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SALINE-SODIC SOIL RECLAMATION WITH STABILIZED SEWAGE SLUDGE AND RECYCLED WASTEWATER

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Abstract

Saline-sodic soil reclamation can be enhanced with a sustainable and environmental approach by using organic waste materials. Moreover, wastewater can be used in leaching processes to protect freshwater resources in regions with water shortages. The aim of this study was to reclaim saline-sodic soil by using recycled wastewater and stabilized sewage sludge together with gypsum. The experiment was designed with three replicates using four different sewage sludge doses (0, 4, 8 and 12 kg m^{-2}), and four different leaching variants from alternative applications of freshwater and wastewater (4 sludge doses × 4 leaching variants × 3 replicates = 48 columns). The soil depth tested was 30 cm and sewage sludge was mixed in the top 15 cm of soil. While leaching variants had no effect on improving reclamation, sewage sludge introduced important effects. Soil salinity was reduced by 79% and 86% in the top and sub layer, respectively. Wet aggregate stability, organic matter, and exchangeable Ca+Mg content were increased in the upper layer, while pH, exchangeable Na, and exchangeable sodium percentage (ESP) were decreased. Sodium removal ratio reached 64%, whereas this ratio was 41.7% for the columns with no sewage sludge. Hydraulic conductivity was negatively affected due to high ESP, low electrolyte concentration and weak aggregate stability in the sub layer. The total N and P accumulation in the columns was restricted due to leaching effects. Although it was observed that heavy metals tended to accumulate in the sub layer, the concentrations did not exceed the pollution limit values.

Keywords: aggregate stability, heavy metal, hydraulic conductivity, saline-sodic soil, sewage sludge

1. Introduction

Land degradation from salt accumulation adversely affects agricultural production. It has been estimated worldwide that 20% of cultivated areas and 33% of irrigated areas are afflicted with high salinity, which translates to 1 billion hectares (Shrivastava and Kumar, 2015). Only leaching is suitable for the reclamation of saline soils. The reclamation processes of sodic soils are complex due to low water permeability. Excessive contents of exchangeable Na on cation exchange sites in soil causes structural degradation due to clay dispersion and plugging of the soil pores. These harmful effects from excessive Na are explained by diffuse double layer theory. The negative charges on the exterior surface of the clay minerals are balanced by ions with positive charges such as Ca, Mg, Na and K. This structure that consists of positive counter ions balanced with negative charges on the clay surface is referred to as the electric double layer (Mahanta et al., 2014). The monovalent Na ions have a weak clay-binding capability. When the clay particles get wet, the Na ions hydrate and the plates move away from the ions and decrease the attraction force. The plates can move enough to eliminate the attraction forces and dispersion occurs (Hensen and Smit, 2002). However, divalent Ca ions have good clay-binding capability. Since the double charged Ca ions are more strongly attracted, the thickness of the double layer is low and swelling is restricted. Therefore, the basic approach in the improvement of sodic and/or saline-sodic soils

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permeability is to increase Ca in the cation exchange complex for the removal of Na accumulated in the exchange complex (Qadir et al., 2003). Introducing Ca ions into the soil provides a favorable electrolyte concentration with high Ca/Na ratio in soil solution and the replaced Na with Ca is removed out of the soil profile by leaching water, and thus clay flocculation and aggregate stability are promoted (Alcívar et al., 2018).

Gypsum as a direct Ca source is widely used for many reasons. These include being more affordable than other chemicals, having relatively good solubility, they are readily available and easy to use. Although gypsum application improves aggregation and hydraulic properties of sodic and/or saline-sodic soils and decreases exchangeable sodium percentage, combined use of organic and inorganic amendments accelerates the rate of soil amelioration (Prapagar et al., 2013; Qadir et al., 2007). Applying organic materials into the soil reduces the pH and enhances the CO_2 production, and increased carbonic acid in the soil causes dissolution of the calcite (Alcívar et al., 2018; Šimůnek and Suarez, 1997). Thus, while Ca release from natural calcite minerals facilitates the removal of Na, the increase of permeability can be supported by new pores that are created through the formation of water stable aggregates in the soil (Saifullah et al., 2018).

Organic materials such as mulch, farm manure, green manure, compost and other recyclable organic waste-residues and urban sewage sludge are used as organic amendments in the reclamation of sodic and/or saline-sodic soils (Leogrande and Vitti, 2019; Matosic et al., 2018; Muyen and Wrigley, 2016). Stabilized sewage sludge containing large amounts of organic matter can be used as a good soil amendment to improve the physical and hydraulic properties soil (Fan et al., 2016). Although sewage sludge is rich in organic and mineral content, it also has undesirable environmental risks due to heavy metals and organic pollutants (da Silva et al., 2007). Since metals are one of the main pollutants from wastes and have significant mobility, they can affect the ecosystems with the bioaccumulation processes (Janadeleh et al., 2018). Therefore, sewage sludge can easily create a significant environmental problem if it is not managed properly (Muyen and Wrigley, 2016).

Low saline waters (< 0.5 dS m^{-1} and especially < 0.2 dS m^{-1}) accelerate the leaching of soluble salts, especially Ca from the surface layer and excessive leaching of Ca greatly reduces aggregate binding and stability (Ayers and Westcot, 1985). The presence of salts in the soil solution reduces the osmotic gradient between the exterior and interior of the clay plate (Rao et al., 2006). Therefore, when sodic soils are treated with waters with a mild or moderate electrolyte concentration, generally clay plates remain intact and are not dispersed. The leaching of sodic soils with saline waters containing favorable levels of Ca (the ratio of the total of the divalent cations to total cations is at least 0.3) can lead to great increases in soil hydraulic properties due to the high electrolyte concentration of the saline water (Qadir et al., 2007). Koç (2011) and Marchuk (2013) emphasized that leaching water with a high electrolyte concentration has a positive effect on the permeability/infiltration of sodic soils. Furthermore, in a study carried out by Kameli et al. (2017) on the use of wastewater in the reclamation of saline-sodic soils, significant reductions in salinity and ESP values were observed.

The short-time reclamation of salt-affected lands with practical, sustainable and environmental practices has become inevitable due to the limited soil resources in the world compared to the population increase. It has been observed in previous studies that the application of organic amendment to sodic or saline-sodic soils can accelerate Na leaching and increase soil hydraulic properties and aggregation. Additionally, the use of wastewater with high electrolyte concentration from Ca and Mg concentrations in the leaching process can provide effective contributions to ameliorate soil without the use of freshwater. This study is one of a very limited number of studies in literature investigating the combined effects of liquid and solid materials from municipal waste plants on the amelioration of saline-sodic soil. It is also important to reduce environmentally negative impacts incurred by the disposal of wastewater and sewage sludge in the reclamation processes. In this study, the aim was to improve the performance of the reclamation process in saline-sodic soil treated with gypsum, by adding stabilized sewage sludge (treated by anaerobic and mesophilic processes) to the soil at certain doses and by applying recycled wastewater and freshwater combinations to maintain the electrolyte concentration. Therefore, the expectations of the current study were to firstly improve saline-sodic soil amelioration by sewage sludge, secondly to achieve an effective leaching process with wastewater, thirdly to reduce freshwater use, and fourthly to create an efficient, low cost and environmentally acceptable amelioration practice.

2. Material and methods

2.1. Experimental design and applications

A stand including 48 metal columns was designed in laboratory conditions for the experiment (Fig 1). The experiment was carried out with three replications using a completely randomized factorial design with four different stabilized sewage sludge doses (D) and four different leaching variants (L) with fresh and recycled wastewaters. Sewage sludge doses as the main treatments were selected as 0, 4, 8, and 12 kg m^{-2} in D1 (control), D2, D3, and D4 treatments, respectively, considering recommendations from previous studies by Ors et al. (2015) and Sahin et al. (2008). Leaching variants were applied as sub treatments as follows:
1) Recycled wastewater in three portions (L1);
2) Recycled wastewater in first and second portions and freshwater in third portion (L2);

3) Recycled wastewater in first portion and freshwater in second and third portions (L3);
4) Freshwater in three portions (L4).

Fig. 1. Experimental setup

Experimental soil was collected from the surface layer of a saline-sodic field in the Iğdır plain in eastern Turkey. The agricultural production area in this plain is 92200 ha and it has salinity-sodicity problems at different levels in one-third of the total area (Temel et al., 2016). Humidity and temperature regimes of the studied Typic Natrargid soil are aridic and thermic (Soil Survey Staff, 1999). The soil and sewage sludge properties prior to the experiment are given in Table 1.

The soil and sewage sludge were air dried in laboratory conditions and then sieved through a 1 cm mesh. Soil columns were prepared as shown in Fig. 1. The bottom of each column was padded with sand-gravel material to facilitate drainage. The sieved soil was fitted into the columns at a depth of 30 cm as two layers of 15 cm. Soil columns were tapped 25 times for every 15 cm of added soil (Sahin et al., 2011). Air-dry sewage sludge was mixed to the top 15 cm layer of the column at selected doses. The soil columns were wetted with capillarity using freshwater and the columns isolated from atmospheric conditions were incubated for one month. The theoretical gypsum requirement (6.19 kg m^{-2}) according to the final ESP value of 10% was calculated with the equation given by Lebron et al. (2002), and the finely ground gypsum (< 0.25 mm) was incorporated into the surface soil layer of 2-3 cm (Munsuz et al., 2001). In many studies, water amounts equal to 1.5 times the soil depth to be ameliorated have been considered in the leaching process as a practical approach (Ors et al., 2015; Örs and Anapalı, 2014; Sahin et al., 2003; 2011). Therefore, a total of 45 cm of leaching water was applied to soil columns as three equal portions with an intermittent ponding technique (Sahin et al., 2002). This technique requires less water than the continuous ponding approach to achieve the same amelioration rates (Ayers and Westcot, 1985; Koç, 2011; Sahin et al., 2003). Kanber and Ünlü (2010) stated that the water requirement in the intermittent ponding technique is one-third of the amount applied in continuous ponding. The next portion application was carried out 24 hours after the previous portion of water was fully infiltrated into the soil column (Ors et al., 2015; Sahin and Anapali, 2005).

Table 1. Quality analysis results of the experimental soil and stabilized sewage sludge

Parameter	Soil	Sewage Sludge
Clay (%)	29.3	-
Silt (%)	23.3	-
Sand (%)	47.4	-
Texture	Sandy clay loam	-
Wet aggregate stability (%)	2.20	-
pH[#]	9.58	7.89
Electrical conductivity[#] (dS m^{-1})	29.60	8.97
CaCO$_3$ (%)	7.92	1.47
Organic matter (%)	1.43	44.0
Cation Exchange capacity (cmol kg^{-1})	31.53	60.62
Exchangeable Na (cmol kg^{-1})	21.60	37.78
Exchangeable Ca+Mg (cmol kg^{-1})	9.18	14.0
Exchangeable K (cmol kg^{-1})	0.75	8.84
Exchangeable sodium percentage (%)	68.51	-
Total N (%)	0.38	4.55
P (mg kg^{-1})	277.2	877.6
Micro element and heavy metals (mg kg^{-1})		
B	0.15	11.2
Fe	2.33	146.7
Mn	35.5	131.1
Zn	1.73	201
Cu	9.23	5.77
Ni	0.66	5.71
Pb	0.77	1.88
Cd	-	0.01

[#] *in saturation extract*

The stabilized sewage sludge and recycled wastewater were provided from the biological wastewater treatment plant of Erzurum Metropolitan Municipality in Turkey (39.956° N and 41.092° E). The stabilization process was carried out with a sludge-decaying tank in anaerobic and mesophilic conditions (at 35-37° C) with the activity of acetogen and methanogen bacteria (ESKI, 2016). The municipal wastewater used includes mostly domestic waste and a small amount of meat and dairy industrial wastes.

2.2. Analysis, measurement and evaluations of water, soil and sewage sludge

The pH and electrical conductivity (EC) of the leaching waters was measured directly by using a pH-meter and an EC-meter (Tüzüner, 1990). Total N was determined by the Kjeldahl method (EPA, 2001), total P by orthophosphate measurement (Nollet, 2000), Ca+Mg by EDTA titration, Na and K with a flame photometry, B by Carmin method (Eltan, 1998), CO_3 and HCO_3 by sulfuric acid titration, Cl by using silver nitrate (Tüzüner, 1990), SO_4 with a spectrophotometer using barium chloride solution (Eltan, 1998). Micro elements and heavy metals (Fe, Mn, Zn, Cu, Ni, Pb, and Cd) were measured with an atomic absorption spectrometer (Karadede and Ünlü, 2000). Suspended solid material was determined by dry-weighing material accumulated on the Whatman filter paper (APHA, 1995). The sodium adsorption ratio (SAR) was calculated from Na, Ca and Mg concentrations (Kanber and Ünlü, 2010).

The leaching water quality properties are shown in Table 2. The pH, EC and SAR values and micro element and heavy metal contents in the used water are within suitable limits according to the FAO irrigation water quality standards (pH 6.5–8.4, EC < 0.7 dS m^{-1}, SAR < 3, B < 0.7 mg l^{-1}, Fe < 5 mg l^{-1}, Mn < 0.20 mg l^{-1}, Zn < 2 mg l^{-1}, Cu < 0.20 mg l^{-1}, Ni < 0.20 mg l^{-1}, Pb < 5 mg l^{-1}, and Cd < 0.01 mg l^{-1}) (Ayers and Westcot, 1985). The water qualities for micro element and heavy metal contents are first class according to the limit values (Fe < 0.3 mg l^{-1}, Mn < 0.10 mg l^{-1}, Zn < 0.2 mg l^{-1}, Cu < 0.02 mg l^{-1}, Ni < 0.02 mg l^{-1}, Pb < 0.01 mg l^{-1}, and Cd < 0.002 mg l^{-1}) given in the national above-ground water quality regulation in Turkey (The Official Gazette, 2015). However, total N (1.5–5 mg l^{-1}), and P (> 0.65 mg l^{-1}) contents in recycled wastewater were high and the recycled wastewater quality was commensurate with third and fourth class for total N and P concentrations, respectively. Saturated hydraulic conductivity (HC) measurements in all soil columns were done under 15 cm constant water head for 6, 12, 24 and 48 h, and outflow volumes were recorded for the test times. The HC (cm h^{-1}) was calculated by the Darcy approach given below (Sahin et al., 2011) (Eq. 1).

$$HC = \frac{V}{A \times t} \times \frac{L}{L+H} \qquad (1)$$

where: V is the outflow volume (cm^3); A is the cross-sectional surface area of the soil column (cm^2); t is elapsed time (h); L is the height the soil column (cm); H is the height of water ponded on the soil surface (cm).

Table 2. Chemical and physical properties of the waters used in the leaching process

Parameter	Freshwater	Recycled wastewater
pH	7.24	6.68
Electrical conductivity (dS m^{-1})	0.345	0.644
Suspended solid material (mg l^{-1})	0.01	2.90
Cations (me l^{-1})		
Na	0.68	3.02
K	0.10	0.38
Mg	0.73	0.91
Ca	1.91	2.26
Anions (me l^{-1})		
CO$_3$	-	-
HCO$_3$	2.50	3.25
Cl	0.42	1.75
SO$_4$	0.48	1.21
Total N (mg l^{-1})	–	4.67
Total P (mg l^{-1})	–	1.68
Sodium adsorption ratio	0.59	2.40
Micro element and heavy metals (mg l^{-1})		
B	-	0.289
Fe	0.041	0.374
Mn	0.008	0.045
Zn	-	0.009
Cu	-	0.010
Ni	-	0.018
Pb	-	0.005
Cd	-	0.001

After the HC measurements, soil columns were sampled in two layers, the top layer of 0-15 cm (added sewage sludge) and the sub layer of 15-30 cm. Soil pH was measured in the saturation extract with a pH-meter (McLean, 1982). An EC-meter was used to measure the EC in the saturation extract (Rhoades 1996). Organic matter was determined by the Smith-Weldon method (Nelson and Sommers, 1982), CaCO$_3$ with a Scheibler Calcimeter (Nelson, 1982), total N by the Kjeldahl method (Kacar, 2009), and P by the Olsen method (Olsen and Sommers, 1982), Na and K using a flame photometer (Richards, 1954), and cation exchange capacity (CEC) was determined by the ammonium acetate method (Rhoades, 1982). Exchangeable Ca+Mg was determined by subtracting exchangeable Na and K concentrations from the CEC (Anapalı, 1994). All micro element and heavy metal (B, Fe, Mn, Zn, Cu, Cd, Pb, Ni) measurements were done by an ICP- MS (Kacar, 2009). Exchangeable sodium percentage (ESP) was calculated by Eq. (2) where the concentrations of exchangeable cations are expressed in cmol kg^{-1} (Mao et al., 2016) (Eq. 2).

$$ESP = \frac{Na}{Na + Ca + Mg + K} \times 100 \qquad (2)$$

The sodium removal efficiency was calculated by Eq. (3) (Mahdy, 2011).

$$Sodium\ removal\ efficiency = \frac{ESP_i - ESP_f}{ESP_i} \times 100 \qquad (3)$$

where: ESP_i is the initial ESP value (%); ESP_f is the final ESP value (%).

Particle size distribution of soil used was determined by the Bouyoucos hydrometer method (Gee and Bauder, 1986), and aggregate stability was determined with a wet sieving apparatus described by Yoder (stroke length = 12.7 mm and frequency = 42 cycles min[-1]) (Kemper and Rosenau, 1986). Stabilized sewage sludge properties were determined by the methods used in soil analysis.

Measured data were evaluated by variance analysis (ANOVA) in the SPSS (Ver. 21) program and means were compared with Duncan multiple range test.

3. Results and discussion

Although wastewater has higher concentrations of cations and anions than freshwater as shown in Table 2, both waters used were within a suitable class for irrigation in terms of pH, EC and SAR values (Ayers and Westcot, 1985). Therefore, remarkable results could not be determined for the soil properties investigated with different leaching variants with these waters.

These findings clearly indicated that the water quality differences at the levels shown in Table 2 would not result in significant differences in the leaching process. On the contrary, stabilized sewage sludge application in increased doses resulted in significant changes in soil properties (Table 3). The differences between the values of the top and sub layers were also significant.

3.1. Soil pH and electrical conductivity (EC)

Soil pH values decreased below the initial values shown in Table 1 after the trial, and the top layer values were lower than the sub layer values. The adding of sewage sludge significantly decreased soil pH values in the top layer compared to the control (D1), while the lowest value in the sub layer was obtained with the highest dose (Table 3).

Although pH values in the top layer firstly decreased with the increase of the dose and then increased, the values decreased with the dose increase in the sub layer. Significant quadratic and linear relationships in the top and sub layers were obtained with pH decrease and organic matter increase (Fig. 2). Similar to our findings, Mazen et al. (2010) determined that organic matter increased with the addition of sewage sludge while pH value decreased. Domene et al. (2010) also observed that the pH in some alkaline soils applied treated with sewage sludge with high organic matter at higher doses trended towards neutral.

In addition, Khurana and Singh (2012), Kiziloglu et al. (2008) and Singh and Agrawal (2012) stated that different organic compounds can decrease soil pH values.

Table 3. Soil properties (mean±SEM) after trial in different stabilized sewage sludge doses as the mean of leaching variants

Property	Depth (cm)	Sewage sludge doses				Mean
		D1	D2	D3	D4	
pH[#]	0-15	8.42±0.01 a**	7.94±0.09 b	7.73±0.13 b	8.05±0.01 b	8.03 **
	15-30	9.26±0.05 a*	9.05±0.11 a	9.02±0.09 a	8.82±0.18 b	9.04
EC[#] (dS m[-1])	0-15	6.41±0.09 c**	6.27±0.12 bc	7.04±0.18 b	8.59±0.31 a	7.07 **
	15-30	3.59±0.54	4.10±0.36	4.63±0.43	4.44±051	4.19
OM (%)	0-15	1.81±0.04	2.06±0.01	2.16±0.17	2.46±0.11	2.12 **
	15-30	1.14±0.05 b**	1.68±0.09 a	1.99±0.05 a	2.07±0.10 a	1.72
CaCO₃ (%)	0-15	4.24±0.16 c**	5.17±0.21 b	5.21±0.11 b	6.36±0.21 a	5.25 **
	15-30	5.41±0.30 b**	5.86±0.27 b	6.20±0.22 ab	7.04±0.14a	6.13
Exc. Na (cmol kg[-1])	0-15	11.55±0.61 a**	7.84±0.39 b	7.56±0.51 b	8.58±.021 b	8.88 **
	15-30	16.11±0.50 a*	14.63±0.13 b	16.46±0.64 a	15.77±0.56 ab	15.74
Exc. Ca+Mg (cmol kg[-1])	0-15	15.76±0.71 c**	19.61±0.39 b	20.95±0.50 ab	22.11±0.43 a	19.61 **
	15-30	13.29±0.27	13.97±0.15	13.28±0.66	14.92±0.41	13.86
Exc. K (cmol kg[-1])	0-15	1.64±0.02 b**	1.60±0.01 b	1.72±0.01 a	1.73±0.02 a	1.67
	15-30	1.69±0.004 a *	1.65±0.01 b	1.62±0.02 b	1.69±0.02 a	1.66
CEC (cmol kg[-1])	0-15	28.95±0.30 c**	29.04±0.34 c	30.24±0.11 b	32.42±0.39 a	30.16 **
	15-30	31.08±0.33 bc**	30.24±0.20 c	31.35±0.19 b	32.37±0.24 a	31.26
ESP (%)	0-15	39.92±2.14 a**	26.97±1.24 b	25.01±1.67 b	26.47±0.70 b	29.59 **
	15-30	51.79±1.17	48.38±0.32	52.49±2.05	48.68±1.47	50.34
Wet aggregate stability (%)	0-15	3.68±0.49 b*	5.45±0.31 a	5.25±0.25 a	4.96±0.69 a	4.83 **
	15-30	0.67±0.05	0.53±0.03	0.48±0.06	0.57±0.13	0.56

SEM: Standard error of means; D1, D2, D3, and D4: 0, 4, 8, and 12 kg m[-2], respectively; ECe: Electrical conductivity; OM: Organic matter; CEC: Cation exchange capacity; ESP: Exchangeable sodium percentage; [#]: in saturation extract; The means marked with different lower-case letters in each row are different significantly (*p<0.05 or **p<0.01)

The mineralization of organic matter results in the formation of organic and inorganic acids, which reduces soil pH (Özyazıcı et al., 2012; Singh and Agrawal, 2008). A study by Hussein (2009) revealed that soil pH value decreased with the increase of the sewage sludge dose due to increased organic acids from mineralization of organic matter and improved the nitrification of ammonium.

Changes in soil pH could be also related to the $CaCO_3$ concentration (Singh and Agrawal 2008). The results of this study showed that $CaCO_3$ concentration decreased after the trial (Table 3) compared to the soil initial concentration (7.92%) given in Table 1. Concerning the influence of soil $CaCO_3$ content on the soil pH, Mazen et al. (2010) expressed that lower pH values could be the result of both $CaCO_3$ dissolution and organic acids formation. Leogrande and Vitti (2019) reported that the formation of organic acids during the organic matter decomposition lowers the pH and as a consequence increases soil native $CaCO_3$ dissolution in soil. Similarly, Fan et al. (2016) observed significant decreases in the pH of the saline-sodic soil in which the waste with sewage sludge was applied at high doses, and the reduction was related to the reduction of $CO_3 + HCO_3$ concentration in soil.

The EC values of the soils were significantly reduced considering the initial soil EC value (29.60 dS m^{-1}), and salinity limit value (4 dS m^{-1}) was reached in the sub layer (Table 3). Despite this general reduction in the EC, the EC values in the top layer of the soil increased in proportion with the sewage sludge dose (salinity level 8.97 dS m^{-1}) and the highest EC value was obtained with the highest dose. Therefore, a significant ($p<0.01$) quadratic relationship was determined which expresses the increases of EC with increased doses in the top layer (Fig. 3).

Although salinity increase with dose increase was determined in the sub layer, this increase was not significant (Table 3). This finding is compatible with the results of many researchers who observed an increase in EC with the increase of sewage sludge doses in their studies (Domene et al., 2010; Ferreira et al., 2011; Hussein 2009; Lakhdar et al., 2010).

3.2. Organic matter and $CaCO_3$ contents

The sewage sludge with high organic matter content (44%) added to the soil provided an increase in organic matter content compared to the initial value (1.43%), the values were significantly higher in the top layer compared to the sub layer (Table 3). A significant quadratic increase in the organic matter content ($p<0.01$) with increasing doses in the top layer was observed, while the increase speed slowed with the dose increase in the sub layer (Fig. 4).

Higher organic matter accumulations have been observed with increasing application doses in many studies that have used sewage sludge (Hussein, 2009; Mazen et al, 2010; Muyen and Wrigley, 2016; Salhab, 2014). Although the content of organic matter in the surface layer increased based on an increase in the dose, it is probable that the mineralization of organic matter limited more accumulation (Angın and Yağanoğlu, 2009).

Similarly, Li et al. (2013) determined an increase in the organic matter content with the increase of sewage sludge amount at the beginning, but this increase did not stop and then decreased sharply due to rapid mineralization and nitrification that occurred during incubation. Higher $CaCO_3$ contents were found in the columns with sewage sludge compared to the control.
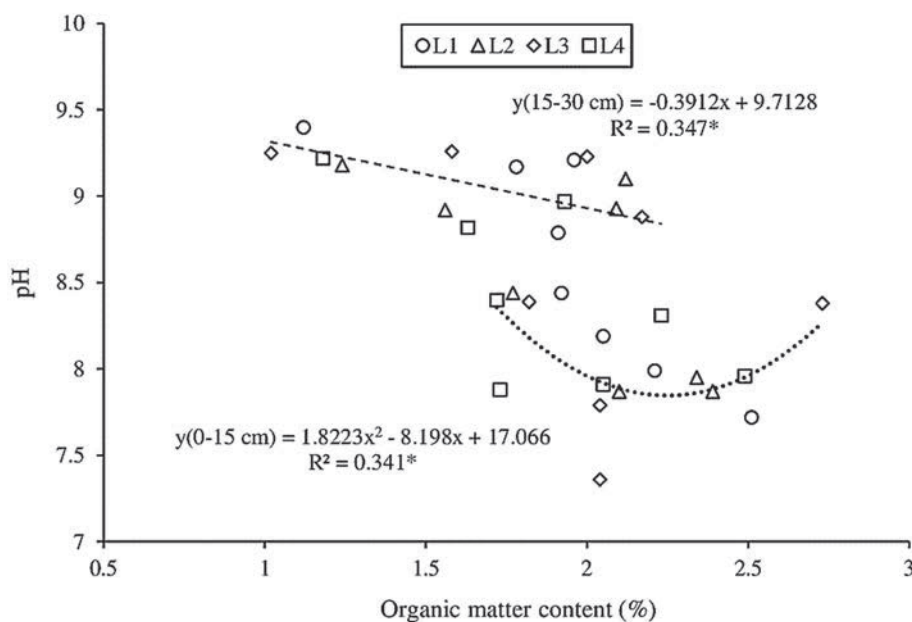

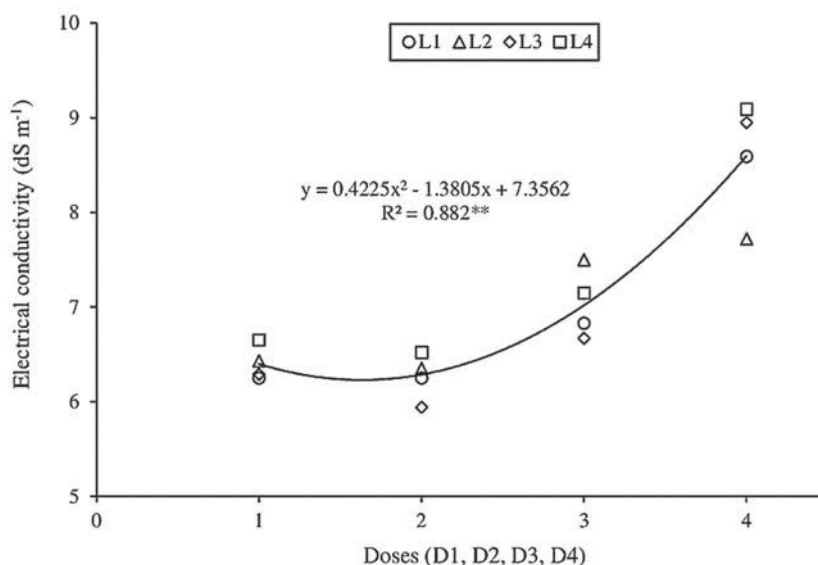**Fig. 2.** pH–organic matter content relationships in two soil layers (*$p<0.05$)

Fig. 3. Change in electrical conductivity in the top layer (0-15 cm) by stabilized sewage sludge dose (**p<0.01)
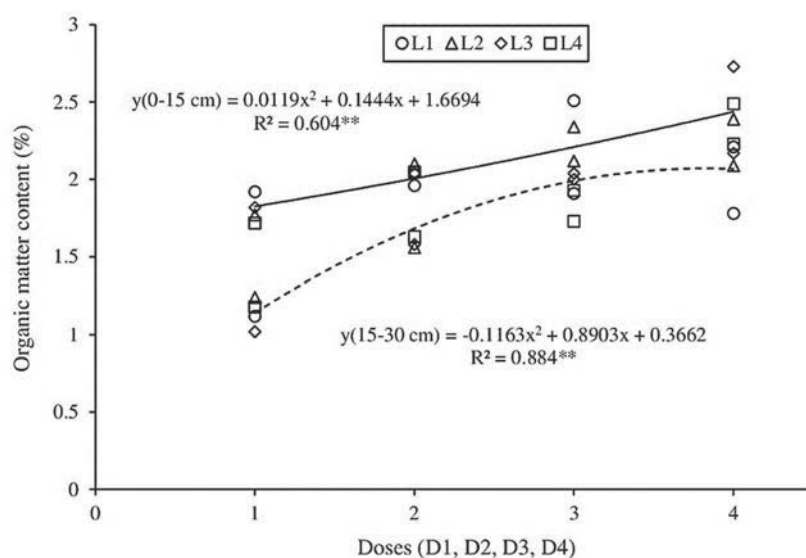
Equation shown on Fig. 3:

$$y = 0.4225x^2 - 1.3805x + 7.3562$$
$$R^2 = 0.882^{**}$$

Fig. 4. Change in organic matter content by stabilized sewage sludge dose in two soil layers (**p<0.01)

Equations shown on Fig. 4:

$$y(0\text{-}15\ cm) = 0.0119x^2 + 0.1444x + 1.6694$$
$$R^2 = 0.604^{**}$$

$$y(15\text{-}30\ cm) = -0.1163x^2 + 0.8903x + 0.3662$$
$$R^2 = 0.884^{**}$$

The highest contents were determined in the highest dose for both layers as well, and the contents were significantly (p<0.01) higher than the values of control treatment. However, significant reductions in $CaCO_3$ contents were observed after the application considering the initial value (7.92%) (Table 3). This could be explained by the increase in $CaCO_3$ solubility due to decreased pH. Parallel to our results, Abdel-Fattah (2012) stated that the addition of organic materials to saline-sodic soils may lead to an increase in the solubility of $CaCO_3$ due to the decrease in soil pH. Lower pH values in the surface layer also supports this because the $CaCO_3$ content in the surface layer was significantly lower than that of sub layer. In addition, higher $CaCO_3$ contents in the sub layer could also be a result of the leaching-precipitation process from the surface layer. The determination of the highest $CaCO_3$ contents in sewage sludge treatment at the highest dose in both layers showed that adding sewage sludge, contributed to $CaCO_3$ content at higher doses despite the low lime content (1.47%).

3.3. Exchangeable cations, cation exchange capacity (CEC) and exchangeable sodium percentage (ESP)

The exchangeable Na contents in the top layer decreased below the pre-trial value (21.60 cmol kg^{-1}), and higher reductions in the treatments with sewage sludge were statistically different from that of the D1 value (Table 3). Higher levels of exchangeable Ca+Mg contents were found in the treatments with sewage sludge in the top layer compared to the D1 dose, and these values also were higher than the initial value (9.18 cmol kg^{-1}). Sodium and Ca+Mg contents

in the top layer showed significant quadratic changes with the dose increase (Fig. 5). While the exchangeable Ca+Mg content increased with the dose increase, Na content firstly decreased and then increased with the highest dose. Sewage sludge lowered exchangeable Na content in the sub layer, but increasing doses caused similar results with control treatment (Table 3). On the other hand, the increase trend in Ca+Mg contents slowed down with the dose increase.

From the trends in Fig. 5 it is observed that the increase in Na from the sewage sludge with a high amount of Na content (37.78 cmol kg^{-1}) could not be completely suppressed by the Ca+Mg increase in higher doses. However, sewage sludge application has made significant contributions to reducing Na and increasing Ca+Mg. Similarly, Tamanini et al. (2008) found an increase in soil Ca content with the increase of sewage sludge dose and this changing trend was defined by a quadratic relationship. Increased Ca + Mg content in the top layer in our study could be explained by the considerable gaining of Ca and Mg from gypsum and sewage sludge. During the decomposition of organic matter, cations such as Ca, Mg and K are released (Abdel-Fattah, 2012). Jalali and Ranjbar (2009) and Leogrande and Vitti (2019) stated that the application of organic materials to soil causes adsorption of cations such as Ca, Mg and K at higher rates compared to Na.

As can be seen from Table 3, the decrease of soil CaCO$_3$ content after the trials showed that Ca content can also be increased by calcite dissolution (Qadir et al., 2003). As a result of the increase of Ca in the surface soil, the increase in the amount of Na released from the cation exchange complex causes Na removal by leaching (Alcívar et al., 2018). Therefore, it can be said that the leaching from the top layer was an important reason for the Na accumulation in the sub layer. After the applications, the exchangeable K content was higher than the initial value (0.75 cmol kg^{-1}). Although higher contents were found in higher

sewage sludge doses in the top layer, the contents were similar statistically in the two soil layers. The contents in the sub layer were similar for D1 and D4 doses statistically, while K contents in the D2 and D3 doses were lower than the D1 dose (Table 3).

Higher K contents under higher doses in the top layer can be the result of the K content (8.84 cmol kg^{-1}) of the added sewage sludge. However, although an increase was observed from sewage sludge application, its contribution was small. Parallel to our finding, Lakhdar et al. (2010) observed non-significant changes or minor increases in the soil K content under different application doses of sewage sludge. Bilen and Sezen (1993) stated that lime, pH, and some other properties of soil affect the release or retention of potassium. Moreover, Kundu et al. (2014) found positive and significant correlations between the content of K with the CEC, clay and silt content, while they found a negative correlation with the sand content. Ghiri et al. (2010) also determined significant correlations between exchangeable K and CaCO$_3$ content, clay content and CEC. They concluded that while K content is greater in soils with higher CaCO$_3$ content, the leaching of K is also greater in these soils. Similarly, Jalali and Rowell (2003) expressed that calcite and gypsum have an induced effect on both the K content increase in soil solution and the leaching of this element from soil.

The CEC values in the top layer were significantly lower than in the sub layer, and all values except for D4 application were below the initial value (31.53 cmol kg^{-1}) (Table 3). A dosage increase raised the CEC values in both soil layers significantly ($p<0.01$), and the highest CEC value was obtained with the highest dose.

Organic matter content in soil is one of the main components that affect the CEC due to having negative charges such as clay (Esmaeilzadeh and Ahangar, 2014). Therefore, the increase in CEC with the dose increase was likely due to the increase of organic matter content in our study (Table 3).
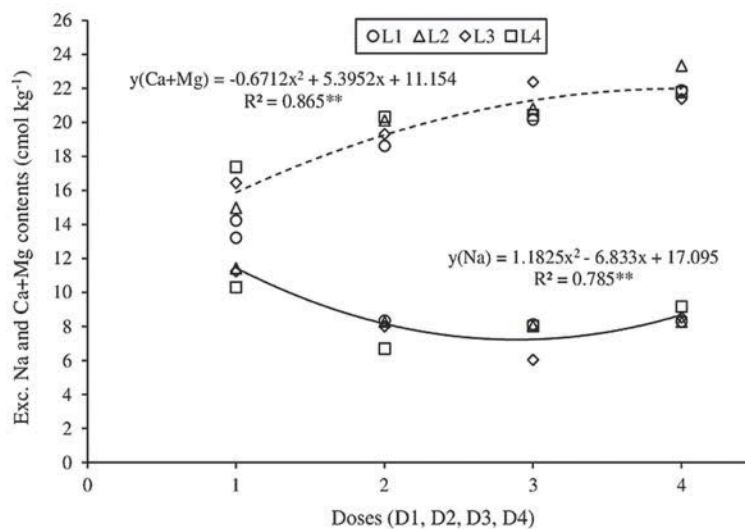

Fig. 5. Change in exchangeable Na and Ca+Mg contents in the top layer (0-15 cm) by stabilized sewage sludge dose (**$p<0.01$)

Parallel to this finding, Singh and Agrawal (2008) expressed that the application of partially high doses of sewage sludge increased the CEC together with total organic carbon. Lower CEC values in the top layer could be explained by the lower pH values (Table 3). Low pH neutralizes the negative charge of colloids, and thereby decreases CEC (Esmaeilzadeh and Ahangar, 2014). Tomašić et al. (2013) stated that soil pH is positively correlated with CEC because a higher pH increases the numbers of negative charges on the colloids. Similarly, Domene et al. (2010) and Jaramillo and Restrepo (2017) indicated that the change in soil pH affects the CEC. Although the increase in CEC in the sub layer could be explained by the increase in organic matter with dose increase, this result could also be supported with the transport and accumulation of clay to the sub layer (Sezen, 1995). Our observations indicated that after leaching, there was a siltation in the sand-gravel material in the column bottom. According to our visual observations, approximately one-third of the porosity in the sand-gravel media was filled with residue.

ESP values were found significantly higher in the sub layer with higher Na content compared to the top layer (Table 3). The effects of sewage sludge doses on the ESP in the sub layer were non-significant and the sodium removal efficiency varied between 23.4 and 29.4% (Fig. 6). Although different sewage sludge doses had shown similar effects in the top layer, the treatments with sewage sludge reduced the ESP values significantly compared to the D1 treatment. Sodium removal efficiencies in the top layer were realized with 41.7%, 60.6, 63.5 and 61.4% in D1, D2, D3 and D4 treatments, respectively (Fig. 6). This finding for the top layer could be caused by increased Ca content from gypsum and sewage sludge, and the considerable removal of Na by leaching (Tables 1 and 3). Gypsum is an important source of Ca which can be easily obtained from the soil solution that replaces exchangeable Na (Qadir et al., 2003). Jalali and Ranjbar (2009) have also stated that mixing organic material increases Na removal and provides lower ESP values.

As a result of the applications of organic materials decreases in ESP values have been observed in many different previous studies as well (Ors et al., 2015; Tejada et al., 2006; Yaduvanshi and Swarup, 2005).

3.4. Wet aggregate stability

Wet aggregate stability values were significantly higher in the top layer than in the sub layer, and the top layer values were found to be more than double the initial value (2.20%) while they were approximately four times less in the sub layer (Table 3). The effect of the treatments in the sub layer was similar. However, a quadratic relationship was found between wet aggregate stability and sewage sludge doses in the top soil layer (Fig. 7).

The aggregate stability values significantly increased from the application of sewage sludge and gypsum to the top layer. The Ca ions from $CaCO_3$ dissolution also can be the cause of better aggregation. Gutiérrez et al. (2016) expressed that Ca prevented clay dispersion and developed the structure by forming a cationic bridge between clay particles and soil organic matter. Haynes and Naidu (1998) indicated that high Ca contents can result in the compression of the double layer thereby reducing the repulsive force between soil colloids.

As can be seen from Fig. 4, the aggregate stability initially increased with the dose increase then decreased (Fig. 7). Parallel to our findings, Yılmaz et al. (2005) reported that although the increase of organic matter increased aggregate stability, the stability values when organic matter content exceeded 2% did not increase significantly. The same researchers also stated that $CaCO_3$ content is another factor affecting aggregate stability, and that Ca has a positive contribution to aggregate stability by bridging soil colloids and increasing biological activity. As a supporting finding, Haynes and Naidu (1998) reported that microbial biomass concentration, soil respiration rate, enzyme activity and microbial metabolic quotient have been increased with liming.
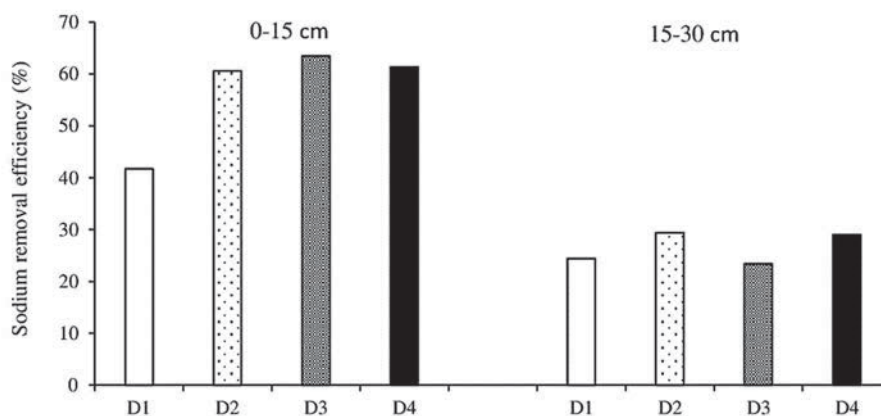

Fig. 6. Change in sodium removal efficiency by stabilized sewage sludge dose in two soil layers as the mean of leaching variants

Increased biological activity produces gelatinous polysaccharides which act as binding agents in soils, and cause an increase in aggregate stability. Moreover, fungal hyphae can create a network of enmeshing hyphae to promote aggregation. It was stated by Yılmaz et al. (2005) that cationic polymers provide a negative electrostatic charge balance on clay minerals and thus coagulating properties.

In many different studies conducted in the past, it has been indicated that the addition of organic matter to soil increased aggregate stability. In one of the studies, Hanay et al. (2004) stated that the addition of municipal solid waste compost following the gypsum application into saline-sodic soil provided significant increases in the aggregate stability with the increase of organic matter. In another study, Sahin et al. (2008) found that mixing sewage sludge into saline-sodic soils reduced the negative effects of freeze-thaw processes by increasing wet aggregate stability. Similarly, Annabi et al. (2011) found that different organic materials appeared to have a positive effect on the aggregate stability of silty soil with the increase of organic carbon content. Therefore, major effectiveness of the municipal solid waste in increasing soil organic C levels could be explained with the biodegradability of this material that directly stimulated soil microbial activity.

However, increase of ESP and decrease in the electrolyte concentration significantly decreased the aggregate stability in the sub layer compared to the top layer (Table 3). Similarly, Sahin et al. (2008) indicated that soils with high ESP and low EC have weak aggregate stability value.

3.5. Hydraulic conductivity

The values measured during the 6, 12, 24- and 48-hour periods showed that adding sewage sludge reduced the hydraulic conductivity in high doses (Fig.

8). However, the values measured in the columns with sewage sludge were not statistically different from the no sludge value. Moreover, the values measured in increased time periods increased also, but the reduced trend with dose increase did not change.

This finding indicated that adding sewage sludge to only the top layer could not have contributed positively to water permeability. It could be said that the hydraulic conductivity was negatively affected by weak aggregate stability in the sub layer from the lower electrolyte concentration and higher ESP. Therefore, the positive contributions of lower ESP and higher aggregate stability values in the top soil layer could not be observed for the entire soil column. Remarkably, Ors et al. (2015) determined that a sub layer with higher ESP had lower hydraulic conductivity values in saline-sodic soils compared to the top layer which had been treated with sewage sludge and fly ash materials.

Similarly, Dikinya and Totolo (2008) expressed that an increase in sodicity and decrease in electrolyte concentration in saline-sodic soil resulted in a decrease in permeability. Also, Sahin et al. (2002) determined that although salinity after leaching in saline-sodic soil reached low levels, hydraulic conductivity decreased from increased ESP with increased soil depth.

The possible changes in the pore size distribution with the dose increase should also be considered in decreasing hydraulic conductivity. Ferreira et al. (2011) asserted that electron microscope images indicated that the covering of the surface of sand grains with small particles originating from sewage sludge reduced hydraulic conductivity. They observed a small reduction in hydraulic conductivity with increasing sewage sludge doses. Jnad et al. (2001) found a decrease in hydraulic conductivity due to the decrease in the volume of large pores as a result of the accumulation of organic matter in pores because of wastewater applications
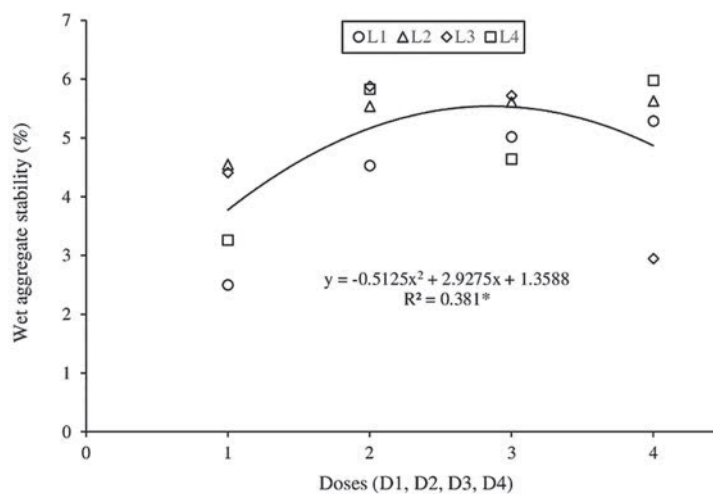
The plotted equation shown in the figure:
$$y = -0.5125x^2 + 2.9275x + 1.3588$$
$$R^2 = 0.381^*$$

Fig. 7. Change in wet aggregate stability in the top layer (0-15 cm) by stabilized sewage sludge dose (*p<0.05)
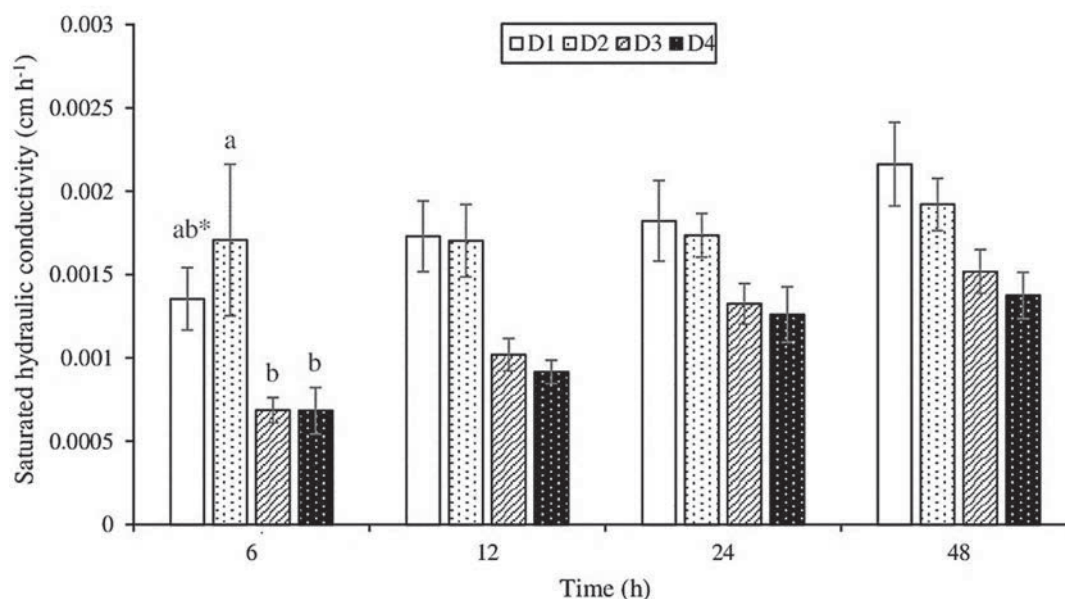
Fig. 8. Change in saturated hydraulic conductivity values by stabilized sewage sludge doses at different measurement times as the mean of leaching variants (*p<0.05)

In another study, Schneider et al. (2009) indicated that the hydraulic conductivity decrease could possibly be explained by the increase in hydrophobicity that causes resistance to water permeability and the occlusion of pores with coarse organic matter. Therefore, it can be considered from the above information that the addition of bio-waste in high sodicity conditions initially creates a contraction in the pores. The observed increase trend in hydraulic conductivity over time could be evaluated with this idea (Fig. 8). As a supporting finding, a study revealed that sludge application generated a significant increase in hydraulic conductivity after incubation for 27 days (Singh and Agrawal, 2008).

3.6. Total N and P contents

Contents of total N in the top layer were found to be significantly higher than the values in the sub layer (Fig. 9). In both layers, only the highest sludge dose increased the total N content significantly, while the effect of other applications was statistically similar, and also the total N content of these applications was below the initial value (0.38%). The increase caused by the highest dose in both layers revealed a significant quadratic relationship (Fig. 9).

Leaching is the main factor in the transport of N in soil (Favaretto et al., 2012). Therefore, it can be said that while the increase in the highest dose could be caused by the total N content (4.55%) of the sewage sludge, the leaching effect is more dominant in lower doses. Similarly, the results of Lakhdar et al. (2010) showed that sewage sludge induced significant increases of N content due to its high N content, especially in treatments with higher doses. In a soil column study, Fernández-Sanjurjo et al. (2014) observed that high amounts of ammonium and nitrate

nitrogen were discharged from the columns within a few days by leaching. In their study of nitrogen transport in different irrigation methods, Özçelik and Usta (2008) found that the loss of nitrogen in the form of ammonium and nitrate was very high with the first irrigation. In addition, Sato and Morgan (2008) reported a high ratio of ammonium and nitrate nitrogen discharges at the beginning in the leaching columns.

Phosphorus contents in the top layer were determined to be significantly (p<0.01) higher than in the sub layer (Fig. 10). While we observed a significant (p<0.01) linear increase between P content with dose increase in the sub layer, the dose increases for the top layer had no effect on the P content. Moreover, the P contents in the soil columns after applications were lower than the initial value (Table 1). Although sewage sludge had high P content (877.6 mg kg[-1]), lower P contents from the prior value (277.2 mg kg[-1]) in Table 1 could be evaluated as the result of discharge with leaching. The mobility of P is low compared to other nutrients, but it increases under continuous P application (Jalali and Arfania, 2010). Fernández-Sanjurjo et al. (2014) indicated that phosphorus transport, as in nitrogen, was significantly affected by leaching applications. Many studies have demonstrated that sewage sludge has contributed to the increase in soil N and P contents under no-leaching or limited leaching conditions. Singh and Agrawal (2008) reported that the application of treatment sludge increased the N and P content of soils. Ferreira et al. (2011) observed that the increasing sewage sludge doses increased the content of P in the column surface layer and the N content was higher in the sub layer. Hussein (2009) determined that the available P content increased with the increase of sewage sludge doses in two sandy and calcareous soils.
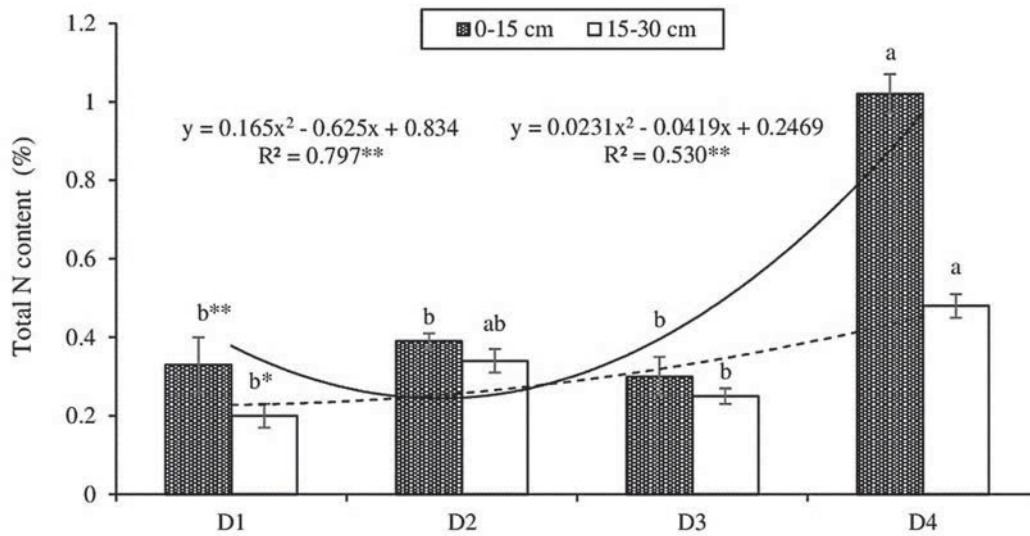
Fig. 9. Change in total N content by stabilized sewage sludge doses in two soil layers as the mean of leaching variants (**$p<0.01$; *$p<0.05$)
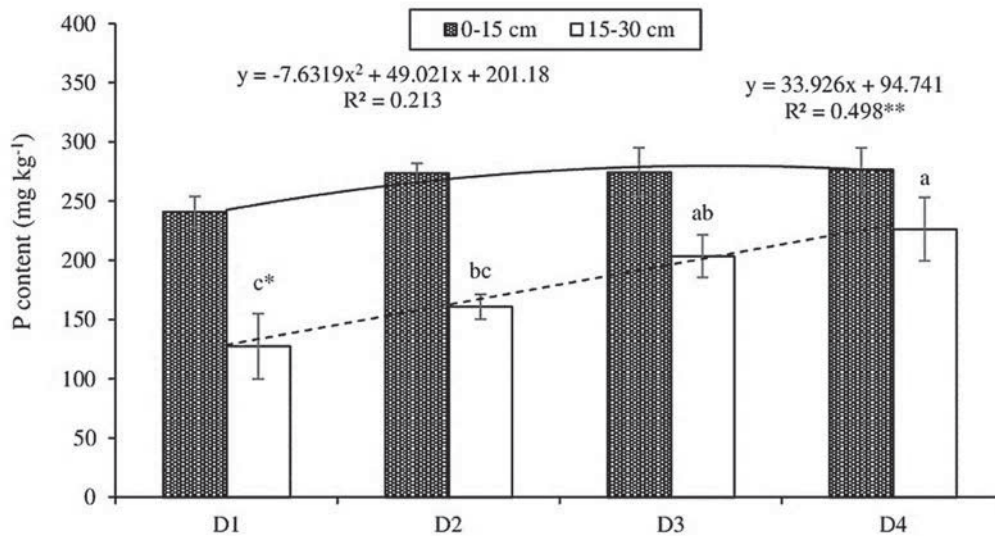

Fig. 10. Change in soil P content by stabilized sewage sludge doses in two soil layers as the mean of leaching variants (**$p<0.01$; *$p<0.05$)

3.7. Micro element and heavy metal contents

Adding sewage sludge increased the microelements and heavy metal contents (Fig. 11). The sub layer values, except Zn content, were significantly higher than the top layer values. Cd was not detected due to low initial values (soil: 0 mg kg^{-1}, sewage sludge: 0.01 mg kg^{-1} and recycled wastewater: 0.001 mg kg^{-1}) in the soil samples after the experiment. The contents of Zn and Cu decreased and the other elements increased considering initial values (Table 1). Micro element and heavy metal accumulations observed in the soil after sewage sludge applications could be interpreted as an effect of the contents found in the sewage sludge (Table 1). The increases in the sub layer could be explained with the effect of leaching-accumulation. Under leaching conditions, metals added to soils in applications of sewage sludge can be leached downwards through the soil profile and also leaching can also completely remove the contaminants and hence ensure a rapid cleanup (Wuana and Okieimen, 2011).

The values in this study were quite below the limit values for soils with pH > 7 considering the values given in the Turkey National Regulation on use of domestic and municipal sewage sludge in soil (Zn: 200 mg kg^{-1}, Cu: 100 mg kg^{-1}, Cd: 1.5 mg kg^{-1}, Ni: 70 mg kg^{-1}, Pb: 100 mg kg^{-1}) (The Official Gazette,

2010). This regulation covers the technical principles for the controlled use of sewage sludge, so as not to harm soil, plants, animals and people. The WHO limit values (Zn: 300 mg kg^{-1}, Cu: 140 mg kg^{-1}, Cd: 3 mg kg^{-1}, Ni: 75 mg kg^{-1}, Pb: 300 mg kg^{-1}) also showed similar results (Khan et al., 2013).

In addition, B content (< 1 mg kg^{-1}) did not reach pollution risk level (Gupta, 2007). From previous studies, it has been observed that adding sewage sludge enabled significant changes in the soil micro element and heavy metal contents. However, the application dose of sewage sludge is important. Singh and Agrawal (2008) stated that the bioavailability of heavy metals increased with

excessive application of sewage sludge to soil, however applications of sewage sludge in low doses did not cause a significant increase in heavy metal concentrations. Hussein (2009) expressed that there was high correlation between the sewage sludge application dose and soil chemical contents (Fe, Mn, Cu, Zn, Cd, Pb, Ni). Similarly, Ferreira et al. (2011) observed an increase in soil Fe, Zn, Cu, and Cd contents with the increase of the sewage sludge dose. In another study, it was stated that the amount of soil Fe, Zn, and Cu increased significantly with the addition of sewage sludge and this could be attributed to the direct effect of the treatment sludge (Özyazıcı et al., 2012).
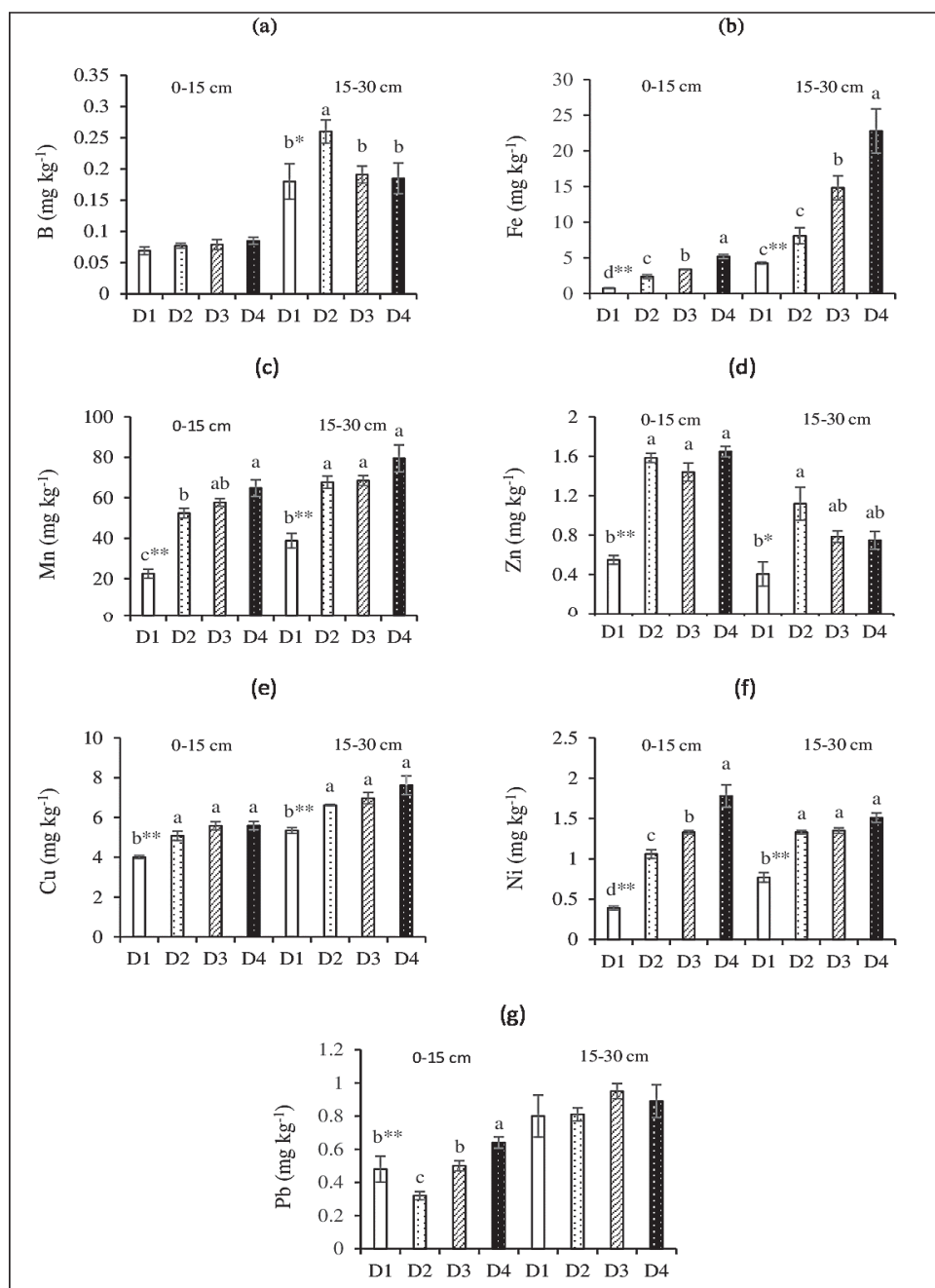

Fig. 11. Change in micro element and heavy metal contents by stabilized sewage sludge doses in two soil layers as the mean of leaching variants (**p<0.01; *p<0.05): (a) represents B (b) represents Fe (c) represents Mn (d) represents Zn (e) represents Cu (f) represents Ni (g) represents Pb

4. Conclusions

The fact that recycled wastewater has shown a similar effect with freshwater applications in the improvement of saline-sodic soil means that municipal wastewater with mild or medium salinity and low sodicity can be used readily in the leaching process to preserve freshwater resources. It could be said that recycled wastewaters with a similar quality can be used as leaching water instead of freshwater in arid and semi-arid regions with water shortages.

It is clear that even low doses of stabilized sewage sludge make a significant positive contribution in decreasing the sodicity problem. However, low doses by leaching effect did not contribute to the increase of N and P nutrients in soil. There was a limited increase in micro element contents, but heavy metal contents did not cause any pollution in the soil. Therefore, it is concluded that by choosing high doses (8 and 12 kg m^{-2}) of sewage sludge, reclamation success can be increased and advantages can be achieved in terms of soil productivity.

However, it should be researched whether adding sewage sludge to the entire problematic layer, instead of just the surface layer, could result in more positive findings to decrease salinity and sodicity and increase hydraulic conductivity.

Acknowledgement

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