Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2014.904818

54 (2015) 2723–2730
May

Taylor & Francis
Taylor & Francis Group

Using natural zeolite for contamination reduction of agricultural soil irrigated with treated urban wastewater

Hajar Taheri-Sodejani[a], Mahdi Ghobadinia[a,*], Sayyed-Hassan Tabatabaei[a], Hossein Kazemian[b]

[a]*Faculty of Agriculture, Department of Water Engineering, Shahrekord University, Shahrekord, P.O. BOX: 115, Iran, Email: ghobadi-m@agr.sku.ac.ir*
[b]*Faculty of Engineering, Department of Chemical and Biochemical Engineering, Western University, London, Thompson Engineering Building, Ontario, CAN N6A 5B9 5196612111, Canada*

Received 24 July 2013; Accepted 1 March 2014

ABSTRACT

Nowadays, due to the global water crisis, reusing treated wastewater is being considered as water resources for different purposes, particularly for agricultural applications. Irrigating soil using urban wastewaters which are not treated properly, however, has negative effect on the soil and its subsequent drained water. Because of that, selective adsorption properties of the natural zeolite were used to improve filtering properties of the soil irrigated with wastewater. The effects of application method, dosage, and particle size of natural zeolite were studied on the EC, pH, BOD_5, Na^+, Ca^{2+}+Mg^{2+}, and nitrate concentration of an urban wastewater by passing it through zeolite-added soil columns. The result shows that by adding zeolite to the soil column, the value of pH, EC, and Na^+ of the column outlet were increased, while its Ca^{2+}+Mg^{2+}, nitrate concentration, as well as the BOD_5 were decreased. BOD_5 level of the column effluent in the control, mixed, and layered treated soil with zeolite were lower than BOD_5 of used fresh wastewater by 38.42, 54.98, and 71.84%, respectively. However, the nitrate concentration of the column effluent in the control, mixed, and layered treated soil with zeolite were lower than the nitrate content of the fresh wastewater by 12.18, 32.19, and 54.90%, respectively. It can be concluded that application of the natural zeolite into the soil in a layered treatment does more effectively reduce the pollutant transferred to the soil-depth and consequently can improve the quality of drainage water.

Keywords: Layer form application; Natural zeolite; Nitrate; Soil columns; Wastewater

1. Introduction

Because of the global water crisis, using low-quality water sources, such as different wastewater streams after proper treatment is an inevitable necessity [1]. In addition, using wastewater in agriculture as a source of nutrients for plants has been widely accepted in areas with water shortage [2]. However, considering adverse effect of using such poor quality resources on irrigated soils, it should be carefully tested for the content of dissolved cations, particularly calcium and magnesium, and nitrogen and organic carbon [1]. Increasing the cation-exchange capacity of soil can minimize some diverse effects of using poor quality waters. Natural materials, such as zeolite

*Corresponding author.

minerals with high cation-exchange capacity can be considered as potential additives for improving the properties of the soils that are irrigated with low quality wastewaters [3,4]. Zeolites are crystalline aluminosilicates, consisting of SiO_4 and AlO_4 tetrahedral units, in which the oxygen bridge connects the Si and Al atoms creating a three-dimensional rigid structure with molecular scale pores and cavities [5]. Hydrated cations of alkaline and alkaline-earth elements compensate negative charge of natural zeolite framework. These cations are mobile, which means they can easily exchange with other cations in the surrounding environment [6–8].

Using natural and synthetic zeolites for different contaminants' removal including heavy metal cations [9,10], radionuclide [10], oxy-anions [11], and volatile organic molecules [12] from various polluted water and wastewater streams have been studied. Furthermore, remediation of soil contaminated with heavy metals, such as cadmium has been reported [4].

According to a research report, the EC of the leached water has been decreased by adding zeolite to the soil. Additionally, the pH of lysimeters drainage water of was also increased by adding zeolite. Moreover, organic matters content of the soil treated with zeolite was higher because of the high CEC and microporous structure of zeolite. Nevertheless, the efficiency of biochemical oxygen demand (BOD) removal was also improved in the presence of zeolite [13].

Nitrates, which are negatively charged ions cannot be adsorbed by the soil because the framework of aluminosilicate clays are negative; thus, nitrate anions, which are highly soluble in water can easily leach out of the soil profile [14]. It has been reported that the mixed method application of zeolite remarkably reduces nitrate leaching. By adding 2, 4, and 8 g of zeolite to 1 kg of soil, nitrate leaching is reduced to 6, 28.7 and 47.6%, respectively [15]. It has been also claimed by researchers that fine particles of natural Clinoptilolite can effectively reduce the nitrogen washed out from the soil fertilized by urea [16]. Using a layer of natural zeolite on the surface of the soil increases the adsorbed nitrate [17]. In this research, the effect of application method (i.e. mixed and layered method) of natural zeolite (Clinoptilolite) on the soil that irrigated with urban treated wastewater (TWW) was studied in a column reactor. Likewise, the influences of different factors, such as zeolite particle size on the water chemical parameters were evaluated after passing the column.

2. Materials and methods

The TWW used in this research was obtained from Shahrekord wastewater treatment plant. Some physic-chemical properties of the soil, TWW, and Tap water are shown in Tables 1 and 2.

Natural zeolite used in this research was Clinoptilolite from Semnan region of Iran. The zeolite was pulverized, using ball-mill technique (Sanat Ceram Company). Two desired particle sizes of zeolite (i.e. 63–125 μm and less than 63 μm) were selected using the ASTM standard sieves. The zeolite had the following chemical compositions (wt%) $SiO_2 = 65.90$, $Al_2O_3 = 11.20$, $Na_2O = 2.10$, $K_2O = 2.31$, $CaO = 3.20$, $Fe_2O_3 = 1.25$, $MgO = 0.52$, LOI = 11.89, and $SiO2/Al_2O_3 = 5.9$ [17].

In this study, 27 columns made of PVC with 10.5 cm inner diameters and 60 cm height were used for the tests with nine treatments and three replications. Schematic diagram of experimental setup is illustrated in Fig. 1. In order to prevent lateral flowing of water through the gaps between the soil and the column walls (i.e. bypass flow), the inner walls of the column were covered with a very thin layer of grease in order to make the walls hydrophobic. A support mesh was installed at the bottom of each column to keep soil inside the column. In order to fill the columns, the first five centimeters of each column was filled with gravel as a drain filter according to the USBR (United States Bureau of Reclamation) guideline. Then 40 cm of the columns were filled with the soil or mixture of soil and zeolite. At the top, a layer of gravel with 5 cm thickness was used to prevent the soil surface from disturbance caused by irrigating water. Nevertheless, the upper 10 cm of each column was used as a water reservoir. The layered treatments were filled similar to mixed treatments, in which desired amount of zeolite was added as a middle layer in the soil columns. To stabilize the condition of the columns prior to the experiments, all of the 27 columns were irrigated three

Table 1
Chemical and physical properties of the soil at the beginning of the experiment

Soil texture	CEC ($cmol_c$/kg)	pH	EC* (dS/m)	HCO_3^-* (meq/L)	CO_3^{2-}* (meq/L)	$CaCO_3$ (%)	N (mg/kg)	Na^+* (meq/L)
Silt loam	25.3	8.5	0.3	2.3	0	25.2	14.32	0.8

*Measurements in water and soil extracts were 2:1.

Table 2
Chemical characteristics of the TWW and tap water

	pH	EC (dS/m)	TDS (mg/L)	TSS (mg/L)	HCO^{-}_{3} (meq/L)	N–NO_3 (mg/L)	BOD_5 (meq/L)	SAR $(mmol/L)^{0.5}$	Ca^{2+}+Mg^{2+} (meq/L)	Na^{+} (meq/L)
TWW	7.9	0.8	429	30	5.2	14.2	15.8	2.4	4.6	3.6
Tap water	7.5	0.3	38	0	–	2.6	3.9	0.13	–	–

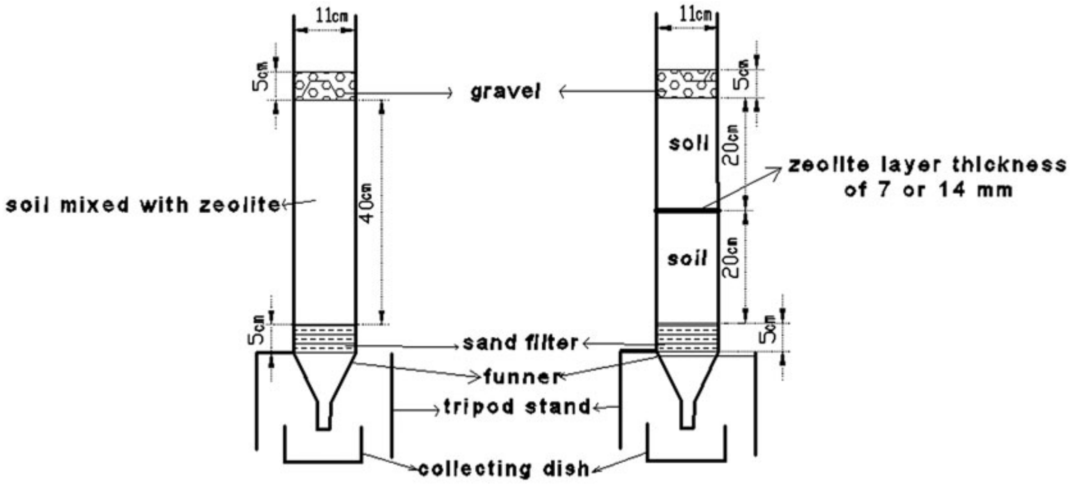

Fig. 1. Schematic diagrams of the columns used for the mixed soil-zeolite tests (left) and the layered soil-zeolite experiments (right).

times by tap water with 1.5 nv (n and v refer to porosity and total volume of soil column).

The series of conducted experiments consisted of nine treatments in three replications. In order to optimize the treatment condition for maximum efficiency, a factorial experimental design was employed with three factors at two levels. The first factor was zeolite application method including two levels (i.e. mixed with soil and as layered in soil). The second one was zeolite particle size (i.e. less than 63 and 125–63 μm). And finally, the third factor was the zeolite dosage (i.e. 2 and 4% of the total weight). Tests' conditions are summarized in Table 3.

In this study, wastewater was added to the columns 13 times on a weekly basis. Volume of wastewater, in all irrigations, was set at "nv." During the first, fourth, ninth, and thirteenth irrigations, three samples of the wastewater used for irrigation and 27 samples of the column effluents (one from each column) were examined using the following techniques: pH by pH meter (ELMETRON CP-501) [18], EC: EC meter (Jenway 4010) [18], BOD_5 : BOD meter [18], Total calcium and magnesium: Titration method with EDTA [18], Sodium: Flame photometer [18], Nitrate: Spectrophotometric method [19], and soil final infiltration rate by Falling-head method [20].

For each of the wastewater components, the change percentage was calculated by the Eq. (1):

$$PC(\%) = \frac{(C_{0w} - C_{tw})}{C_{0w}} \times 100 \quad (1)$$

where C_{tw} is the concentration of the output drainage water, C_{0w} is the concentration of the input wastewater, and PC is the change percentage.

Using SAS software version 9, Statistical analysis was performed including the Duncan mean comparison test at 95% confidence level.

3. Results and discussion

3.1. pH changes

According to the experimental results, the pH values of all columns' effluents increased, however, this change was remarkable in the soil column modified with zeolite (Fig. 2). As it is shown in Table 4,

Table 3
Tests' conditions based on experimental factorial design

Symbol treatment	Zeolite application method	The size of the zeolite (μm)	Weight of zeolite in the soil (%)
CTRL	–	–	0
MB2	Zeolite mixed with soil	63–125	2
MB4	Zeolite mixed with soil	63–125	4
MA2	Zeolite mixed with soil	Less than 63	2
MA4	Zeolite mixed with soil	Less than 63	4
LB2	Zeolite layer of thickness 7 mm in the soil	63–125	2
LB4	Zeolite layer thickness of 14 mm in the soil	63–125	4
LA2	Zeolite layer of thickness 7 mm in the soil	Less than 63	2
LA4	Zeolite layer of thickness 14 mm in the soil	Less than 63	4

increasing the zeolite dosage resulted in more significant changes in the pH of the column effluents. Furthermore, mixed applying method was more effective than the layered method in this regard. Statistically, the difference between treatments was significant at 1% level. The pH of higher effluents can be attributed to the release of sodium ions from the zeolite exchange sites into the surrounding medium as a result of ion-exchange reaction between zeolite and cations in the soil and wastewater. This phenomenon can increase the soil alkalinity. The above-mentioned observation is in agreement with other studies too [13].

3.2. Changes in electrical conductivity (EC)

According to the experimental results, EC of the effluents was increased along with zeolite dosage enhancement in layered method (Fig. 3 and Table 4). Salt (i.e. cations) can adsorb or desorb from zeolite structure depending on the chemical characteristics of the tested zeolite [13]. Our experimental results revealed that increasing the zeolite dosage causes higher cation-exchange capacity of the column, which means that more sodium cations are replaced by calcium and magnesium and are released from zeolite into the surrounding medium. Consequently, EC increases in the column effluents. Moreover, reducing the zeolite particle size in mixed treatment results in decrease in EC of the column effluent, whereas using finer particles in the layered method increases the EC.

3.3. Biochemical oxygen demand (BOD)

The average amount of BOD_5 in the effluents of all columns was always lower than in used wastewater for irrigation (Fig. 4). The results uncovered that in layered method of zeolite application, increasing zeolite dosage and decreasing zeolite particle size leads to more decrease in BOD_5 of the effluent (Table 4). These

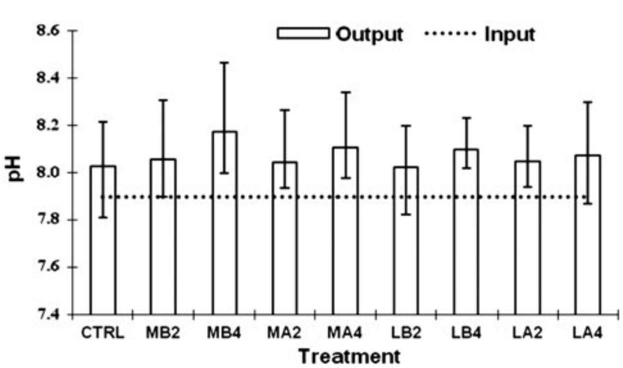

Fig. 2. pH average in input wastewater and output drainage throughout the research.

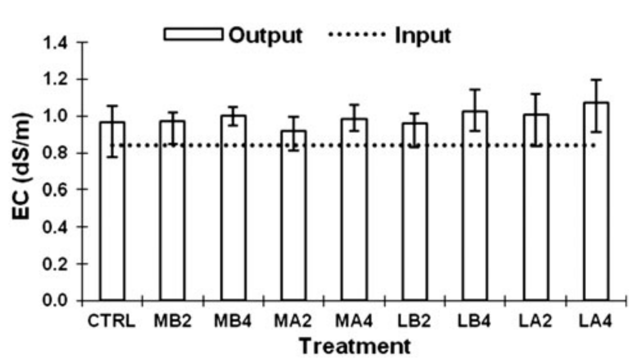

Fig. 3. EC average of the input wastewater and output drainage throughout the research.

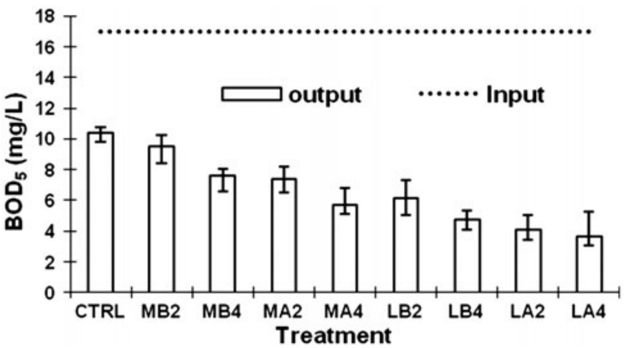

Fig. 4. BOD_5 value (average) of the input wastewater and output drainage throughout the research.

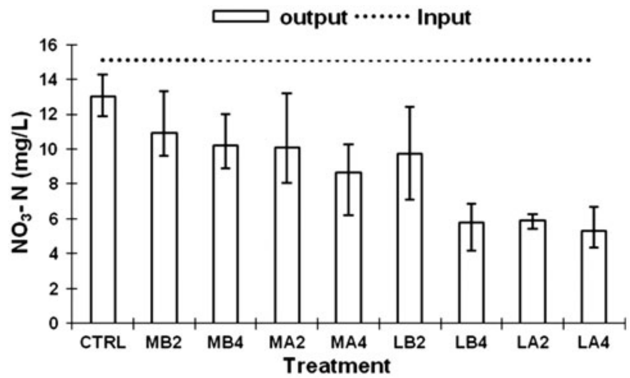

Fig. 5. NO_3-N content (average) in the input wastewater and output drainage throughout the research.

observations can be attributed to the fact that compact zeolite layer due to its very fine particles, acts as an obstacle for organic matter to pass through the layer. Therefore, the BOD of the effluent is diminished as a result of lower concentration of organic matter. Finer particles of zeolite have larger surface area in comparison to the coarser particles, which can be attributed to more organic matter adsorption. Therefore, zeolite dosage increase would lead to the efficiency of BOD_5 removal increase as well. As a result, the layer treatments with smaller particles (i.e. LA2 and LA4) had greater effect on reducing BOD_5 of the wastewater.

3.4. Nitrate

The nitrate content of the drained water of all treatments was always lesser than the inlet wastewater (Fig. 5 and Table 4). Applying natural zeolite, in both layered and mixed method caused an increase in the nitrate absorption capacity of the soil. It can be concluded that using natural zeolite, the N-fertilizer demand of the plants will be remarkably reduced. The reason is that the nitrate leaching out of soil is likely to reduce. Furthermore, nitrate contamination of the groundwater as a result of fertilizer leaching can be prevented.

Nitrate content of the column effluent of the zeolite modified soil in both mixed and layered treatments was reduced by 32.19 and 54.90%, respectively; whereas, the nitrate reduction of the control experiment (i.e. column of soil without zeolite) was just 12.18%. This indicates that the layered treatment is more efficient in comparison to the mixed method. Results also showed that decrease in the nitrate adsorption in control and mixed method treatment was faster than the layered one (Fig. 6).

The experimental results are in good agreement with literature. It has been reported that application of zeolite mixed with soil will decrease the nitrate leaching form the soil modified with zeolite. In this particular report, the reduction is attributed to the trapping of nitrate into the pores of zeolite framework [15,16]. In another research, applications of zeolite in layered method resulted in an increase on the adsorption of nitrogen from urea of fertilizer in the soil [17]. Application of finer particles of zeolite resulted in decreasing the nitrate leaching rate. The reduction of nitrogen leaching rate by increasing zeolite content in a mixed method is also reported [15–17].

3.5. Sodium adsorption ratio (SAR)

According to the experimental results (Fig. 7 and Table 4), effluents' sodium content of the columns contained zeolite in both mixed and layered treatments increased compared to the control column. This could be attributed to the ion exchange reaction between mobile sodium ions of the zeolite cation sites and other cations in the surrounding environment [21]. Sodium concentration of drained water in mixed

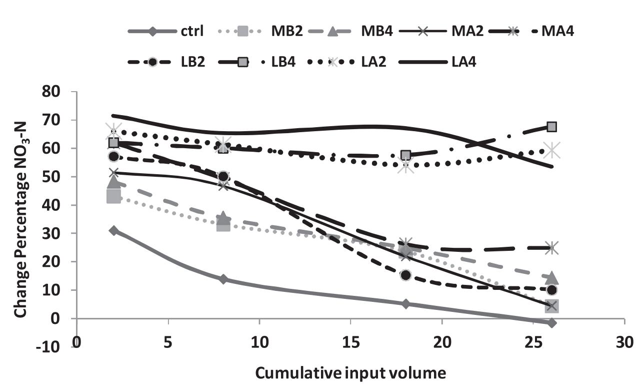

Fig. 6. NO3-N Change Percentage vs. Cumulative input volume during experiment.

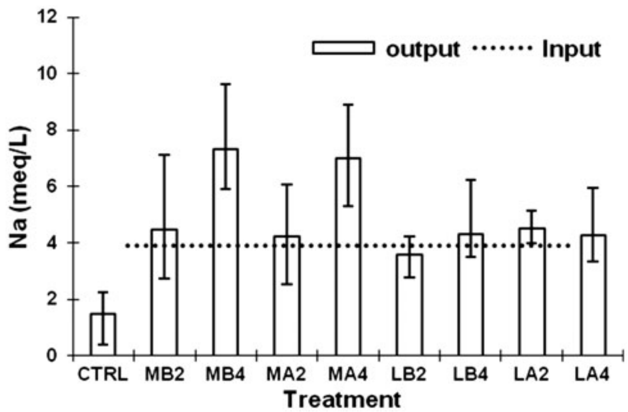

Fig. 7. Na content (average) of input wastewater and output drainage throughout the research.

treatments was more than of those of the layered treatment (Fig. 7). By reducing particle size of zeolite, the sodium content of the effluent in mixed and layered treatments decreased and increased, respectively. Whereas, by increasing zeolite dosage, sodium content of the drained wastewater increased in both methods.

Furthermore, the total amount of calcium and magnesium content of the effluent increased in both control and layered treatments; however, the Ca and Mg content of the effluent decreased in the mixed treatments. This means that the Ca and Mg absorption capacity is improved in the modified soil with zeolite method (Fig. 8). In spite of the higher level of calcium and magnesium in the effluent of the layered treatment, it was still lower than the control treatment (Table 4). These observations can be explained as a result of adsorbing calcium and magnesium cations by cation-exchange mechanism with the mobile sodium cations inside the zeolite pores and channels [10,13]. Using finer zeolite particles and increasing the zeolite dosage, calcium and magnesium leaching decreased in both layered and mixed treatments. Through decreasing zeolite particle size, the specific surface area will be increased and consequently access to the zeolite cationic sites will facilitate cation-exchange reaction. This may result in improvement of the adsorption capacity and efficiency [22].

As it can be seen in Fig. 9, the control treatment has the lowest SAR output, which can be attributed to the lower content of the calcium and magnesium ions and also absorption of sodium ions by the soil. Both methods of zeolite applications would lead to SAR increase of the output. Nevertheless, the rise in mixed treatments was higher than the layered ones. Using of finer particles of zeolite in the mixed and layered treatments brought about decrease and increase in the SAR changes, respectively. Increasing zeolite dosage caused an increase in effluent's SAR of mixed treatments. However, there was no significant difference between the SAR of layered and control treatments (Table 4).

The layered method however was more effective in reducing the nitrate and the BOD_5 of the wastewater drained from the column. It has been reported that at lower flow rate of water, because of longer contact between the adsorbent (soil, etc.) and adsorbate, and therefore, more efficient chemical and biological reactions, more pollutants will be adsorbed by the soil. Consequently, the quality of drained water will be improved [23]. Accordingly, it can be concluded that the zeolite layer decreases the hydraulic conductivity of wastewater into the soil, and thus, increases the time that wastewater remains in contact with the soil. This would account for adsorption of more pollutants from wastewater. Average ECs of the layered and mixed treatments were 0.78 and 0.57 cm/minute, respectively (Fig. 10). Hence, the higher capacity of

Table 4
Average of chemical characteristic change percentage throughout the research

Treatment	pH	EC	BOD	NO_3-N	Na	Ca+Mg	SAR
CTRL	-1.63^{a}	-15.60^{cb}	38.42^{h}	12.18^{f}	64.99^{a}	-52.68^{g}	70.79^{a}
MB2	-1.98^{a}	-16.72^{cb}	43.56^{g}	26.10^{e}	-14.37^{c}	-1.15^{d}	-18.91^{d}
MB4	-3.65^{b}	-20.95^{ba}	54.85^{f}	30.82^{e}	-89.57^{e}	42.21^{a}	-174.80^{f}
MA2	-1.83^{a}	-10.54^{c}	55.70^{f}	31.18^{e}	-8.11^{c}	18.22^{c}	-22.26^{d}
MA4	-2.69^{ab}	-18.91^{bc}	65.82^{d}	40.65^{c}	-80.91^{d}	30.91^{b}	-135.13^{e}
LB2	-1.55^{a}	-14.94^{cb}	63.16^{e}	33.20^{d}	9.03^{b}	-15.98^{f}	19.91^{b}
LB4	-2.54^{ab}	-23.93^{ba}	71.35^{c}	61.81^{b}	-10.64^{c}	-13.21^{ef}	1.34^{c}
LA2	-1.85^{a}	-21.04^{ba}	75.28^{b}	60.24^{b}	-15.87^{c}	-9.14^{ef}	-5.29^{c}
LA4	-2.24^{a}	-29.26^{a}	77.56^{a}	64.33^{a}	-9.58^{c}	-6.32^{e}	-1.09^{c}

Note: The letters show differences between the average base on the mean analysis and the same letter shows that there is no difference between the two numbers.

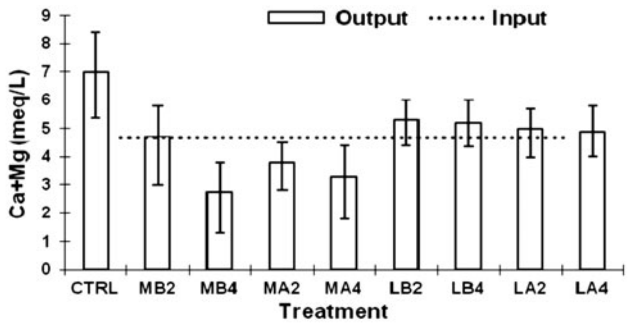

Fig. 8. Ca^{2+}+Mg^{2+}content (average) of input wastewater and output drainage throughout the research.

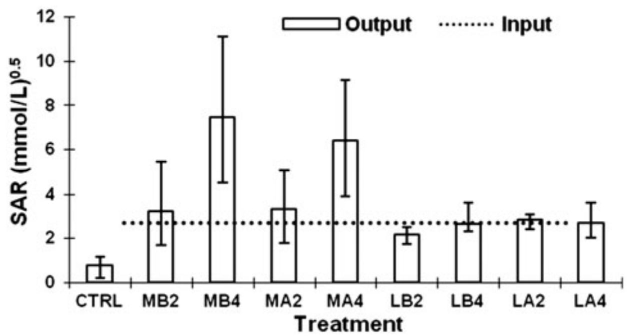

Fig. 9. SAR value (average) of input wastewater and output drainage throughout the research.

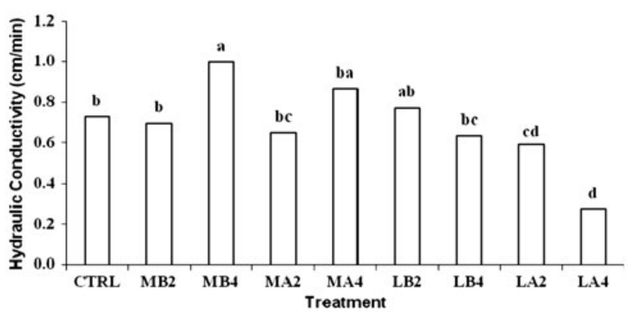

Fig. 10. The average hydraulic conductivity of the entire period.

layered treatments for removing BOD_5 is explainable by the lower flow rate of wastewater transfer through the column, in these treatments.

4. Conclusion

In this research, the effect of adding zeolite as a soil modifier was studied and major outcomes can be summarized as follows:

- Adding natural zeolite to the soil, which is irrigated by treated urban wastewater, will increase the pH, EC, and the sodium content of the effluent.
- Zeolite application in layered method, smaller particle size (less than 63 μm) and higher dosage (4%), is more effective in reducing the BOD_5 and nitrate in wastewater drainage.
- For calcium and magnesium, mixed treatments, smaller zeolite particles (less than 63 μm), and higher dosage (4%) resulted in higher adsorption capacity of adsorbing calcium and magnesium cations from the wastewater.
- Eventually, it can be concluded that application of the natural zeolite to the soil in a layered treatment is more effective for reduction of pollutants transferred to the soil-depth and consequently improves the quality of drainage water.

Acknowledgment

Financial support of this study was provided by a grant from the office of vice dean for Research at Shahrekord University. Additionally, the authors would like to express their thanks to the manager and personnel of Bahram-Abad Water Treatment Plant, Research Center of Agriculture and Natural Resources for their cordial supports and also authors greatly appreciate AfrandToska Company for supplying zeolite for this research.

List of symbols

C_{tw} — the concentration of the output drainage water
C_{0w} — the concentration of the input wastewater
PC — change percentage

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