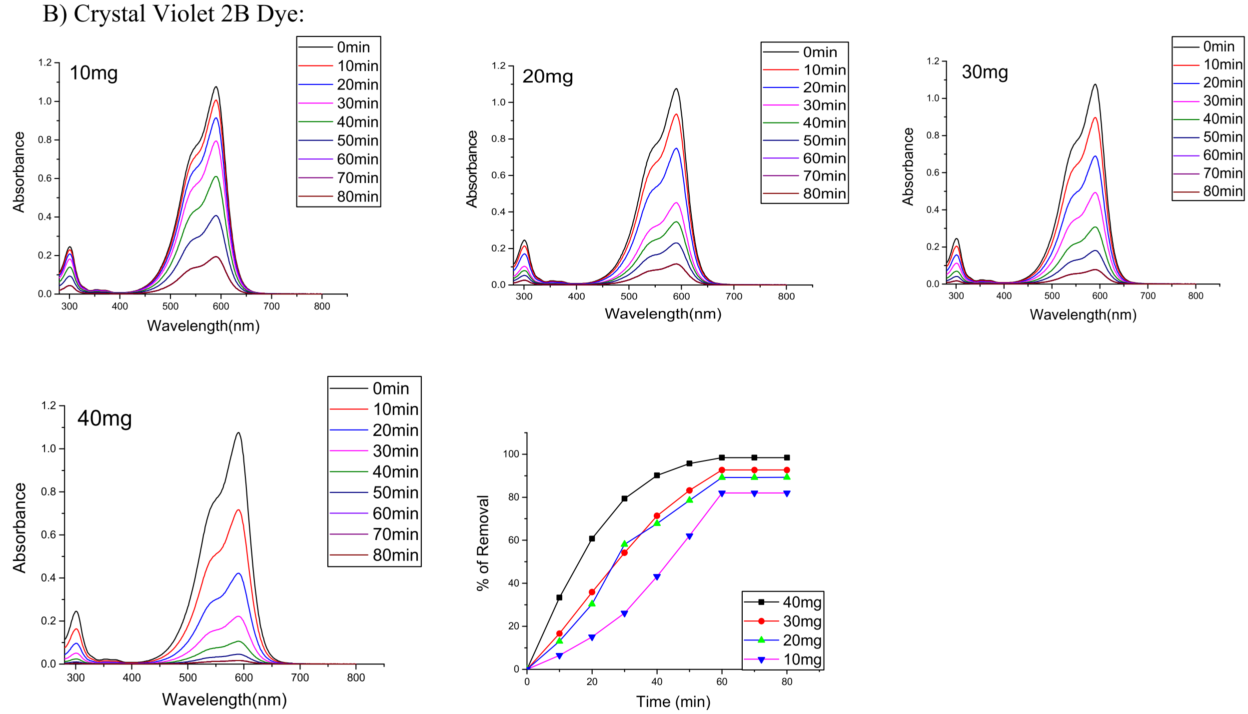


Fig. 5a-c Removal of three dyes using different doses of MWNT through 80 min of constant time, 240 rpm of agitation speed and 10 mg/l of dyes concentration.

C) Brilliant Acid Scarlet 3R dye:

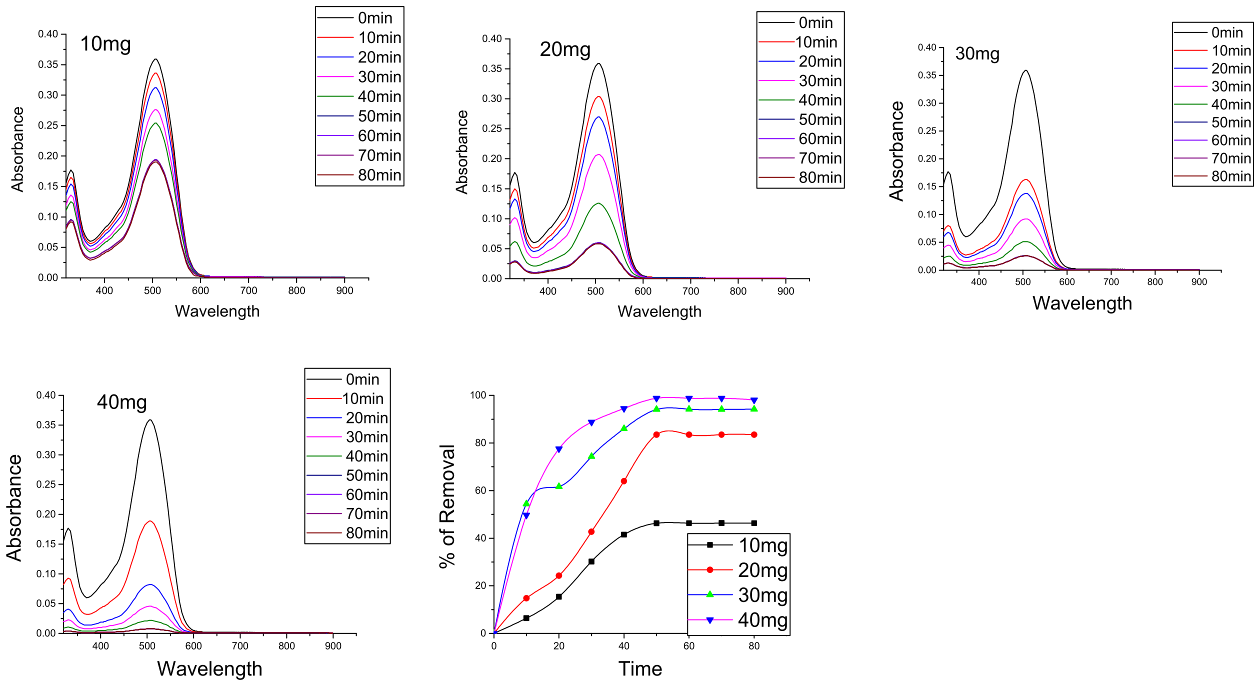


Fig. 5 (*continued*)

2.3. Characterization of MWNTs

The morphology of MWNTs was characterized by using JEOL2100 high resolution transmission electron microscope (TEM) and quanta FE250 scanning electron microscope (SEM) to study distribution of pore size and uniformity of MWNTs surface and Raman analysis is performed using Bruker Senterra Raman spectrometer. Fig. 1

3. Results and discussion

3.1. TEM and SEM microscopic analysis

TEM and SEM images Fig. 2a and c showed the morphology of MWNTs. They showed the crystalline tubular structure of nanotubes. Multi-walled carbon nanotube (MWNTs) had inner diameter (ID) of 5–10 nm, outer diameter 10–50 nm diameter, 5–30 µm length and with purity $\geq$ 90%. TEM and SEM images given in Fig. 2b and d showed MWNTs after adsorption of dyes. A clump of adsorbed dyes was observed over MWNTs surface as can be seen from the images.

3.2. Raman spectral analysis

Raman spectra of MWNTs before and after the adsorption of dyes were shown in Figs. 3a and 3b. This figure showed the comparative intensity of D-band at 1329 cm^{-1} with G-band at 1566 cm^{-1} for MWNTs, which appeared due to acid treatment of purified MWNTs resulted from a defects on the surface of MWNTs due to the attachment of functional groups. Also, the second harmonic 2D-band occurred at 2673 cm^{-1}.

The attachment of functional groups at the surface of MWNTs provided hydrophilic nature to MWNTs (Janas and Stando, 2017). When dyes adsorbed on MWNTs surface, a shift in position of D-band, G-band and 2D-band to higher Raman shift values was observed. Due to the strong attachment of the dyes to MWNTs may be produce the higher shift of the Raman characteristic peaks. This is due to the increase in the elastic constant of the harmonic oscillator of the dyes that adsorbed on MWNTs. OR the van der Waals attraction between the dyes and the graphite sheets of nanotubes may be raise the energy needed for vibrations to occur, which lead to the higher frequency of Raman peaks (Sinani et al., 2005; Hu et al., 2008; Fornasiero et al., 2008; Jain and Sikarwar, 2008).

3.3. BET surface area analysis

The BET surface area of MWNTs was shown in Fig. 4a which showed that it had surface area of 244.86 m^2 /g. Fig. 4b showed that MWNTs had pore size ranged from 1.65 to 1.72 nm and the pore volume was.271 cm^3 /g. So, the higher the surface area the higher the adsorption capacity and efficiency of MWNTs (Li et al., 2003). Fig. 5a

3.4. Effect of adsorbent dose

The quantity of adsorbent in the system is an extensive factor that affects the adsorption rate. The adsorption systems were

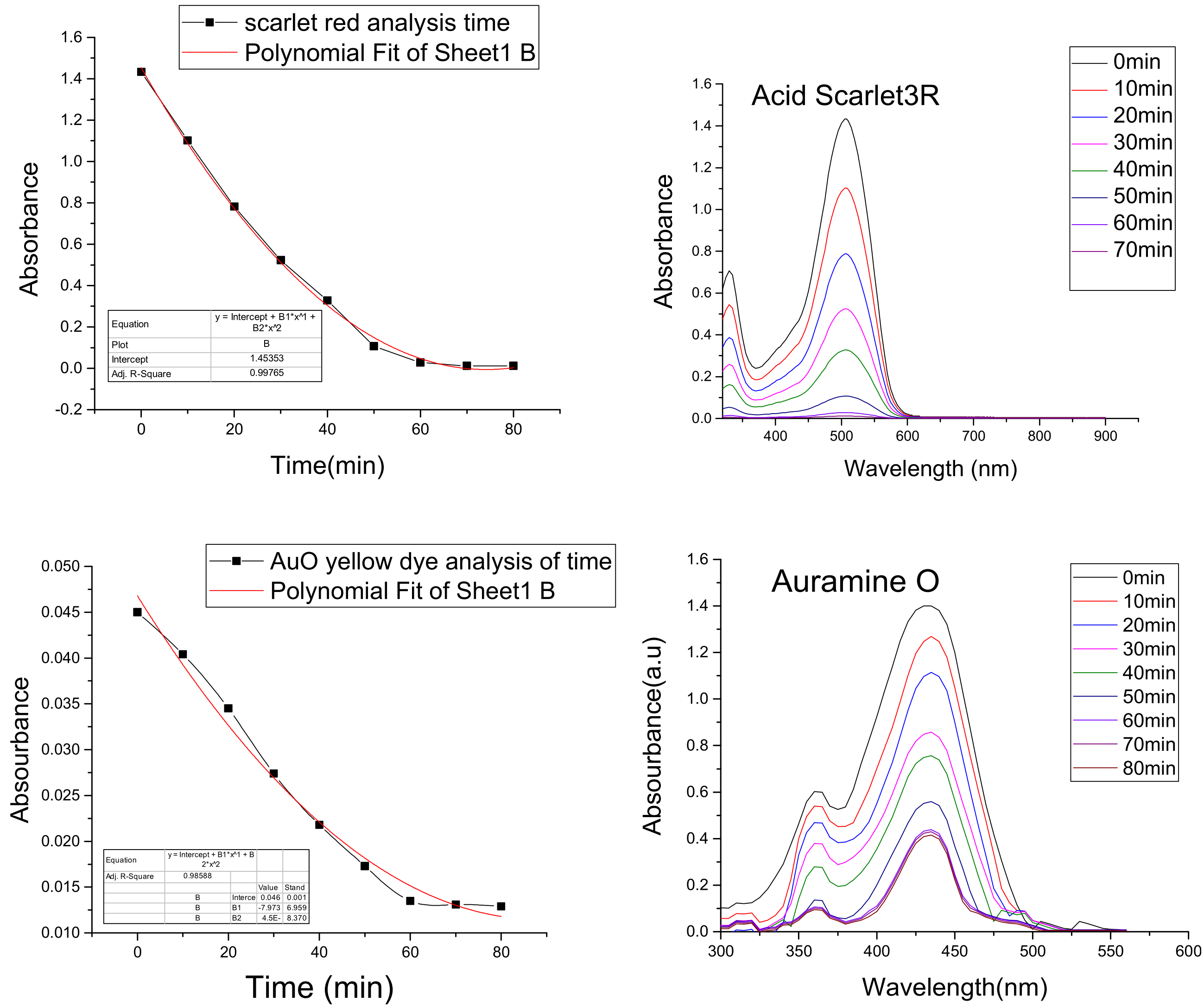


Fig. 6 The effect of contact time on removal of dyes with constant concentration at the presence of constant dose of MWNTs and constant agitation speed at 50 min.

carried out by preparing different concentration of MWNTs ranged from 10 mg to 40 mg and were applied on the three dyes (cationic and anionic dyes). The agitation speed and pH were fixed at 240 rpm and 7, respectively. The results were shown in Fig. 5.

The results showed that the rate of adsorption is directly proportional with the amount of adsorbent in both cationic and anionic dyes (Yang et al., 2019; Krasnov and Basova, 2018). As adsorbent dose increase, the adsorption rate increase. This due to adsorption rate which based on the more possible surface site for adsorption as amount of MWNT increase the surface area increase. So, more space to occupy with dyes (Saleh and Gupta, 2012; Yao et al., 2010; Yao et al., 2011). At 40 mg of MWNTs, the results showed that almost complete removal was observed after 60 min for acid scarlet 3R anionic dye (reach 98%), while removal of auramine O yellow and crystal violet 2B cationic dyes reached 97.2% and 97.6%, respectively.

3.5. Effect of contact time

The adsorption behavior of dyes with the considered adsorbent (MWNTs) as a function of contact time was observed by proliferate the contact time from 10 min to 80 min at a dyes concentration of 10 mg/L, pH of 7, and the dose of MWNT as adsorbent was 40 mg. The agitation speed was fixed at 240 rpm along the experiment. The results presented in Fig. 6 showed that, the removal rate of dyes increase as contact time increase as the absorbance decrease until the adsorption rate reached the equilibrium for adsorbent after 60 min in case of auramine O yellow cationic dye and acid scarlet 3R anionic dye (Covaliu et al., ; Chen et al., 2018). This is due to normal adsorption phenomena. Firstly, the adsorbate molecules diffused from boundary layer film onto the adsorbent surface because of the boundary layer effects, and then they will diffuse into the porous structure of the adsorbent. So, this phenomenon will take a long contact time (Tahermansouri et al., 2015).

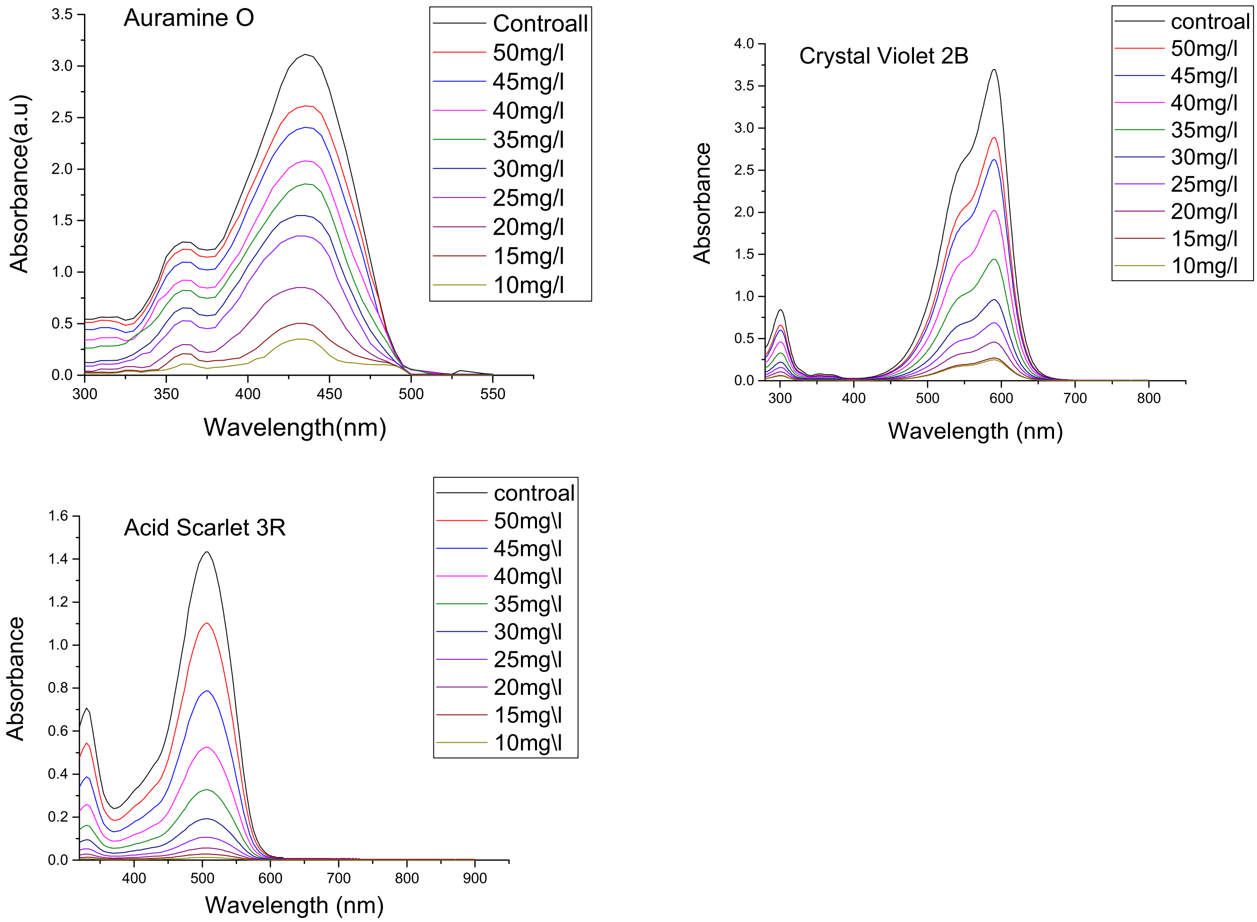


Fig. 7 Effect of dyes concentrations under optimum experimental conditions of pH 7, t = 50 min, MWNTs dose of 40 mg and 240 rpm.

3.6. Effect of initial concentration of dyes

At this experiment, the effect of varying concentrations from 10 mg to 50 mg was studied with respect to adsorbent concentration of 40 mg, pH = 7 and agitation speed of 240 rpm. Fig. 7 showed that the present of removal of dyes decreased as the concentration of dyes increase (Pérez-Ramírez et al., 2019; Khalil et al., 2019). To overcome all mass transfer resistance of the molecules between the aqueous phases (including dyes and H_2O, especially adsorbate dyes) and the solid phase (adsorbent MWNTs), so the initial concentration of auramine O yellow, crystal violet 2B and acid scarlet 3R dyes provided an important driving force to overcome their mass transfer resistance between the aqueous and the solid phases. Therefor the removable of dyes increased as initial concentration decrease (Covaliu et al., ; Shahryari et al., 2010; Ali and Alharbi, 2019; Elzain et al., 2019).

3.7. Effect of pH on dyes removal

In this study, the variation of pH had very important rule that affected the present of removal. To cover the three pH states (acid, base, neutral), so, the work was carried out at pH = 3 (acid medium), pH = 7.56 (7–8, neutral medium) and pH = 9 (basic medium). The other parameters including contact time, dosage of adsorbent and agitation speed were kept constant at 60 min, 40 mg and 240 rpm, respectively.

From the data represented in Fig. 8, it was observed that removal of acid scarlet 3R dye and both cationic dyes (auramine O and crystal violet 2B) was affect with pH variation where maximum removal was obtained at neutral medium (pH = 7–8) for the three dyes under study. The change in pH lead to alternation of surface of adsorbent that affect sorption efficacy. Since the zero potential charge of MWNTs due to it is formation in CVD equals 6.25 (de la Cruz et al., 2012; Dutta et al., 2017), so above that the charge of the surface is positive and below that the charge of the surface is negative in pH range 3–11. So, at pH = 3, for acidic dye (acid scarlet 3R) electrostatic attraction was formed between protonated dye and the surface of MWNTs. While, at pH = 9, the electrostatic repulsion takes place between surface of MWNTs and OH^- and acid scarlet 3R dye. Therefor in both cases of pH = 3 and 9 (acidic and alkaline medium), the removal percent is low. In neutral pH medium (7–8), attraction and repulsion forces were avoided and maximum removal was occurred due to wander vales force. The basic dyes like crystal violet 2B and auramine yellow are also enter between the intestinal of dyes and electrostatic attraction was formed on the surface of dyes (Konicki, 2019; Lawal et al., 2019:). This may be due to difference of H^+ and OH^- concentrations although the zero potential charge of CNT as previously mentioned in literature (Calvete et al., 2010; Cardoso et al., 2011; Machado et al., 2011).Fig. 9.

3.8. Regeneration of MWNTs

Regeneration of the adsorbent (MWNTs) gives it an economic viability. This was carried out by treating the adsorbent with acid and performing the adsorption–desorption experiment. It was found that the percent of removal is slightly changed as seen from Fig. 8. The experiment was tested using auramine yellow and acid scarlet 3R dyes at pH = 7, agitation speed 240 rpm and 10 mg/l of dyes concentration.

The data given in Figure (9) showed that there is slight difference between the original MWNTs and the MWNTs used in dyes adsorption. So, this can give an idea that it can be used more than one time which gives it an economic advantage (Dutta et al., 2017).

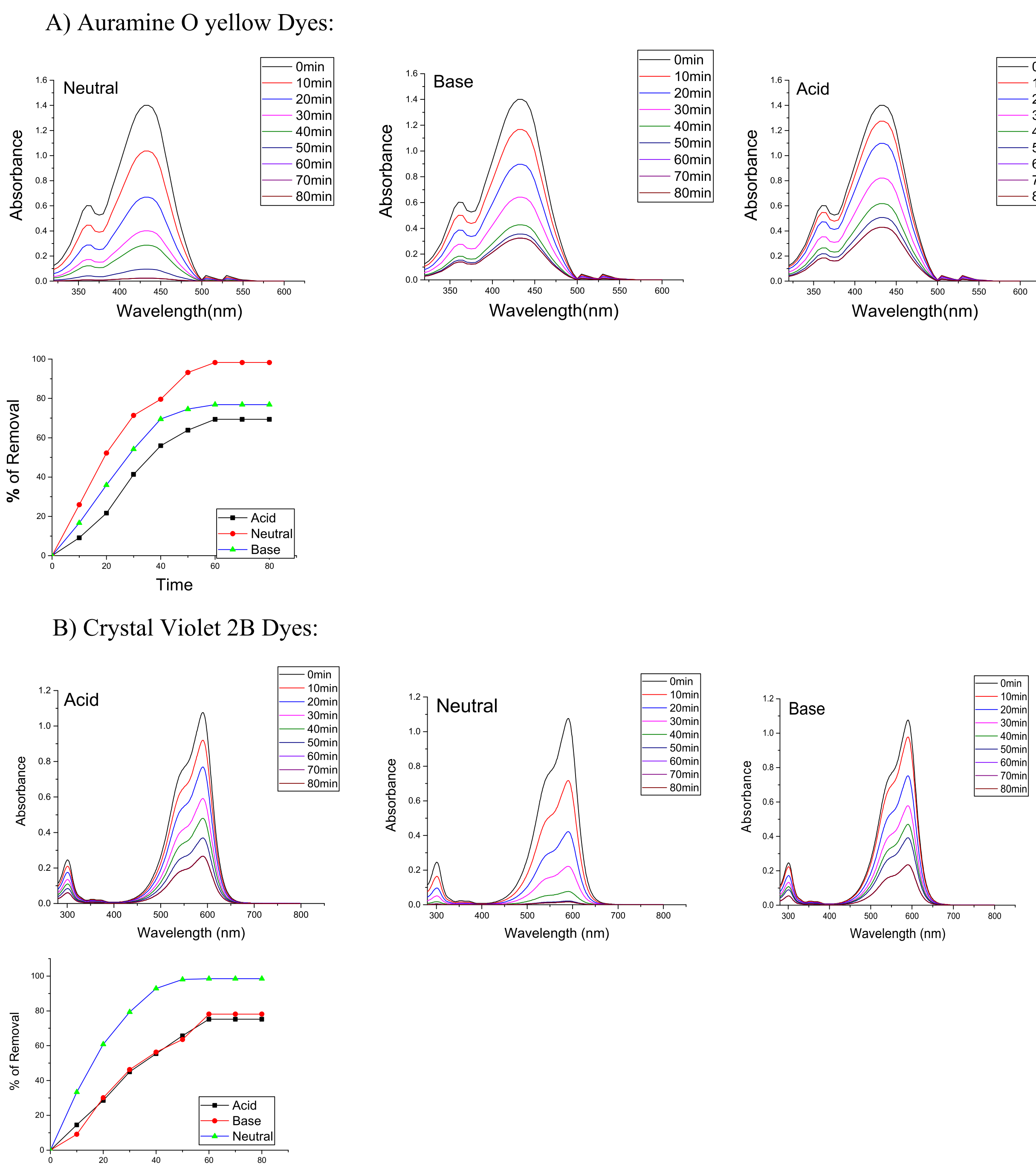


Fig. 8 Effect of pH on dyes removal (a) acid scarlet 3R, (b) auramine O yellow and (c) crystal violet 2B under optimum conditions of initial dyes concentration 10 mg/L, time 60 min, adsorbent doe 40 mg and 240 rpm.

C) Brilliant Acid Scarlet 3R dyes:

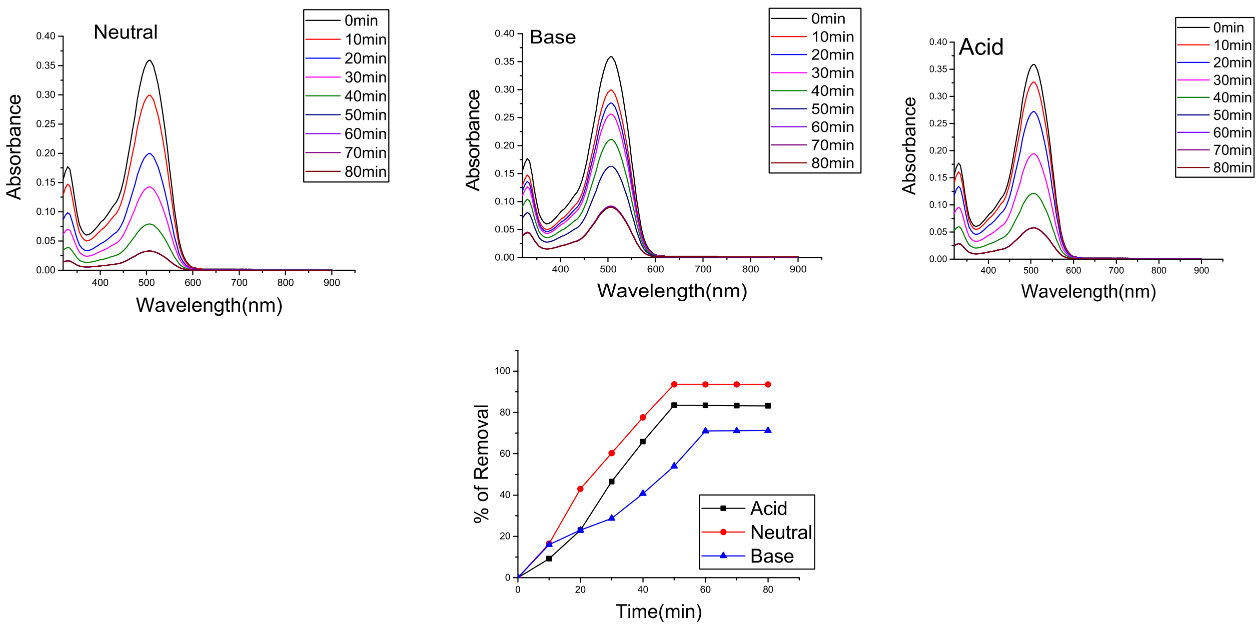


Fig. 8 (*continued*)

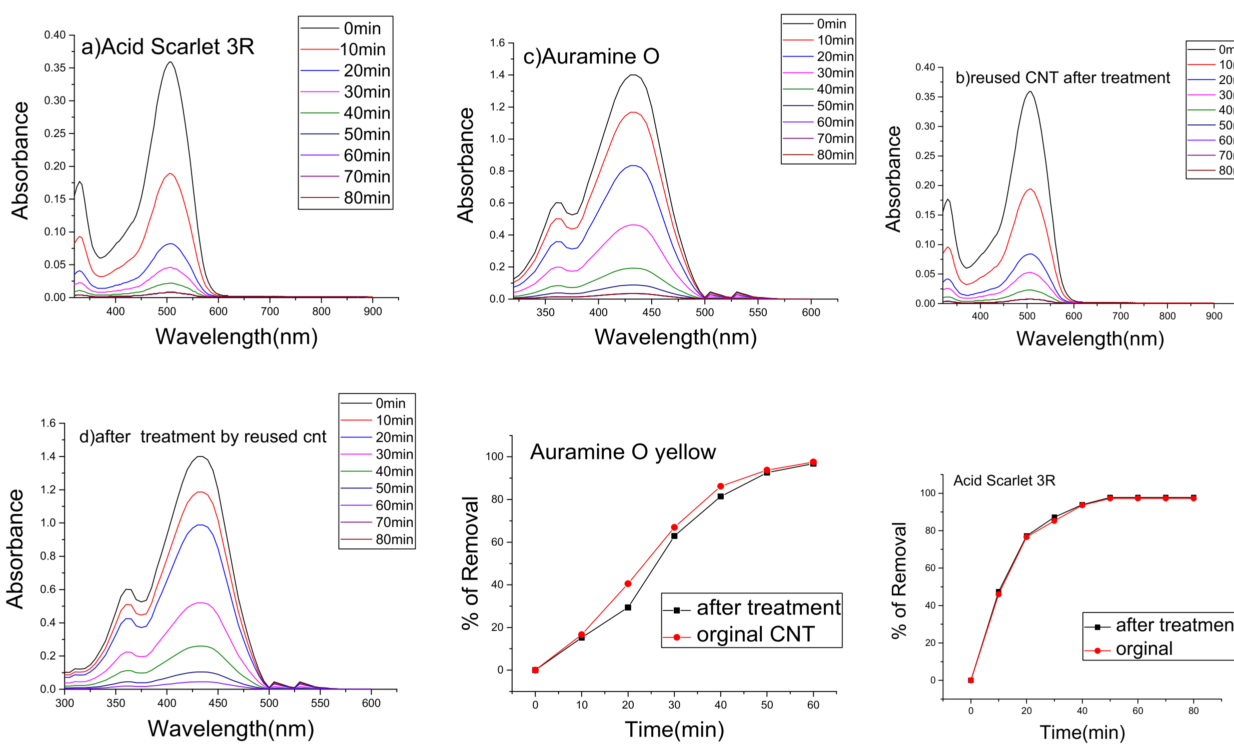


Fig. 9 Regeneration of MWNTs adsorbent.

4. Conclusion

Most of adsorption parameters as concentration of dyes, pH, contact time and adsorbent dose have the effect on removal of different dyes using MWNTs. The optimum conditions to achieved high adsorption capacity at room temperature were contact time 60 min, agitation speed 240 rpm and dose of MWNTs 40 mg. The best removal efficiency of both cationic and anionic dyes was achieved on neutral medium which is useful on applied scale. It was concluded from these results that MWNTs can be considered as highly effective adsorbent for removal of different dyes from waste water and can be reused which has economic viability.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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