




# Greywater Treatment Using Single and Combined Adsorbents for Landscape Irrigation

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## Abstract

Greywater reuse can be considered as an additional supply of water not only to protect water resources but also to reduce water shortage. Thus, the aim of this research is to examine the feasibility of three adsorbents (activated carbon (AC), Iranian natural zeolite (Z) and stabilized nano zero-valent iron (nZVI)) in single and combined forms to treat greywater for reuse in landscape irrigation. For this purpose, greywater samples were collected from student hostel of Fasa University and analyzed in a batch mode experiment. Also, the adsorption of COD was studied in terms of kinetic, isotherm and thermodynamic models. The results indicated that among the seven treatments, the combination of AC, Z and nZVI had the best performance in greywater treatment. 85.75% removal of COD, 91.81% of TDS and 98.1% of turbidity were achieved by triple combined treatment. In double combined adsorbents, the highest adsorption rate of COD, TDS and turbidity was accomplished when AC+nZVI, AC+Z and nZVI+Z were used, respectively. The results also indicate that single adsorbents alone are not adequate to guarantee a sufficient reduction of COD, TDS and turbidity. Kinetics of COD removal by all treatments obeyed to pseudo-second-order model and the Freundlich isotherm closely fitted the experimental data. The COD data demonstrated that the sorption process is spontaneous and endothermic. The results indicated that the triple combined adsorbents were able to reduce the COD, TDS, turbidity and pH level to the required reuse in landscape irrigation and the results were satisfactory according to Iranian standards.

**Keywords** Water shortage · Pseudo-second-order model · Freundlich model · Iranian standards

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## 1 Introduction

Nowadays, water scarcity is one of the most perilous affairs which threatens the world. In 2025, 33% of the world population will live in water scarcity conditions (Alcamo et al. 2000). Industrial, domestic and agricultural demands compete for surface and groundwater resources in many parts of the world, especially in developing countries such as Iran (Amiri et al. 2015). Water pollution has an important role in water scarcity when the freshwater supplies are going to be finished. Moreover, global warming and climate changes are other reasons to put pressure on water resources. However, water scarcity in both quality and quantity is the main problem in semi-arid and arid regions like Iran. Due to these reasons, finding methods which can save water or reuse polluted water is necessary. In this regard, the use of wastewaters can play a significant role in convincing the rising water demands (Amiri et al. 2015)

Domestic wastewater is potentially a local water source which has some special characteristics in terms of solids, organics, and microbiological content. However, domestic wastewater presents several advantages compared to other unconventional waters such as industrial wastewaters and urban storm runoff, including lower heavy metals and toxic substances, and lower treatment cost (Yocum 2006). Domestic wastewaters contain two different parts: black water and greywater. The greywater includes shower, hand washing basin, laundry, washing machine, and kitchen sewage, while toilet sewage is called black water (Eriksson et al. 2002; Wilderer 2004). Kitchen sink water is classified as dark greywater, whereas the wastewater of shower is called light greywater (Friedler and Hadari 2006). Unfortunately, a separation system of black water and greywater does not exist in many countries and it is the main reason that greywater is almost wasted. Furthermore, information on the properties of greywater and black water is still weak and design values are not available (Sievers et al. 2016). In some countries, non-treated greywaters have been used for irrigation of sod and silviculture. Albeit, this type of application can be harmful as it may cause soil salinity (Pedrero et al. 2010). Today, it is proven that treated greywaters can be applied for washing, cleaning, irrigation, industry and many other non-potable uses (United States Environmental Protection Agency (USEPA) 2004).

Nowadays, membrane bioreactor, sand filtration and adsorption, and photocatalysis have been used as the biological, physical and chemical methods, respectively, for greywater treatment (Li et al. 2009; Amiri et al. 2015). Unfortunately, some attempts have been made to treat greywater and reduce the chemical oxygen demand (COD) and total dissolved solids (TDS). However, several researches including bed biofilm reactor (Chripim and Nolasco 2017), trickle media filtration system (Shamabadi et al. 2015), sand filter (Albalawneh and Chang 2015), coarse filter (March et al. 2004), coagulation (Pidou et al. 2008) and phytoremediation (Vymazal 2005) have been performed on greywater treatment.

Adsorption is a common technique in wastewater treatment because of the simplicity, enforceability for multiple reuses, and low cost (Aghakhani et al. 2011; Aghakhani et al. 2012; Amiri et al. 2016). Comparatively, the adsorption technique could be a low cost alternative to available biological and chemical methods for greywater treatment. The organic matter of greywater is commonly evaluated using COD. High COD concentration in greywater could be harmful when it is being used for agriculture and industry (Fatta-Kassinos et al. 2011). Other important indices which describe the quality of water are TDS and turbidity. Consequently, selection of adsorbents with high adsorption capacity of COD, TDS and turbidity is essential.

Activated carbon (AC) is an almost perfect adsorbent because of its high surface area to volume ratio and porosity (Zipf et al. 2016). Low cost of materials used for the production of AC makes it a favorable reagent. It was used for a wide domain of pollutants such as heavy metals and organic compounds (Gharaibeh et al. 1998), desalination of saline and drainage water (Aghakhani et al. 2011; Aghakhani et al. 2012). It is reported that AC is the most efficient material to remove organic pollutants from the aqueous or gas media (Leboda 1993). Natural zeolite (Z) consists of  $\text{SiO}_4$  and  $\text{AlO}_4$  tetrahedrals linked by shared oxygen atoms. It is well-known that zeolite is extensively employed as a natural ion exchanger to eliminate inorganic pollutants from wastewater (Abdul Halim et al. 2010). It has been reported that unmodified zeolite can be useful in the reduction of COD in wastewater treatment (Abdul Halim et al. 2010). Also, zeolite as crystalline aluminosilicates was successfully applied for desalination of drainage water (Aghakhani et al. 2012), removal of organic and inorganic pollutants (Malekian et al. 2011). Nowadays, nanotechnology has been extensively applied in separation and purification processes (Qu et al. 2013). Nano zero-valent iron (nZVI) particle is one of the most attractive adsorbents for ex situ and in situ methods due to the high surface reactivity and surface area (Eslamian et al. 2013). This adsorbent was successfully applied to uptake heavy metals (Amiri et al. 2017; Gil et al. 2018) and chlorinated organic pollutants (Eslamian et al. 2013). Moreover, nZVI has been used successfully as a permeable reactive barrier in removing nitrate from contaminated groundwater (Della Rocca et al. 2007).

The goal of this paper is to investigate the effectiveness and performance of three efficient adsorbents, i.e., AC, Z and nZVI in single and combined forms for the removal of COD, TDS and turbidity from greywater. By using low and high cost materials together, the wastewater treatment cost may be decreased. The influence of key factors including reaction time, initial pH, temperature and initial COD concentration were studied. Furthermore, the adsorption of COD was studied in terms of kinetic, isotherm and thermodynamic models to comprehend information about the interaction between COD and proposed adsorbents. To the best knowledge of the authors, there are no reports about the application of single and combined adsorbents in greywater treatment.

## 2 Materials and Methods

Combined samples were obtained by combination of two or three single adsorbents from AC, Z and nZVI. The list and their symbols are given in Table 1.

### 2.1 Adsorbents

#### 2.1.1 Activated Carbon

The AC studied in the present work (product number 1025141000) was purchased from Merck Co, Darmstadt, Germany.

#### 2.1.2 Zeolite

Iranian clinoptilolite Z was obtained from a mine in Darab, Fars province, Iran. This material was washed by water and was dried at air-dry conditions. This method is useful due to reduction of dust of Z and so its performance becomes better.

### 2.1.3 Nano Zero-Valent Iron

The oxidation and aggregation of the uncoated nZVI in the aqueous solution are the main challenges of its use in separation and purification processes (Eslamian et al. 2013). For this reason, nZVI was immobilized on bone ash as a porous material to overcome the drawback of these fine particles and was synthesized using the sodium borohydride reduction procedure, as reported by Amiri et al. (2017). Some of the physicochemical characteristics of three adsorbents used in this research are presented in Table 2. To identify the surface morphology of AC, Iranian clinoptilolite Z and stabilized nZVI, scanning electron microscopy (SEM, Seron-AIS2100 and TESCAN-Vega 3) was used. Iranian clinoptilolite Z has a rough surface which includes coarse particles with irregular shape and size (see Fig. 1a). Stabilized nZVI has an assemblage of bone ash particles by different pore sizes aggregated with numerous nanoparticles (see Fig. 1b). AC indicated well-developed large pore structures with a honeycomb-like structure (see Fig. 1c).

### 2.2 Adsorbate

Greywater used in this study was obtained from the student hostel of Fasa University, Fars province, Iran. Greywater samples were collected from various sources including kitchen, shower, laundry, washing machines, and hand washing basin wastewaters. Characteristics of raw greywater applied in this study with respect to recent researches are tabulated in Table 3. COD and turbidity of studied greywater were higher compared to the respective values of other researches (see Table 3). The suggested quality standards for application of treated greywater in irrigation, according to World Health Organization (WHO 1989) and United States Environmental Protection Agency (USEPA 2004), are presented in Table 4. As seen in Table 4, the standard values of these parameters are different in various countries. Because large amounts of greywater are allocated to irrigation of landscape, strict standard level of quality parameters for treated greywater are given in Table 4.

### 2.3 Adsorption Studies

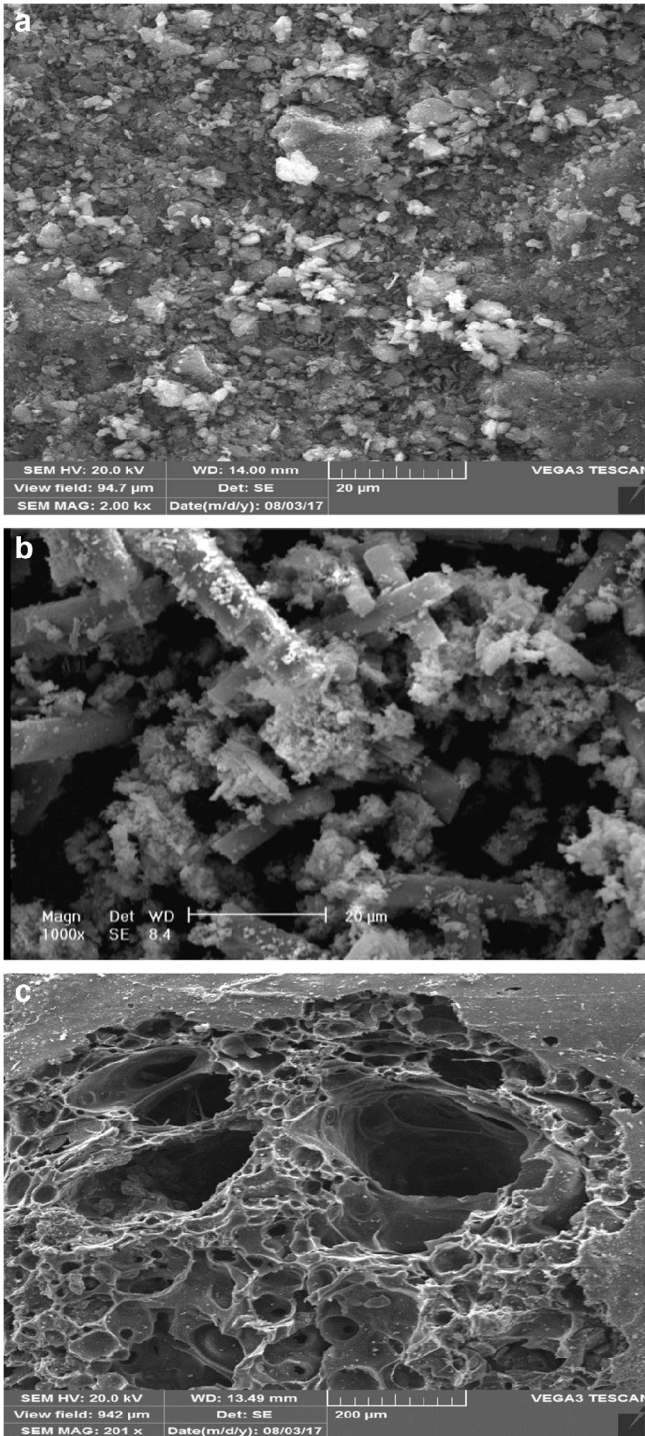
Adsorption studies were performed in a batch reactor as follows: 300 mL of greywater was added into a bottle. Then, the exact amount of adsorbent (0.3 g in single form adsorbents, 0.15 g in two combined adsorbents and 0.1 g in three combined adsorbents) was added to each bottle. After that, bottles were shaken at 180 rpm (rpm) at room temperature ( $25 \pm 2$  °C). The required time in equilibrium condition was obtained from

**Table 1** Single and combined adsorbents used in this research

Adsorbent	Zeolite	Activated carbon	Nano zero valent iron	Zeolite + Activated carbon	Zeolite + Nano zero valent iron	Activated carbon + Nano zero valent iron	Zeolite + Activated carbon + nano zero valent iron
Symbols	Z	AC	nZVI	AC+Z	nZVI+Z	AC+nZVI	AC+nZVI+Z

**Table 2** Physicochemical characteristics of AC, Z and stabilized nZVI

Index	AC	Index	Z	Index	Stabilized nZVI
C (%w)	65	SiO <sub>2</sub> (%)	67.45	Ca (%w)	24.2
Surface area	1000 (m <sup>2</sup> g <sup>-1</sup> )	Al <sub>2</sub> O <sub>3</sub> (%)	11.65	P (%w)	14.1
Micropore volume (cm <sup>3</sup> g <sup>-1</sup> )	0.52	CEC	435 mmol kg <sup>-1</sup>	Fe (%w)	18.9
Mesopore volume (cm <sup>3</sup> g <sup>-1</sup> )	0.36	Average pore size	19.4 nm	Surface area	108.9 (m <sup>2</sup> g <sup>-1</sup> )
Bulk density (g cm <sup>-3</sup> )	2.1	Surface area	16 (m <sup>2</sup> g <sup>-1</sup> )	Average pore size	11.02 (nm)
Particle size	< 100 μ	Particle size	0.25–2 nm	Pore volume (cm <sup>3</sup> g <sup>-1</sup> )	0.31



**Fig. 1** SEM images of (a) Iranian clinoptilolite zeolite (b) stabilized OBA/nZVI (c) activated carbon

initial kinetic tests. The experiment kinetic data of COD show no significant changes in adsorption after 4 h. At the end of every experiment, the adsorbents were filtered from the treated greywater by a Whatman filter paper No. 41. All experiments were performed in three replications and the average of data was recorded as the final result. The ratio of COD reduction was measured by a spectrophotometer using a Unico 2100 instrument. The adsorption capacity ( $q$ ) ( $\text{mg g}^{-1}$ ) and adsorption efficiency ( $R$ ) (%) of COD using AC, Z and nZVI were computed by Eqs. (1) and (2), respectively:

$$q = \frac{(COD_o - COD_f)}{m} \times V \quad (1)$$

$$R = \frac{(COD_o - COD_f)}{COD_o} \times 100 \quad (2)$$

where  $COD_o$  is the initial concentration of COD and  $COD_f$  is the COD after the adsorption ( $\text{mg L}^{-1}$ ).  $V$  is the solution volume (L) and  $m$  is the adsorbent mass (g). Turbidity was measured by turbidity meter (LaMotte 2020we). Also, TDS was measured by TDS meter (HM digital).

## 2.4 Modeling Study

To evaluate the performance of AC, Z and nZVI as efficient materials in the design of adsorption systems, the adsorption of COD was studied in terms of isotherms, thermodynamics, and kinetics. Consequently, the kinetic data of COD were analyzed using intra-particle diffusion, pseudo-first-order and pseudo-second-order models, and equilibrium data were also evaluated by Langmuir (L), Freundlich (F) and Langmuir-Freundlich (L-F) models. Furthermore, the influence of temperature (5, 25 and 50 °C) on COD adsorption was investigated, and associated thermodynamic parameters such as standard enthalpy ( $\Delta H^\circ$ ), standard entropy ( $\Delta S^\circ$ ) and standard free energy ( $\Delta G^\circ$ ) were also determined. In our previous works (Amiri et al. 2016; Gil et al. 2018), isotherm kinetic

**Table 3** Comparison of studied raw greywater parameters with that used in other studies

Parameter	This study	Zipf et al. (2016)	Shamabadi et al. (2015)	Chrispim and Nolasco (2017)	Sievers et al. (2016)
	Bathroom sinks, showers, hand basins, and kitchen sink	Lavatory sinks in a university campus	Bath/shower	Showers	Household greywater
COD ( $\text{mg L}^{-1}$ )	180–550	145.8 ± 79.1	291	272.8	746
BOD <sub>5</sub> ( $\text{mg L}^{-1}$ )	129–360	56.0 ± 15.9	129	123.1	460
TDS ( $\text{mg L}^{-1}$ )	1800–2800	–	–	–	–
Turbidity (NTU)	54–280	35.8 ± 45.1	55	100	–
Fecal coliforms (NMP/100 mL)	None detected	–	–	–	–
EC ( $\mu\text{S/m}$ )	921–1550	–	–	–	–
pH	6.5–8.26	7.7 ± 0.6	6.3	–	–



**Table 4** The required degree of treatment and quality standards for application in irrigation

Applying for	Recommended by	Permitted limits						
		COD (mg L <sup>-1</sup> )	BOD (mg L <sup>-1</sup> )	TDS (mg L <sup>-1</sup> )	pH	Turbidity (NTU)	Coliforms (CFU/100 mL <sup>-1</sup> )	
For restricted irrigation	Iranian Standards (Shamabadi et al. 2015)	150	30	< 1000	6.5–8.5	–	1000	
For unrestricted irrigation	Iranian Standards (Shamabadi et al. 2015)	30	10	450	6.5–8.5	10	400	
Irrigation of ornamental fruit trees and fodder crops	WHO ( 1989)	–	240	–	–	–	1000	
Irrigation of vegetables likely to be eaten uncooked	WHO ( 1989)	–	20	–	–	–	200	
Irrigation for public areas and food crops which will not be commercially processed and any crop eaten raw	USEPA ( 2004)	–	10	–	6–9	2	0	
Irrigation of restricted areas and food crops that will be commercially processed, non-food crops and pastures	USEPA ( 2004)	–	30	–	6–9	–	100	
a: Cooked vegetables, parking areas, playgrounds and side of roads inside cities	Jordanian Standard (WHO 2006)	100 <sup>a</sup> and 500 <sup>b,c</sup>	30 <sup>a</sup> , 200 <sup>b</sup> and 300 <sup>c</sup>	1500	6–9	10	100 <sup>a</sup> and 1000 <sup>b</sup>	
b: Plenteous trees and green areas, side of roads outside cities	Jordanian Standard (WHO 2006)	150	60	–	6–9	–	1000	
c: Field crops, industrial crops and forestry	Jordanian Standard (WHO 2006)	50	15	–	6–9	2	< 2.2	
Discharge to streams and storage	Tunisian Standards (WHO 2006)	90	30	–	6.5–8.5	–	–	
Groundwater recharge	Saudi Arabia Standards (WHO 2006)	–	40	2000	6–8.5	–	–	
Irrigation of restricted areas								
Irrigation of restricted areas								



and thermodynamic parameters were explained in detail. In Table 5, a summary of the investigated models is presented.

### 2.5 Evaluation Criteria

The goodness of fit between model estimation and experimental data was judged using the standard error of estimate (SEE) and coefficient of determination (R<sup>2</sup>). Eqs. (3) and (4) show the respective formulas (Amiri et al. 2013).

$$SEE = 1 - \sqrt{\frac{\sum_{i=1}^n (COD_m - COD_p)^2}{n-2}} \tag{3}$$

$$R^2 = \frac{\left[ \sum_{i=1}^n (COD_m - \overline{COD}_m) (COD_p - \overline{COD}_p) \right]^2}{\sum_{i=1}^n (COD_m - \overline{COD}_m)^2 (COD_p - \overline{COD}_p)^2} \tag{4}$$

where COD<sub>m</sub> and COD<sub>p</sub> are the measured and predicted values of COD, respectively.  $\overline{COD}_m$  and  $\overline{COD}_p$  are the average of COD<sub>m</sub> and COD<sub>p</sub>, respectively, and *n* is the total numbers of measurements.

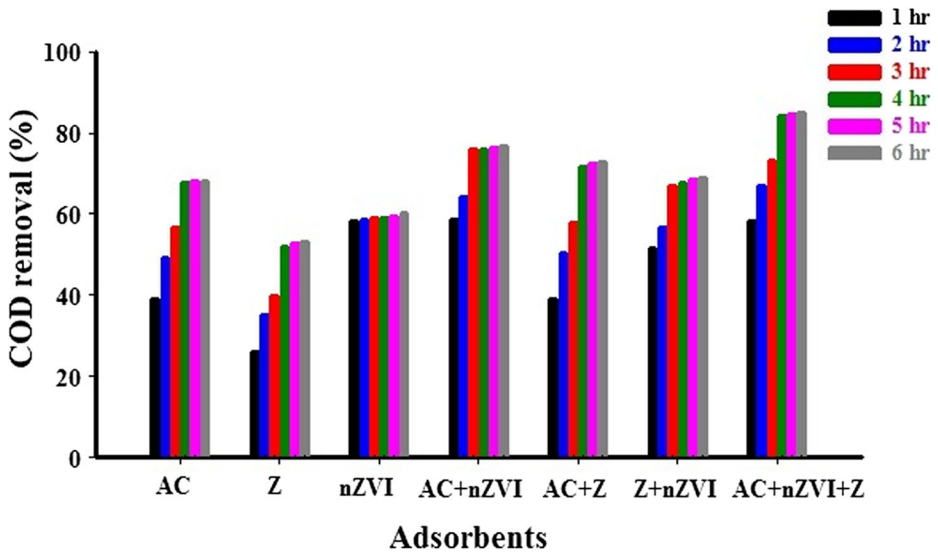
## 3 Results and Discussion

### 3.1 COD

The results of COD removal as a function of time using various treatments at initial COD concentration of 350 mg L<sup>-1</sup> are shown in Fig. 2. As seen in this Figure, the triple combined

**Table 5** Isotherm, kinetic and thermodynamic models used in this work

Model	Equation	Pparameter and dimension
<b>Kinetic models</b>		
Pseudo-first-order (Lagergem)	$q_t = q_e(1 - \exp(-k_f t))$	$K_f$ (1/min) $q_e, q_t$ (mg/g)
Pseudo-second-order (Ho)	$q_t = \frac{k_s q_e^2 t}{1 + q_e k_s t}$	$k_s$ (mg/g.min) $t$ : time (min)
Intra-particle diffusion	$q_t = k_{int} t^{0.5}$	$k_{int}$ (mg g <sup>-1</sup> min <sup>-0.5</sup> ), $t$ (min)
<b>Isotherm models</b>		
Langmuir (L)	$q_e = \frac{b q_{max} C_e}{1 + b C_e}$	$q_{max}$ (mg/g) $b$ (L/mg)
Freundlich (F)	$q_e = K_F C_e^{\frac{1}{n}}$	$K_F$ (mg/g)(mg/L) <sup>-n</sup> $n$ : model exponent (-)
Langmuir-Freundlich (L-F)	$q_e = \frac{q_{max} (b C_e)^n}{1 + (b C_e)^n}$	$n(-)$ (0 < $n$ < 1)
<b>Thermodynamic equations</b>		
$\Delta S^\circ, \Delta H^\circ$	$\ln k_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT}$	$R$ (314 / 8 J / mol k)
$k_d$	$k_d = \frac{q_e}{C_e}$	$K_d$ (l/g)
$\Delta G^\circ$	$\Delta G^\circ = -RT \ln k_d$	$T$ (k)



**Fig. 2** COD removal (%) by using single and combined adsorbents of AC, Z and nZVI (temperature,  $25 \pm 1$  °C; Initial COD concentration,  $350 \text{ mg L}^{-1}$ ; pH, natural, adsorbent dose,  $0.3 \text{ g}$ )

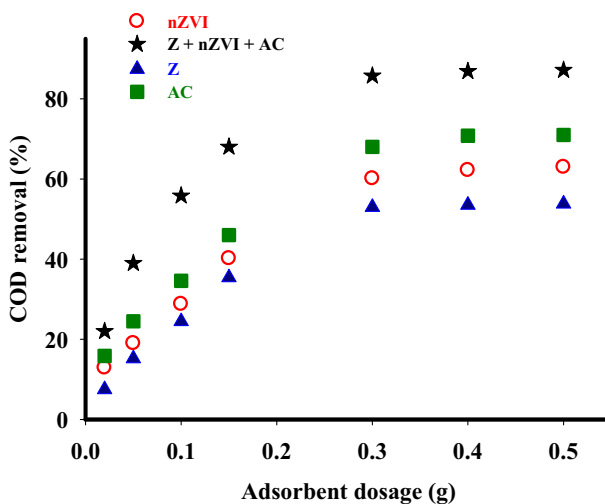
adsorbents uptake more than 85% of COD after 6 h and were the best. With double adsorbents, the percent removals of COD were 76.88% by AC+nZVI, 72.59% by AC+Z and 69% by nZVI+Z, which ranked in a second, third and fourth places, respectively. AC alone adsorbed more than 68% of COD. Indeed, the high potential of AC to remove COD from greywater may be attributed to the hydrophobic surface, a broad range of pore size distribution and great surface area (see Table 2) (Abdul Halim et al. 2010). nZVI and Iranian Z adsorbed more than 60.14% and 52.98% of COD, respectively. In fact, nZVI acts as electron donor for degradation of organic pollutants and is a powerful reducing agent for oxidation of COD (Jagadevan et al. 2012). The shell of nZVI includes FeO and the core of it comprises Fe<sup>0</sup> which is effective in the mechanism of nZVI (Eslamian et al. 2013). The lowest adsorption of COD happened when zeolite was used. Similar results were observed by Abdul Halim et al. (2010). The order of COD elimination in various treatments was: AC + nZVI+Z > AC+nZVI > AC+Z > nZVI+Z > AC > nZVI > Z. Itayama et al. (2004) reported 85% reduction of COD from domestic greywater by using a slanted soil filter.

The treated greywater using all treatments, except Iranian Z, was appropriate for applying in restricted irrigation of landscape (see Table 4), where the residual COD in treated greywater was lower than  $150 \text{ mg L}^{-1}$ . None of the single and double combined adsorbents were effective in treating greywaters in a permissible level of COD for groundwater recharge ( $< 50 \text{ mg L}^{-1}$ ) and reuse for restricted irrigation according to Tunisian standards ( $< 90 \text{ mg L}^{-1}$ ) (see Table 4). Accordingly, the performance of triple combined adsorbents was acceptable, for which the COD residual reached  $49.87 \text{ mg L}^{-1}$  ( $< 50 \text{ mg L}^{-1}$ ). The treated greywater by triple combined adsorbents is appropriate for restricted irrigation of landscape according to the standards of various countries and can be discharged into surface water and groundwater (see Table 4). Therefore, the results indicate that single adsorbents alone are not adequate to guarantee a sufficient reduction of COD.

As seen in Fig. 2, the removal percentage of COD increases with contact time, and approaches equilibrium after 4 h for AC, Z, AC+Z and AC+nZVI+Z treatments. 99.3%,

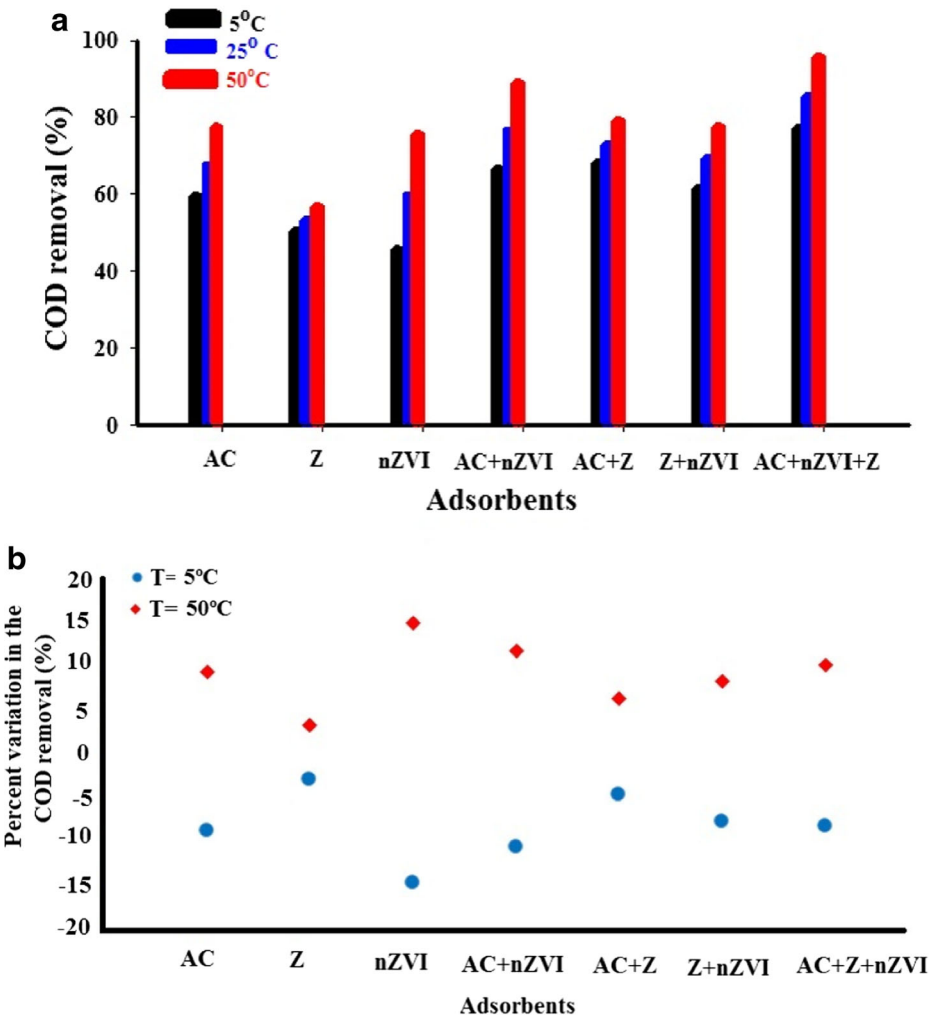
98.11%, 98.76% and 98.64% of initial COD concentration was removed by using AC, Z, AC+Z and AC+nZVI+Z treatments, respectively, within 4 h of agitation and mixing. However, the performance of AC+nZVI+Z treatment was considerable within 3 h of agitation, after which 85.76% of initial COD concentration was removed. Two treatments, including AC+nZVI and nZVI+Z adsorbed more than 98.7% and 96.49% of initial COD concentration, respectively, within 3 h of contact time. It is important to notice that all treatments comprising nZVI removed COD at a lower contact time, i.e., the majority of COD (97.72%) was eliminated within the first 1 h of mixing with the nZVI. In each treatment which includes AC, the removal percentage of COD was significantly increased. Therefore, the AC was more efficient in improving the adsorption capacity ( $\text{mg g}^{-1}$ ) of double and triple combined adsorbents, whereas the nZVI was more effective in improving the kinetic behavior on COD adsorption by using double and triple combined adsorbents.

The adsorption behavior of COD by double and triple combined adsorbents can be explained in terms of adsorbent dosage (see Fig. 3). Indeed, the relation between the removal percentage of COD by Z, nZVI, AC and AC+nZVI+Z against adsorbent dosage is non-linear (polynomial or logarithmic), which results in an increase in the adsorbed amount of COD in combined adsorbents compared to single adsorbents. The removal percentages of COD by Z, nZVI, and AC were 24.46%, 28.79% and 34.6%, respectively, when 0.1 g of adsorbent was used. Also, adsorption efficiency of COD by AC+Z was 85.75%, when 0.1 g of each adsorbent was used. It was found that there was good agreement between obtained adsorption efficiency for AC+nZVI+Z (85.75%) and summation of adsorption efficiencies of Z, nZVI and AC ( $24.46 + 28.79 + 34.6 = 87.85\%$ ), which confirms the non-linearity assumption between the removal percentage of COD and adsorbent dosage. It is apparent that there is no evident change in percentage adsorption of COD after 0.3 g of adsorbent dosage due to achieving the equilibrium adsorption capacity (Amiri et al. 2016).



**Fig. 3** COD removal (%) as a function of adsorbent dosage (temperature,  $25 \pm 1$  °C; Initial COD concentration,  $350 \text{ mg L}^{-1}$ ; pH, natural; contact time, 6 h)

In order to determine the influence of temperature on removal percentage of COD, 0.3 g of adsorbents was added into 300 mL of  $350 \text{ mg L}^{-1}$  COD solution at three levels of temperature (5, 25 and 50 °C) without any changes on the solution pH. A rising in temperature of COD solutions from 5 °C to 25 °C and 50 °C caused an increase in the COD removal (%) for all adsorbents (see Fig. 4a), indicating the thermodynamically desirable adsorption process. This may be due to the formation of new active sites or enlargement of pore size on the adsorbents surface, thus resulting in higher permeation in the adsorbents (Amiri et al. 2016). Highest increase of percent variation in COD removal occurred for temperature change from 5° to 50 °C for nZVI (about 29.91%), whereas the lowest increase occurred for Z (6.2%) (see Fig. 4b); the order was nZVI > AC+nZVI > AC+nZVI+Z > AC > nZVI+Z > AC+Z > Z (see Fig. 4b). In order to a better comprehend the temperature influence at different ranges on the



**Fig. 4** a COD removal (%) as a function of temperature (b) The percentage of variation in COD removal (%) for T = 5 °C and T = 50° (Initial COD concentration,  $350 \text{ mg L}^{-1}$ ; pH, natural, adsorbent dose, 0.3 g; contact time, 6 h)

**Table 6** Thermodynamic parameters for COD removal by different treatments

Adsorbent	$\Delta H^\circ$ (kJ mol <sup>-1</sup> )	$\Delta S^\circ$ (J mol <sup>-1</sup> K <sup>-1</sup> )	T (K)	$\Delta G^\circ$ (kJ mol <sup>-1</sup> )
AC	14.33	54.6	278	-0.848
			298	-1.94
			323	-3.31
Z	4.14	14.93	278	-0.011
			298	-0.309
			323	-0.682
nZVI	21.59	76.08	278	0.439
			298	-1.081
			323	-2.983
AC+nZVI	23.27	88.94	278	-1.45
			298	-3.23
			323	-5.45
AC+Z	9.57	40.56	278	-1.71
			298	-2.51
			323	-3.53
nZVI+Z	12.89	50.13	278	-1.04
			298	-2.05
			323	-3.31
AC+nZVI+Z	30.75	119.68	278	-2.52
			298	-4.91
			323	-7.91

removal of COD, thermodynamic parameters including standard enthalpy ( $\Delta H^\circ$ ), standard entropy ( $\Delta S^\circ$ ) and standard free energy ( $\Delta G^\circ$ ) were also determined (see Table 6). The negative values of  $\Delta G^\circ$  (see Table 6) displays the spontaneous nature of COD adsorption by all adsorbents. As can be seen in Table 6, the  $\Delta G^\circ$  values determined for all adsorbents are in the range of physisorption ( $-20 < \Delta G^\circ < 0$  kJ mol<sup>-1</sup>) (Horsfall Jr and Spiff 2008). The positive amounts of  $\Delta H^\circ$  demonstrate that the elimination of COD by all treatments is endothermic. The  $\Delta H^\circ$  values obtained for AC, Z, Z+AC and Z+nZVI adsorbents are in the range of physisorption ( $2.1 < \Delta H^\circ < 20.9$  kJ mol<sup>-1</sup>), whereas the  $\Delta H^\circ$  values acquired for nZVI, AC+nZVI and AC+nZVI+Z adsorbents are in the range of chemisorption ( $20.9 < \Delta H^\circ < 418.4$  kJ mol<sup>-1</sup>) (Gil et al. 2018). Between all treatments, the highest standard enthalpy was observed for AC+nZVI+Z adsorbent about 30.75 kJ mol<sup>-1</sup>. The positive value of  $\Delta H^\circ$  indicates that the interaction between COD and adsorbents is endothermic. Also, the positive amounts of  $\Delta S^\circ$  suggests an increase in randomness and a rising in the degrees of freedom at the solid-solution interface, where the greatest  $\Delta S$  found for AC+nZVI+Z adsorbent was about 119.68 J mol<sup>-1</sup> K<sup>-1</sup>. The results of  $\Delta G^\circ$  and  $\Delta H^\circ$  demonstrate that the adsorption of COD on Z+AC+nZVI were due to both chemical and physical processes.

Kinetic studies of COD were performed by adding 0.3 g of single and combined adsorbents into 300 mL of 350 mg L<sup>-1</sup> COD solution at six levels of contact time (1, 2, 3, 4, 5 and 6 h) without any changes on the solution pH. The kinetic data acquired from COD adsorption tests were fitted to the pseudo-first-order, pseudo-second-order and intraparticle diffusion models, and the results are presented in Table 7. The results confirmed that the pseudo-second-order model better fitted the kinetic data ( $R^2 > 0.972$ ) compared to the pseudo-first-order and intraparticle diffusion models for all adsorbents (see Table 7). The pseudo-second-order rate constants ( $k_2$ ) were obtained between 0.02 and 0.745 h<sup>-1</sup> for Z and nZVI adsorbents, respectively (see Table 7). The highest calculated adsorption capacity was found for AC+

**Table 7** Kinetic parameters for the adsorption of COD by all treatments

Treatment	First-order model			
	$k_1$ (h <sup>-1</sup> )	$q_l$ (mg g <sup>-1</sup> )	$R^2$	
AC	1.11	28.32	0.968	
Z	1.64	23.47	0.964	
nZVI	0.094	21.27	0.931	
AC+Z	1.32	30.95	0.966	
nZVI+Z	0.467	26.11	0.924	
AC+nZVI	0.436	29.07	0.915	
AC+nZVI+Z	0.633	32.47	0.933	
Treatment	Second-order model			
	$k_2$ (g mg <sup>-1</sup> h <sup>-1</sup> )	$q_2$ (mg g <sup>-1</sup> )	$h$ (mg g <sup>-1</sup> h <sup>-1</sup> )	$R^2$
AC	0.027	29.32	23.36	0.999
Z	0.02	25.12	12.81	0.972
nZVI	0.745	21.14	333.33	0.999
AC+Z	0.02	32.46	21.27	0.984
nZVI+Z	0.077	26.32	53.76	0.997
AC+nZVI	0.0794	29.07	67.11	0.997
AC+nZVI+Z	0.0368	34.013	42.55	0.994
Treatment	Inter-particle model			
		$K_{int}$ (mg g <sup>-1</sup> h <sup>1/2</sup> )	$R^2$	
AC		7.63	0.936	
Z		7.15	0.938	
nZVI		0.371	0.688	
AC+Z		8.87	0.939	
nZVI+Z		4.47	0.87	
AC+nZVI		4.69	0.833	
AC+nZVI+Z		7.13	0.945	

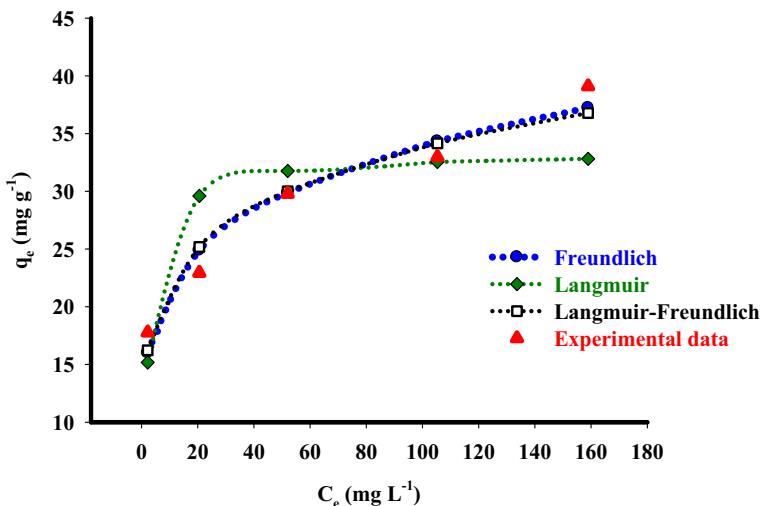
nZVI+Z treatment equal to 34.01 mg g<sup>-1</sup>, whereas the lowest calculated adsorption capacity was 21.14 mg g<sup>-1</sup> for nZVI treatment. However, the greatest initial sorption rate ( $h$ ) was found 333.33 mg g<sup>-1</sup> h<sup>-1</sup> for nZVI adsorbent, which demonstrates a rapid uptake of COD within the first time. The kinetic data demonstrated that intraparticle diffusion was involved in COD adsorption, but it is not the controlling step (see Table 7). It was found that most of the COD uptake (mg g<sup>-1</sup>) data against the square root of the contact time can fit a straight line ( $R^2 > 0.688$ ); but these data do not pass through the origin. The results acquired from kinetic studies confirmed the results from thermodynamic experiments. AC+nZVI+Z treatment follows pseudo-second-order ( $R^2 = 0.994$ ), pseudo-first-order ( $R^2 = 0.933$ ), and intraparticle diffusion ( $R^2 = 0.945$ ) models. These results indicate that the adsorption of COD by triple combined adsorbents are due to both chemical and physical processes, while chemical adsorption is dominant. COD is adsorbed by triple combined adsorbents via ion exchange activity in chemical processes, whereas physical attachment of adsorbate on surface area is responsible for physical processes.

Isotherm studies of COD were performed by adding 0.3 g of single and combined adsorbents into 300 mL of COD solutions at a concentration range 180–550 mg L<sup>-1</sup> for a contact time of 6 h. To determine the correlation between COD concentration in solution and the amount of COD adsorbed on the adsorbents, Langmuir, Freundlich and Langmuir-Freundlich models were employed and the relevant results are presented in Table 8. From this Table, it can be seen that the Freundlich model displays excellent performance with higher  $R^2$  and lower SEE. The Freundlich model is an empirical equation and shows that the surface

**Table 8** Isotherm parameters for the adsorption of COD by all treatments

Treatment	Langmuir (L)				
	<i>b</i>	<i>q<sub>m</sub></i> (mg g <sup>-1</sup> )	<i>R</i> <sup>2</sup>	<i>SEE</i>	
AC	0.038	32.35	0.827	3.02	
Z	0.019	25.76	0.962	0.876	
nZVI	0.035	27.56	0.819	2.35	
AC+Z	0.110	32.87	0.782	3.86	
nZVI+Z	0.043	32.71	0.808	3.32	
AC+nZVI	0.054	33.42	0.794	3.65	
AC+nZVI+Z	0.383	35.64	0.662	5.61	
Treatment	Freundlich (F)				
	<i>K<sub>F</sub></i>	<i>1/n</i>	<i>R</i> <sup>2</sup>	<i>SEE</i>	
AC	6.3	0.292	0.964	1.37	
Z	4.24	0.293	0.995	0.304	
nZVI	6.016	0.263	0.952	1.21	
AC+Z	7.52	0.277	0.959	1.614	
nZVI+Z	6.72	0.287	0.958	1.553	
AC+nZVI	10.35	0.226	0.99	0.732	
AC+nZVI+Z	13.73	0.196	0.955	2.02	
Treatment	Langmuir-Freundlich (L-F)				
	<i>b</i>	<i>1/n</i>	<i>q<sub>m</sub></i> (mg g <sup>-1</sup> )	<i>R</i> <sup>2</sup>	<i>SEE</i>
AC	0.102	0.533	45.51	0.918	2.08
Z	0.009	1.2	24.12	0.943	1.068
nZVI	0.017	1.19	26.17	0.759	2.71
AC+Z	0.111	0.441	55.72	0.921	2.13
nZVI+Z	0.221	0.443	47.32	0.919	2.32
AC+nZVI	0.117	0.425	59.18	0.924	2.23
AC+nZVI+Z	0.07	0.217	211.74	0.949	2.18

of the single and combined adsorbents are heterogeneous and have varying pores, which can be used for multilayer adsorption (Malekian et al. 2011). In the Freundlich model, *K<sub>F</sub>* and *n* are the maximum adsorption capacity and the adsorption intensity, respectively, which are



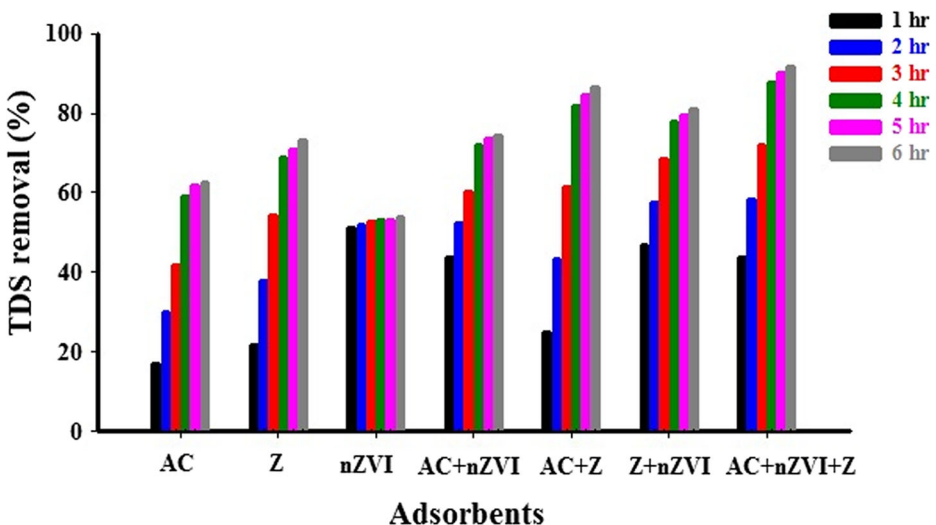
**Fig. 5** The fitting of isotherm models for COD adsorption on AC+nZVI+Z adsorbent



attributed to the affinity of single and combined adsorbents for COD adsorption. Indeed, the value of  $1/n$  in the range of  $0 < 1/n < 1$  displays suitable adsorption isotherm, whereas  $1/n > 1$  indicates unsuitable adsorption isotherm. The values of  $1/n$  for all treatments demonstrate that the adsorption of COD on the single and combined adsorbents is suitable (see Table 8). Higher  $K_F$  values indicate a greater adsorption capacity and lower  $1/n$  values demonstrate stronger adsorption strength. Between the single and combined adsorbents, AC+nZVI+Z had the highest  $K_F$  (13.73) and the lowest  $1/n$  (0.196) values. The  $K_F$  constant values achieved by the Freundlich model were found between 4.24 and 13.73 for single and combined adsorbents. The COD binding affinity of single and combined adsorbents based on the  $K_F$  values was in the order AC+nZVI+Z > AC+nZVI > AC+Z > nZVI+Z > AC > nZVI > Z (see Table 8). Similarly, the maximum adsorption capacities of single and combined adsorbents based on the Langmuir-Freundlich model were also in the order AC+nZVI+Z > AC+nZVI > AC+Z > nZVI+Z > AC > nZVI > Z (see Table 8). The highest calculated adsorption capacity was found for AC+nZVI+Z treatment equal to 211.74 mg g<sup>-1</sup>, whereas the lowest calculated adsorption capacity was 24.12 mg g<sup>-1</sup> for nZVI treatment. The fitting of Langmuir, Freundlich and Langmuir-Freundlich models for COD adsorption on AC+nZVI+Z adsorbent compared with experimental data is illustrated in Fig. 5.

### 3.2 TDS

The consequences of TDS removal as function of time using various treatments at an initial concentration of 2800 mg L<sup>-1</sup> are illustrated in Fig. 6. Between single adsorbents, Iranian Z as a natural and inexpensive material had the best performance in removing TDS from greywater. In single adsorbents, AC, nZVI and Z removed 62.4, 53.8 and 73.1% of initial TDS concentration (2800 mg L<sup>-1</sup>), respectively. Similar results have been reported by Aghakhani et al. (2012), which indicated that Iranian Z has the highest adsorption rate of salinity ions



**Fig. 6** TDS removal (%) by using single and combined adsorbents of AC, Z and nZVI (temperature, 25 ± 1 °C; Initial TDS concentration, 2800 mg L<sup>-1</sup>; pH, natural, adsorbent dose, 0.3 g)

compared to AC and peat. It is important that adsorption efficiency of TDS by Z is greater than AC as commercial and expensive adsorbents. In fact, a cation exchange ability and hydrophilic surface give advantage for zeolite to adsorb TDS. In double treatments, the highest adsorption efficiency was attained by the combination of Z with AC, with TDS removal of 86.72%. In fact, a combination of AC as a carbon-based substance with Z as an alumino-silicates material is favorable because AC+Z is affected by the unique properties of both adsorbents. The highest adsorption efficiency of TDS was observed for AC+nZVI+Z treatment equal to 91.81%, for which the residual TDS reached 244.16 mg L<sup>-1</sup> (< 450 mg L<sup>-1</sup>). The order of TDS removal by the various adsorbents was: AC+nZVI+Z > AC+Z > nZVI+Z > AC+nZVI > Z > AC > nZVI. The treated greywater by triple combined adsorbents is suitable for unrestricted irrigation of landscape according to Iranian standards. The optimum contact time for TDS removal by all treatments, except nZVI, was achieved in 4 h. In the case of nZVI, the majority of TDS (95.33%) was removed within the first 1 h of contact time. In the study by Aghakhani et al. (2012), the highest and lowest adsorption capacity of salinity ions was obtained by using peat and AC, respectively.

### 3.3 Turbidity

The results of turbidity removal as function of time using various treatments at initial concentration of 280 NTU are presented in Fig. 7. As seen in this figure, the triple combined adsorbent removed more than 98.1% of initial turbidity after 6 h of contact time and had the best performance. The residual turbidity of treated greywater by triple combined adsorbent reached 5.3 NTU (< 10 NTU); this greywater can be used for unrestricted irrigation of landscape according to Iranian standards. The order of the turbidity removal in single and combined treatments was: AC+nZVI+Z > nZVI+Z > AC+Z > nZVI+AC > Z >

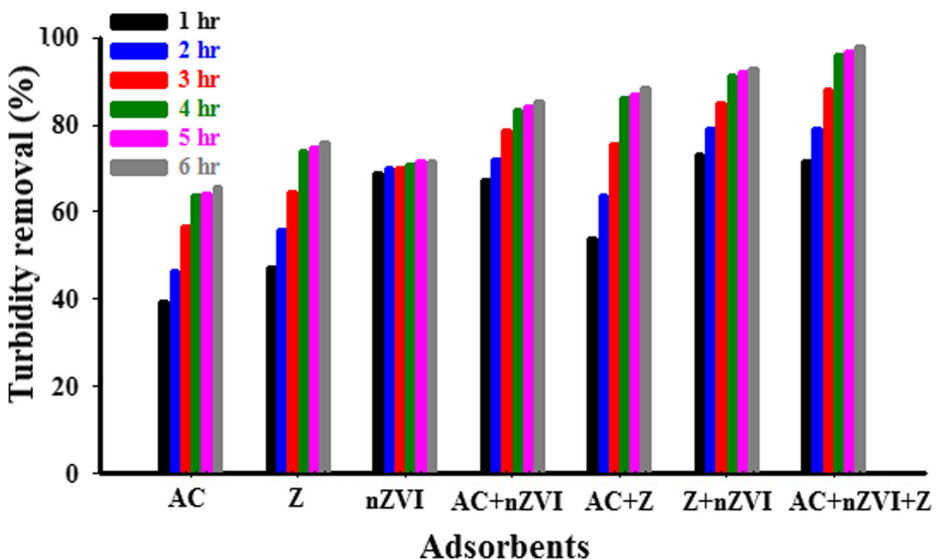


Fig. 7 Turbidity removal (%) by using single and combined adsorbents of AC, Z and nZVI (temperature, 25 ± 1 °C; Initial Turbidity concentration, 280 NTU; pH, natural, adsorbent dose, 0.3 g)

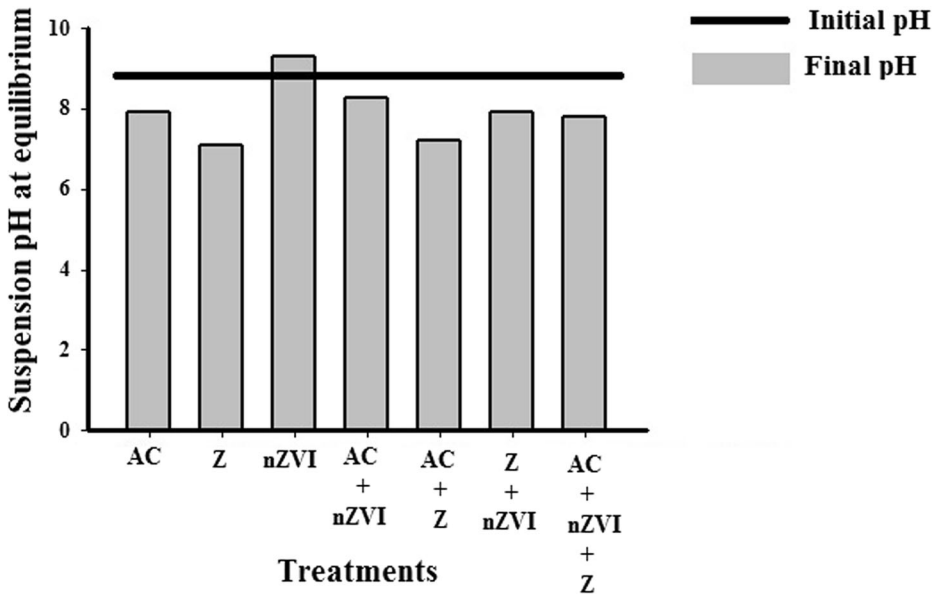


Fig. 8 Initial and final pH of greywater due to usage of single and combined adsorbents

nZVI > AC. Between single adsorbents, Z had the best performance in removing turbidity from greywater, where 75.79% of turbidity was eliminated within 6 h of contact time. Between double treatments, the greatest adsorption efficiency of turbidity belongs to combination of Z with nZVI, with turbidity removal reaching 92.91%. In the study by Pidou (2006), 61% reduction of turbidity from greywater was obtained by using a combination of a sand filter with AC.

### 3.4 pH

The initial and final pH values of seven adsorbents are presented in Fig. 8. As seen in this figure, the final pH values of all adsorbents were observed between 7.1 and 9.3. Therefore, the

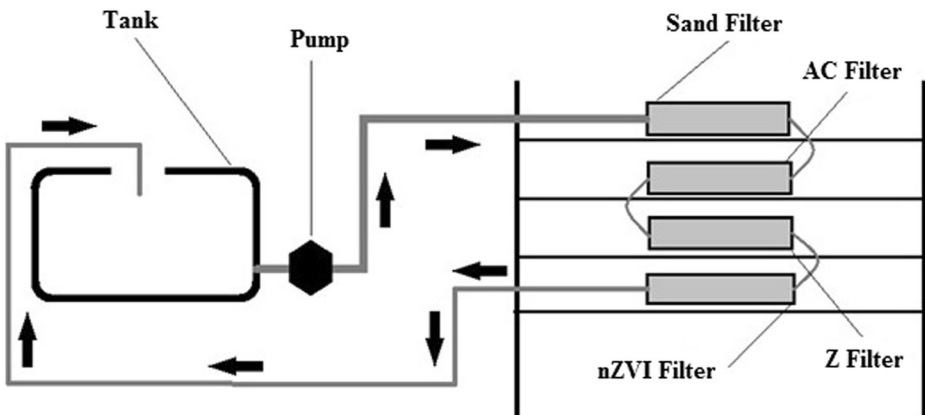


Fig. 9 Proposed wastewater treatment system for triple combined adsorbent

final pH values of all adsorbents, except nZVI, are in the range of 6.5–8.5, which demonstrates that the pH of the treated greywater is appropriate for restricted irrigation of landscape according to Iranian standards.

### 3.5 Economic Evaluation

The approximate cost of the Iranian Z, stabilized nZVI and AC utilized for wastewater treatment in Iran is about 100, 200 and 900 US\$ ton<sup>-1</sup>, respectively, whereas the price of the triple combined adsorbent would be lower than 400 US\$ ton<sup>-1</sup>. The relative price of the triple combined adsorbent employed in this study was much lower than AC as a commercial adsorbent. So, by combining the low and high cost materials, not only the price of wastewater remediation will be reduced but also the adsorption capacity of the product may increase. A proposed greywater treatment system, which can be used for the triple combined adsorbent is presented in Fig. 9. The proposed system consists of a storage tank and a greywater treatment system. The horizontal treatment system includes a slow sand filter followed by AC, Z and nZVI filters (see Fig. 9). Filters and tank are connected together by tubes. The greywater is sent via a pump to the proposed treatment filter, at a constant flow rate controlled by a tap, with a bypass system diverting excess effluent into the sewage tank. After that, the treated greywater is returned to the tank by a tube from the nZVI filter. This cycle continues for 6 h.

## 4 Conclusions

To assess the performance of three adsorbents (AC, Z and nZVI) in single and combined forms to treat greywater, seven treatments were provided in the usual batch mode. The following results were found from this work:

- (1). The triple combination of adsorbents (AC+nZVI+Z) adsorbed the highest percent of COD and TDS, and also removed the turbidity almost completely.
- (2). According to the thermodynamic results of  $\Delta G^\circ$  and  $\Delta H^\circ$  values, the adsorption of COD by the triple combination treatment using the above adsorbents (AC+nZVI+Z) was due to both physical and chemical processes.
- (3). The final pH values of all treatments, except of nZVI treatment, were found to be in the range of acceptable limits.
- (4). The optimum contact time for COD, TDS and turbidity removal by all treatments, except of nZVI treatment, was attained in a time of 4 h.
- (5). The triple combination of adsorbents (AC+nZVI+Z) is able to reduce the COD, TDS, turbidity and pH level to the required reuse in landscape irrigation and the results were satisfactory according to Iranian standards.

### Compliance with Ethical Standards

**Conflicts of Interest** The authors state that there is no conflict of interest.

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