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Landfill leachate treatment in an integrated adsorption-chemical oxidation process including CNT and nZVI-H_2O_2

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ABSTRACT

Landfill leachate has created many health and environmental concerns. This paper aimed at examining the efficiency of two batch systems, including multiple carbon nano-tubes (MCNT) as a first reactor and "nano-particles of zero valent iron (nZVI) + H_2O_2" processes as a second reactor, for treatment of very strong landfill leachate with the following physiochemical characteristics: 85,000 mg/L chemical oxygen demand (COD), 20,000 mg/L 5-d biological oxygen demand (BOD_5), 200,000 mg/L total solids (TS), and 15,000 TCU color. Experiments were performed in two series batch reactors and main influencing factors, pH, reaction time, concentration of iron and H_2O_2 were investigated. The results display the high potential of absorbing organic materials in leachate by carbon nano-tubes in the first stage of treatment (58% COD, 82% color and 33% TS are reduced within 10 min). The highest obtained removal efficiencies with this two-stage novel system was 83.6%, 40%, 76.81% , 90.6% for COD, BOD_5, TS and color, respectively. In addition, nZVI + H_2O_2 process in the second stage of treatment can be applied for landfill leachate because it can augment the BOD_5/COD ratio and increase the biodegrability of this type of wastewater.

Keywords: Leachate; Treatment; Carbon nano-tube; nZVI; H_2O_2

1. Introduction

Leachate is generated when a landfill site is operational and even for a long time after its closure. Composition and quantity of leachate depends on several factors, such as the quantity and quality of waste, physical and biochemical processes of waste degradation, topographic location of the landfill, layering system, hydrogeology and type of vegetation in the landfill site [1,2]. More than 200 types of organic compounds in the leachate have been identified. They can be classified in various groups including cyclic hydrocarbons, bicyclic compounds, aromatic hydrocarbons, substituted benzenes, etc. [3]. Furthermore, around 35 compounds have been identified as primary pollutants, involving chloro- and dichlorobenzene, toluene, ethyl benzene and xylenes [3–5].

Leachate can penetrate the soil and reach underground aquifers, causing contamination of soil and groundwater because it contains different contaminants (e.g. hydrocarbons and heavy metals). Moreover, in municipal landfill sites, leachate can move horizontally and be discharged from the soil in low spots, contaminating surface water resources. Because many people use these resources, they are in danger of being infected by different diseases [2–4]. Therefore, proper management of leachate control will minimize the likelihood of groundwater and surface water contamination [3].

Hamadan is a mountainous city located in the west of Iran and produces nearly 750 tons of domestic waste and 5 tons of hospital waste per day. All this waste is transferred to a landfill which is located 23 km away from the city on the road to Tehran (Fig. 1). There is no leachate treatment facility and raw leachate is discharged into the environment. The leachate in Hamadan landfill is about 5 years old. Theoretically, when a landfill is 3–5 years old, a lot of

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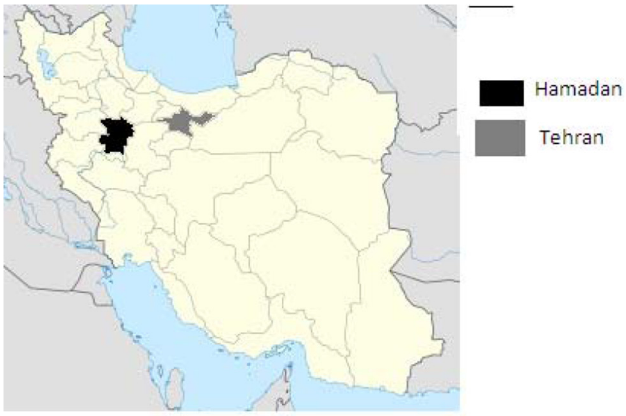

Fig. 1. Location of Hamadan province in Iran.

organic compounds can be found in its leachate, which is obtained through the anaerobic decomposition of waste acidogenes phase [1–5]. Both COD and BOD_5 have very high concentrations, while the pH is very low due to the production a significant amount of volatile fatty acids in this phase. Consequently, the BOD_5 / COD ratio may be more than 0.7 due to biodegradation of organic compounds. Since there are non-biodegradable and toxic substances within the leachate, biological treatment alone cannot eliminate non-biodegradable organic matters. It seems the physical and chemicals processes are necessary as pre-treatment to achieve wastewater treatment standards and complementary purification [1–5].

Different methods can be used for leachate management and treatment such as leachate circulating to landfill cells (in-situ treatment), leachate evaporation, leachate discharge to wastewater treatment plants and leachate treatment by different physicochemical and biological facilities [1–4]. A lot of studies have focused on the landfill leachate treatment by Fenton process as post treatment or pretreatment.

Amor and et al. investigated Fenton and solar photo-Fenton processes for mature leachate remediation. They showed that 63% of COD, 80% of turbidity and 74% of total polyphenols is removed with 2 g L^{-1} of $FeCl_3 \cdot 6H_2O$ at pH 5 [6].

Among the used physicochemical processes, the "nZVI+H_2O_2" process has received a lot of attention in recent years. It is believed to be one of the most effective and economical methods for landfill leachate treatment. Nano-particles have the ability to convert pollutants into less harmful substances because of their small size, high surface area, the crystalline order and unique and highly reactive network [7–11]. Nanoparticles of zero valent Iron (nZVI) are effective for transportation and detoxification a wide range of environmental pollutants, such as chlorinated solvents, oregano Chlorinated pesticides, PCBs, heavy metals, etc. [10–14].

Also there are many reports on the use of carbon nanotubes (CNT_S) as a process of treatment and removal of environmental pollutants [9]. The CNT_S are the hollow ring of carbon atoms that can ne arranged in single-walled or multi-walled forms and also have metallic or semi-conductive properties [15]. Numerous research studies are being conducted to identify the applications of carbon nano-tubes in the water and waste water industry. Nano-tube membranes can remove almost all types of water and wastewater contaminants including metal and heavy metal ions [1], bacteria, viruses, turbidity and organic compounds [16].

Furthermore the adsorption of BPA (bisphenol A) and EE_2 (17α-ethinyl estradiol) from landfill leachate onto single-walled carbon nano-tubes has been studied, with findings demonstrating high removal efficiencies of BPA and EE_2 from landfill leachate using SWCNTs. [15,17–20].

Literature review on leachate treatment shows that the combined processes constitute an effective and economical way for leachate treatment.

Huang et al. demonstrated that a coupled process of coagulation and aerated internal micro-electrolysis (IME) with the in situ addition of H_2O_2 have 79.2%, 79.6%, 81.8% and 90.8% removal efficiencies for COD, total organic carbon, UV254 and color, respectively [21].

In the present study, a novel leachate treatment method is developed using CNT adsorption and nZVI+H_2O_2 process for the first time in the world. This cross-sectional study aimed at investigating the application and effectiveness of carbon nano-tubes and "nZVI+H_2O_2" processes as two sequencing stage treatment systems to reduce COD, BOD_5, TS and color of landfill leachate. Lack of prior research on using CNT adsorption and nZVI+H_2O_2 process prompted this study on the development and modification of a new technology for landfill leachate treatment.

2. Materials and methods

Physiochemical properties of carbon nano-tubes and nZVI are respectively presented in Tables 1 and 2. SEM image of used carbon nano-tubes is presented in Fig. 2. Iron nano-particles were prepared from Plasmachem (GmbH Berlin, Germany). Properties of iron nano-particles and H_2O_2 are presented in Tables 2 and 3. Carbon nano-tubes were purchased from Plasmachem Company (GmbH Berlin, Germany). Properties of used Carbon nano- tubes are presented in Table 1. Other chemicals (e.g. H_2O_2) were purchased from Merck Company. Liquid H_2O_2 with a density of 1.11 kg/L was used in the current study.

About 750 ton of domestic waste and 5 ton of hospital waste enter Hamadan landfill per day. There is not any treatment facility in this place and raw leachate is discharged into the environment without any treatment.

Samples of landfill leachate were collected in bottles made of polyethylene material according to standard procedures and were stored at 4°C [22]. The site of sampling was selected in a way that samples represented whole landfill leachate conditions. Raw leachate was then immediately transferred to the laboratory for analysis. Dilution ratio was determined to characterize the raw leachate. In this regard, ratios of 1/10, 1/50, 1/100, 1/250 and 1/500 were examined. Finally, the ratio of 1/250 was selected as a proper ratio for dilution. All analyses were carried out in accordance with the "standard method" (APHA, 2005) and NCASI instructions.

Chemical oxygen demand (COD), 5-day biochemical oxygen demand (BOD_5) and total solids (TS) were determined according to standard methods [22]. Color was determined by 8025 HACH DR. 2500. Digital BOD bottles

Table 1
Physicochemical properties of used carbon nano-tubes

Appearance	Black powder
Number of walls	3–15
Specific surface area (BET, N_2)	Ca.240 m^2/g
Outer diameter/inner diameter / length	5–20 nm/1–10 µm
Purity(carbon)	>95%
Apparent density	150–350 g/cm^3

Table 2
Physicochemical properties of used iron nano-particles

Properties	Average value
Specific surface area (BET), m^2/g	>12
Average primary particle size, nm	30–60
Particle full range, nm	5–200
Particles, shape	Spherical
Fe-state	Ferromagnetic
Bulk density/cm^3	>0.5
Carbon content, wt%	11–14
–cu	<0.4
–w	<0.2

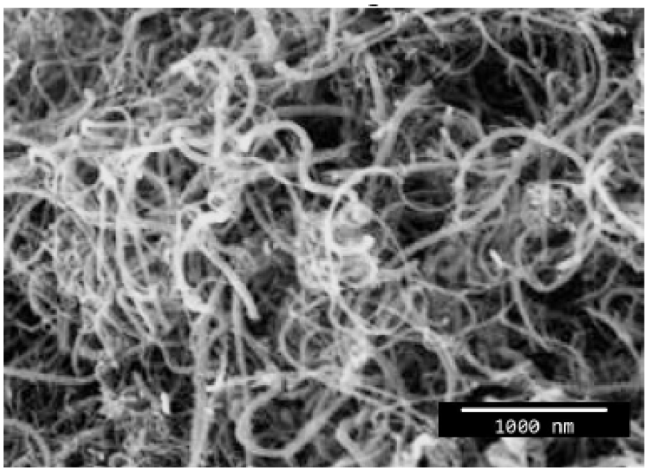

Fig. 2. SEM image of used carbon nanotubes.

and incubator BOD (WTW Germany) were used to measure the BOD_5. America's Hack COD reactor was used to measure the COD concentration. A DR-5000 spectrophotometer (America's Hack Company) was used to measure the color concentration. A digital scale (Sartorius, Germany) with an accuracy of 4×10^{-4} g was used to weigh the substances. A magnetic stirrer (MR Hei-Standard; Heidolph) at set speed and magnet (2.5 cm) was used for mixing. An America's Hack pH meter was used to measure the pH. Heat oven (Iran, Model F.47), vacuum pumps REAX Iran-model 2000, America's Hack Jar Device, Oven (medicine Pars Iran) and Centrifuges 301 Sigma-Germany were used for other

Table 3
Properties of Hamadan landfill raw leachate

Parameter	Range
BOD_5 (mg/L)	20,000
COD (mg/L)	85,000
TS (mg/L)	200,000
Color (TCU)	15,000
pH	6.5
BOD_5/COD	0.23

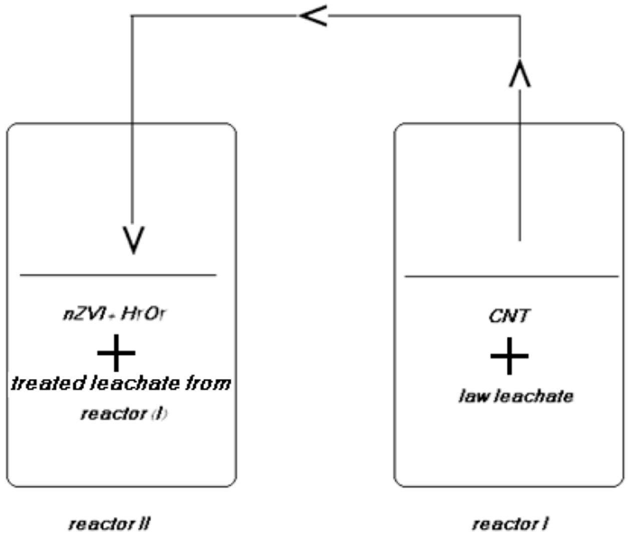

Fig. 3. Schematic of batch experiments.

experiments. The properties of raw leachate are presented in Table 3.

Upon identifying the features of the raw leachate (which is presented in Table 4), coarse particles were removed using a centrifuge to minimize the effect of particles on the oxidation reactions.

Then all experiments were carried out in two separate batch containers as shown in Fig. 2. Carbon nano-tubes and nZVI+H_2O_2 process were used as reactants in the first and second containers respectively. The output of the first container constituted the input for the second container.

The effect of pH in the range 2.5, 4.5, 6.5 and 8.5, contact time in amounts of 10, 20 and 30 min and carbon nano-tube concentrations in 1250, 2500, 4000 and 5000 mg/L was evaluated on the removal efficiency in the first reactor. In order to evaluate the effectiveness and efficiency of each of the factors mentioned in reducing COD, BOD_5, TS, and color by carbon nano-tubes, experiments with different values for each factor was performed while other factors were kept. At last, the final treated leachate in this reactor was used as the input of next reactor.

In the second reactor, nano-particles of zero valent Iron and H_2O_2 were used as reactants. Contact time (in the amount of 10, 20, 30, 40, 60, 80, 90 min), pH (2.5, 4.5, 6.5) concentrations of iron nano-particles (in amounts of 1.25, 2.5 and 5 g/L) and the concentrations of H_2O_2 (in the amount of 14.5, 29.1 and 58.2

Table 4
Physicochemical characteristics of Hamadan landfill treated leachate by two-stage nano systems

Parameter	Properties of law leachate	Optimal efficiency of first reactor	Effluent concentration of first reactor/influent concentration for second reactor	Efficiency of second reactor	Remaining concentration	The total efficiency of the two reactors
COD	85000 mg/L	58.23%	35500 mg/L	60.87%	13940 mg/L	83.6%
BOD_5	20000 mg/L	12.5%	17500 mg/L	30%	12000 mg/L	40%
TS	200000 mg/L	66.87%	66260 mg/L	30%	46380 mg/L	76.81%
Color	15000 TCU	80%	3000 TCU	53%	1410 TCU	90.6%
BOD_5/COD	0.23	–	0.49	–	0.86	–

g/L) were investigated on removal efficiency of the output of first stage. The range of all values for parameters in each process were chosen based on previous studies [14,23].

To increase the reliability and accuracy of experiments, sampling and analysis of samples in each stage was repeated twice and then the results were averaged. The experiments were repeated three times or even four times when the results of the first two tests were not appropriately consistent.

Total number of analyzed samples in this study with regard to the number of samples, frequency, and errors were calculated 292 samples.

Removal efficiency at different stages was calculated through the following equation using the results and the initial concentrations:

$$E = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where C_i and C_f are the initial concentration and final concentration of studied parameters, respectively. Finally, the optimum values of each variable were selected and performance of "nZVI+H_2O_2" carbon nano-tube was evaluated by conducting regression analysis in SPSS 13.

3. Results and discussion

3.1. The effect of contact time variation

The results of One-Way ANOVA showed significant differences for different levels of contact time (for BOD_5, COD, TS and color $p < 0.05$) in both reactors as illustrated in Fig. 4. It means that the removal efficiency of BOD_5, COD, TS and color will be changed by contact time, thus in reactor (I), the changes in BOD_5 increases, while the COD, TS and color reduces. It is obvious that the removal efficiency of COD, TS and color is inversely related to the contact time. The optimal contact time for COD, TS and color is 10 min in reactor (I). The growth in contact time to 20 min or more will decrease the removal efficiency of COD, TS and color, while the BOD_5 removal efficiency increases by rising the contact time to 20 min. This can be attributed to slower reaction rate of BOD_5 than other parameters by chemical removal method (carbon nanotubes). It means that the reaction rate of BOD_5 with chemical method used in this study is slower than that of the COD, TS and color reaction [23]. High removal efficiency of COD, TS and color in a short time (10 min) indicates that carbon nano-tubes are highly capable in terms of the sorption of the organic material in the leachate (58% of COD, 82% of color and 33% of TS are reduced within 10 min), whereas the treatment of biologically degradable organic matters requires more time (30 min) [23]. Similar studies have come to the same conclusion on landfill leachate treatment by CNT [23].

In reactor (II), increasing contact time in the "nZVI+H_2O_2" process increased removal efficiency of BOD_5, COD, color and TS removal efficiency. In this process, increasing the number of collisions with time increases sorption opportunities and so efficient removal of BOD_5, COD, color and TS increased with contact time.

Consequently, the best reaction times were selected for reactor (I) (10 min) and reactor (II) (60 min).

3.2. The effect of pH variation

In reactor (I), pH value plays an important role in the uptake of molecular ions by carbon nano-tubes. When solution pH is above the pH_{PZC} (pH = 8.5), the surface was charged negatively and caused electrostatic interactions that enhance the attraction of cations species (often heavy metals). However, when the solution pH is lower than pH_{PZC} (under acidic conditions), positive charge is produced in a surface of carbon nano-tubes, which improves the process of attracting anionic species; therefore, it can be concluded that negatively charged organic matter has been removed in acidic conditions. Also, at about pH = 6.5, a severe reduction is observed in the absorption of both cationic and anionic species by carbon nano-tubes because the surface charge is neutralized [23]. As shown in Fig. 5, changes in pH alter the removal efficiency of COD, BOD_5, TS and color by carbon nano-tubes in reactor (I). The results of one-way ANOVA for the pH variables showed significant differences ($p < 0.05$) too. The results of this study revealed that the carbon nano-tubes have higher ability to remove organic materials in low acidic pH values. The rise of the pH from 2.5 to 6.5 decreased the removal efficiency of COD, BOD_5, TS and color respectively from 58%, 12.5%, 33.44% and 80% to 3%, 0%, 0% and 23% respectively [23].

The results from nZVI+H_2O_2 process in rector (II) indicate that the removal efficiency of the main factors (BOD_5 and COD) declines with increasing pH from 2.5 to 6.5. The output of one-way ANOVA confirmed these results ($p < 0.05$) for BOD_5, COD, TS and color. In other hands, we can see reduction in BOD_5 and COD removal efficiency

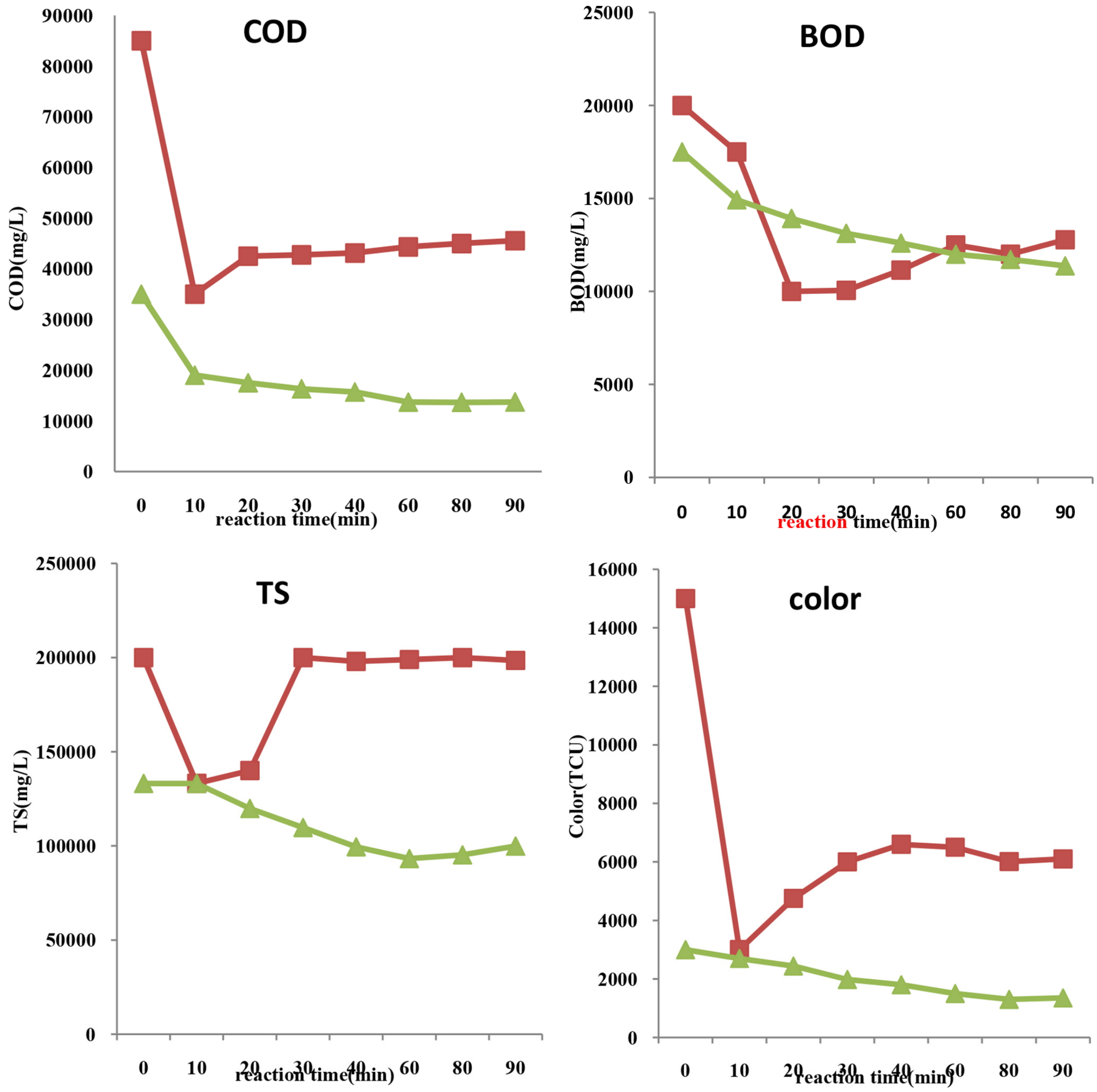

Fig. 4. Effect of contact time variation on the removal efficiency of COD, BOD, TS and color in reactor I (■) (T = 18 ± 1°C, pH = 2.5, the CNT concentration = 2.5 g/L) and in reactor II (▲) (T = 18 ± 1° C, pH = 2.5, the nZVI/ H_2O_2 mass ratio = 0.086).

and increase in TS and the color removal efficiency if pH increases from 2.5 to 6.5 by nZVI + H_2O_2 process. The main factors (BOD_5 and COD) removal efficiency is reduced with increasing pH from 2.5 to 6.5 because the corrosion of iron nano-particles can increase by dropping pH. Additionally, more iron hydroxide is produced in low pH.

Furthermore, it should be noted that oxidation and reduction reactions are performed better at low pH. The Oxidation–Reduction potential at high pH is reduced and so the amount of nZVI added to the reaction will not work properly and will be ineffective.

As observed in Fig. 5 the efficiency of color removal improves as a result of increasing pH. In contrast, the nZVI+H_2O_2 process, which is highly important for COD and BOD5 removal, will be less efficient in the high pH; thus, when pH is high, the efficiency of these factors reduces. Therefore, one can argue that the color removal is not done by absorbing rather than the nZVI+H_2O_2 process. Consequently, more adsorption of color occurs in high pH. Finally, by the importance of COD factor as a main factor, the optimum pH was found to be 2.5 in this study. Similar research projects have indicated that the optimum pH for the nZVI+H_2O_2 process is 2 to 3 [14,24–26]. Consequently, pH = 2.5 was selected as the best pH for both reactor (I) and reactor (II).

3.3. The effect of CNT concentration and nZVI /H_2O_2 ratio variations

In reactor (I), the results of one-way ANOVA showed significant differences for different levels of carbon nano-tubes concentration ($p < 0.05$ for BOD5, COD, TS and color). Accordingly, the concentration of carbon nano-tubes affects the removal efficiency (shown in Fig. 4). The results indicated that removal efficiency of COD, BOD_5, TS and color increased as a result of raising nano-tubes concentration

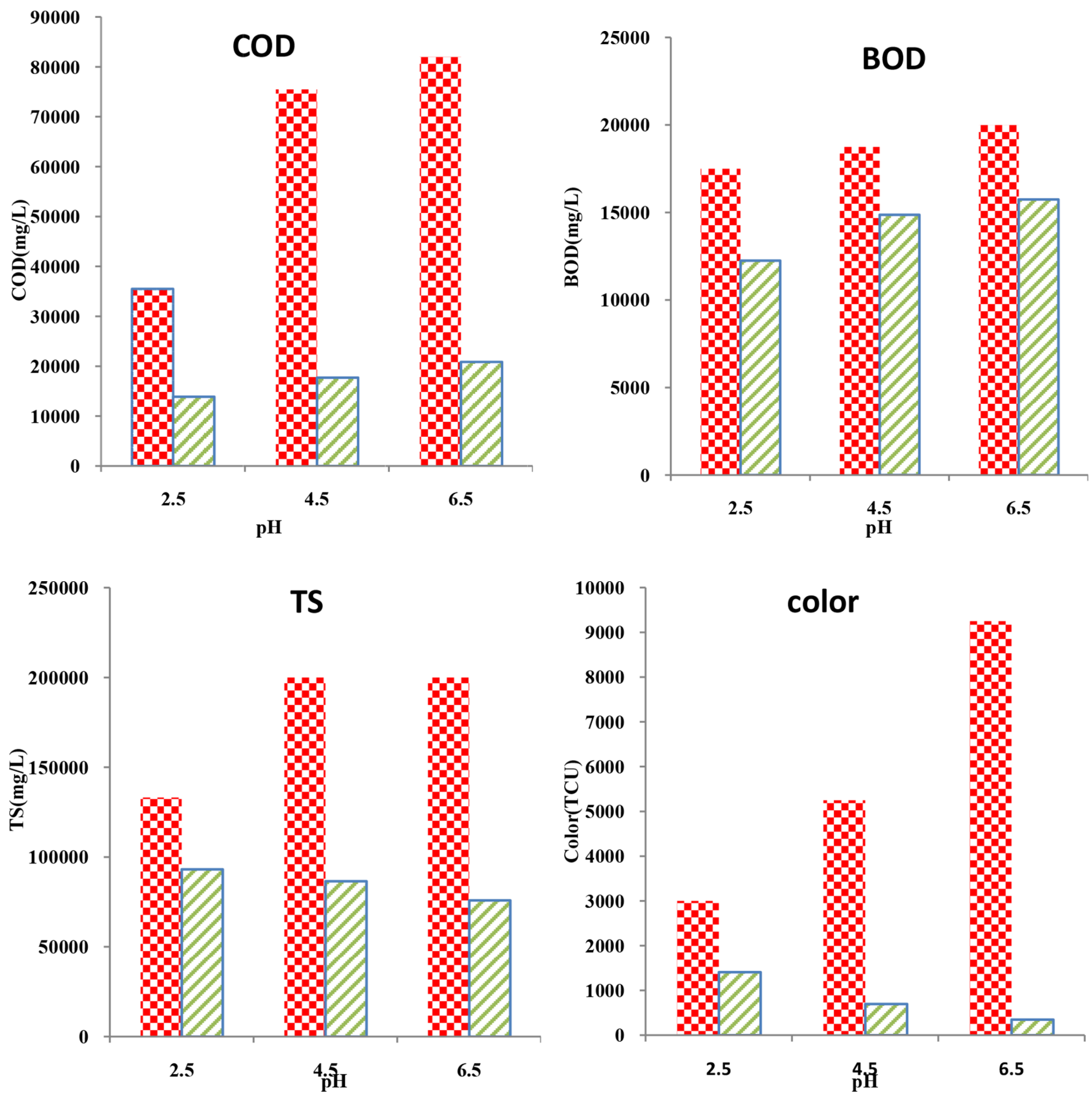

Fig. 5. Effect of pH variation on the removal efficiency of COD, BOD, TS and Color in reactor I ( ) (T = 18 ± 1°C contact time = 10 min, the CNT concentration = 2.5 g/L) and in reactor II ( ) (T = 18 ± 1°C contact time = 60 min, the nZVI/ H_2O_2 mass ratio = 0.086).

from 1250 mg/L to 2500 mg/L. Increasing nano-tubes concentration improves the removal efficiency because it leads to the expansion of the active surface area and so CNT adsorb more of organic and inorganic materials that are present in the leachate [23].

Xiaolei Qu et al. emphasize that the large surface area in CNTs external surface is the key to the high adsorption capacity of CNT. [27].

However, it is important to note that increasing the concentration of carbon nano-tube from 2500 mg/L to 5000 mg/L decreased the removal efficiency of COD, BOD_5, TS and color. These results indicate that the excess ions of carbon nano-tube can cause turbidity in the treated leachate and prevent acts of treatment by making interference in it, hence reducing the efficiency of treatment [18,19,23].

These results are approved by the findings of our previously published study [23]. Also, the adsorption study on the removal efficiency of some other toxic agents (e.g. lead, cadmium and 1, 2-dichlorobenzene) by CNTs have been performed previously, with the results demonstrating that carbon nanotubes are the most reliable and effective adsorbent for removing these toxic materials from aqueous solutions [19,28].

Similarly, in reactor (II), the results of one-way ANOVA showed significant differences for different levels of nZVI / H_2O_2 ratio ($p < 0.05$ for BOD_5, COD, TS and color). It means that the concentrations of H_2O_2 and nZVI are effective on the removal efficiency. In nZVI+H_2O_2 process, removal efficiency of BOD_5, COD, TS and color increased as a result of raising the concentration of nZVI /H_2O_2 to 0.086 g/L. Appropriate results were not obtained for values higher and lower than (0.086). As noted in previous research, the presence of greater or smaller amounts of hydrogen peroxide can change the production quantity of hydroxyl radicals; thus, the COD amount will increase [14,24]. Also, it has

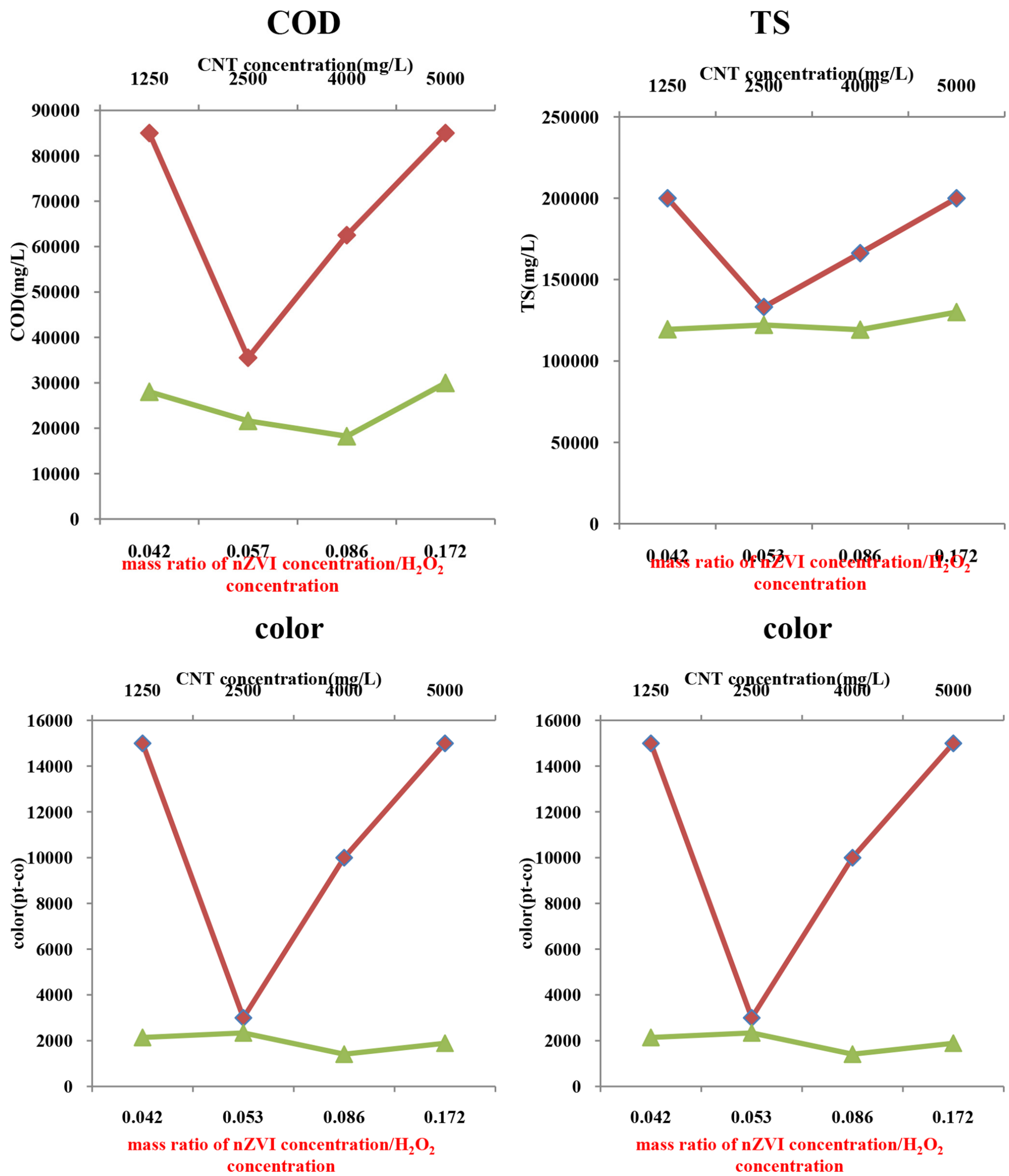

Fig. 6. Effect of CNT concentration and nZVI/H_2O_2 mass ratio variations on the removal efficiency of COD, BOD, TS and Color in reactor I (■) (T = 18 ± 1°C contact time = 10 min, pH = 2.5) and in reactor II (▲) (T = 18 ± 1°C contact time = 60 min, pH = 2.5).

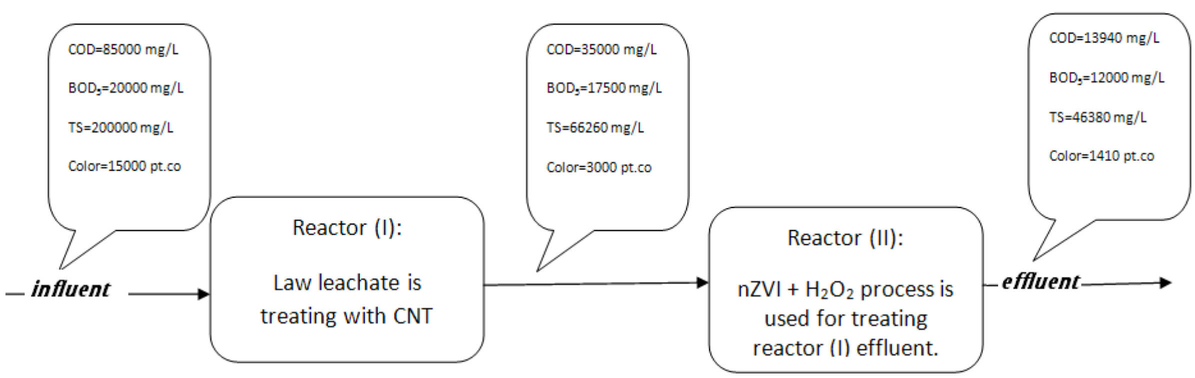

Fig. 7. Schematic of Landfill leachate treatment in two-stage nano reactors; including CNT reactor and "nZVI+H_2O_2" reactor.

been indicated that the greater amount of iron nano-particles can cause turbidity, attack hydroxyl radicals and interfere with treatment, consequently reducing the effectiveness of treatment [14,29]. The obtained results of landfill leachate treatment in the two-stage nano reactors (including CNT reactor and "nZVI+H_2O_2" reactor) are summarized in Table 4 for easier comparison between dates.

4. Conclusions

The present study examined the ability of carbon nano-tubes with combination by nZVI+H_2O_2 process as a novel method for landfill leachate treatment. Leachate was collected from Hamadan landfill in the west of Iran. This study indicates that the use of this innovative method for BOD_5, COD, TS and color removal of Hamadan landfill leachate is influenced by different factors including pH, contact time, concentration of carbon nano-tubes, nZVI /H_2O_2 ratio. The highest obtained removal efficiencies of two-stage system were 83.6%, 40%, 76.81%, 90.6% for COD, BOD_5, TS and color, respectively. The project results showed high potential of absorbing organic materials in leachate by carbon nano-tubes in the first stage of treatment. Also, nZVI+H_2O_2 process can be applied in the second stage of treatment for the landfill leachate because of augmenting the BOD_5/COD ratio and increasing the biodegrability. As a result, it is strongly suggested that a biological process is used as a third and final treatment to get standards for final effluent.

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