Effect of using treated drainage water by modified clay on some plant and soil properties

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Abstract: Shortage of irrigation water sources in Egypt brings out the issue of managing all the available water resources and to re-use the non-conventional water resources such as agricultural drainage water. In this study, treatment of agricultural drainage water by adsorption onto kaolinite or kaolinite-humic complex was carried out by using fixed bed column model. Total organic carbon, heavy metals, EC, pH, major cations and anions were determined before and after treatment process. Agricultural drainage water purification occurred with flow rate of 70 ml/hour for each 10 g of adsorbent to treat one liter. The results showed that the kaolinite-humic complex was better in treatment of agricultural drainage water than kaolinite. Pots experiment was conducted to study the influence of irrigation with treated and non-treated agricultural drainage water on some Jew's Mallow plant properties and soil's chemical and physical properties. Plant and soil analysis showed great deterioration changes in plant and soil properties due to irrigation with treated agricultural drainage water. The results showed that drainage water treatment with modified clay had a significant reduction of total dissolved solids, heavy metals and organic contaminants which in turn enhanced chemical properties of treated agricultural drainage water as well as growth and health features of plants.

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1. Introduction

Water is one of the limiting factors for agricultural development in developing countries in order to meet the growing demand of the increasing population. Increasing shortages of fresh water has led to greater use of treated drainage water for irrigation purposes (Abedi-Koupai *et al.,* 2006). The treatment of Agricultural drainage water (ADW) presents a challenge due to the complex chemical characteristics of most drainage waters as it has possible public health and environmental side effects, as effluent may contain pathogens, high level of salts, detergents and toxic metals (Rani *et al.,* 2007). Previous researches (Al-Othman, 2008; AL-Jaboobi *et al.,* 2014; Nasr and Zahran, 2015), addressed some of the issues on the re-use potential of treated water for irrigation purposes and its effects on soil and plant properties. Treatment approaches can be divided into three general types: physical, chemical and biological. Many processes exhibit both physical and chemical aspects called physicochemical treatment (Tanji and Kielen, 2002).

In order to apply the drainage water for irrigation it should obtain the certain criteria of qualification after treatment, for parameters such as electrical conductivity (EC), total dissolved solids (TDS), and sodium adsorption ratio (SAR). Suspended materials and organic matter are also other parameters, which might be considered before application of drainage water to agricultural lands.

Adsorption has been found to be one of the most effective and economical method with high potential for removing, recovering and recycling of contaminants from water. Adsorption has the additional advantages of applicability at very low concentrations, suitability for using batch and continuous processes, ease of operation little sludge generation, possibility of regeneration and reuse, and low capital cost (Gupta *et al.,* 2009). Further, this process can remove/minimize different type of pollutants and thus it has a wider applicability in water pollution control (Bhatnagar and Minocha, 2006).

Kaolinite is among the most important sorbents for organic and inorganic contaminants in soil and sediments, which is due to their high abundance, low cost, large specific surface area, negative surface charge and reactive surface hydroxyl groups (Heggy *et al.,* 2013). Natural organic matter is a key component of both soils and sediments that clay mineral is likely to be associated with humic substances such as humic acid (HA).

Humic acid molecules, which adsorb onto the surface of soil minerals, would increase the adsorption capacity of metallic cations because HA has strong complexing sites. Moreover, the complexation capacity of HA is significantly higher than that of the usually reported for the adsorption capacities of clay minerals (Abat and Masini, 2005). Also, HA are anionic polyelectrolytes and reactive with heavy metal ions, radionuclides and many other environmental pollutants (Shaker *et al.*, 2012). Therefore, kaolinite and HA play a very important role in drainage water purification.

Accordingly the aim of this study is to investigate the influence of (i) modified kaolinite by humic acid on removing agriculture drainage water contaminates, (ii) applying treated and non-treated ADW in irrigation on some soil and plant properties.

2. Materials and Methods

2.1 Materials

Drainage water sampling

Agricultural drainage water was collected from Sohag, Egypt. Complete chemical analysis of drainage water: EC, pH, TOC, pathogens, major cations, major anions and heavy metals before and after treatment process were determined according to the methods adopted by American Society for Testing and Materials (ASTM) (2002).

Kaolinite

Kaolinite is commonly abundant mineral in soils, was purchased from the source clay minerals Repository, university of Missouri – Colombia, Missouri for using in this study. Kaolinite fraction was sieved to <0.002 mm diameter particles.

Humic acid

Humate salt was obtained from Sigma–Aldrich. Humate salt was used to prepare HA as follows (Swift, 1996): humate salt has been dissolved in deionized water, and precipitated by acidification to a pH of 2. The precipitate was redissolved in deionized water. This precipitation and dissolution were repeated 3 times. This pretreatment procedure could eliminate significantly the levels of other metals from HA, e.g. Al, Fe, Ca and Mg. Humic acid was characterized by FTIR spectra, elemental analysis and scanning electron microscope (SEM).

Soil samples

Soil material was taken from the surface layer (0 - 30 cm) of an experimental farm, at Shandwell Agricultural Research Station, Sohag governorate, Egypt. Physical and chemical analysis of soil such as mechanical analysis, pH value, calcium-carbonate content, organic matter, EC, soluble cations and anions were determined before and after irrigation with non-treated and treated ADW.

Seeds

Egyptian Jew's Mallow seeds (Balady) was used in all experiments. These seeds were obtained from the Ministry of Agriculture of Egypt.

2.2 Methods

Preparation of kaolinite and HA stock solutions

Kaolinite suspensions (25 g L^{-1}) stock solutions were prepared in order to obtain consistent solid concentrations in the equilibrium adsorption experiments. These suspensions were prepared in 0.01 mol. dm^{-3} of KCl for the HA adsorption at pH 6 over a period of 14 days. Moreover, the pH values checked up through the experimental life-time. Stock HA solutions were prepared by dissolving HA in an aqueous solution of NaOH (0.1 mol. dm^{-3}) under continuous stirring for 1 h. HCl or NaOH was then added to adjust its pH to 6. Amounts of KCl were added to HA solutions to adjust to the desired ionic strength (0.01 mol. dm^{-3}). Finally, humic acid concentration reached 416 mg L^{-1}in its stock solution (Komey *et al.*, 2014).

Preparation of modified kaolinite

A fraction of the well-mixed kaolinite suspension (at the desired pH and ionic strength values) was pipetted into HA solutions at concentration (416 mg/L to obtain on maximum adsorption capacity as described in pervious study (Shaker *et al.*, 2012). The suspension were shaken for 24 h on ahorizontal shaker (junior orbit shaker, Lab line Instruments, Melrose Park, IL, USA) to reach equilibrium. The final kaolinite concentration for all adsorption experiments was 12.5 g/L. Preliminary experiments verified that after 4 h no measurable change occurred in the adsorbed amount. Each sample was centrifuged for 5 min at 20000 rpm Damon/IEC Division Model HT High -Speed Centrifuge. Then, kaolinite -humic complex (ka-HA) was resuspended for 24 h in the horizontal shaker. This washing step was repeated three times to exclude the free HA from the solution. The stock ka - HA or kaolinite suspensions was stored overnight and part of them has been freeze-dried and stored. The ka - HA complex or kaolinite adsorbents were characterized using FT-IR spectroscopy, BET surface area and Energy Disperse Analysis X-ray (EDAX).

Treatment of ADW by adsorption on ka-HA and kaolinite

Treatment process was carried out with ka - HA and kaolinite. The treatment of ADW by adsorption process was carried out by using fixed bed column model (Karunarathnea and Amarasinghea, 2013). The column has a diameter of 3 cm and 30 cm length. The rate of adsorption process was calculated hourly.

Applied non-treated and treated agricultural drainage water

Pots experiment was carried out to investigate the influence of treated and non-treated ADW on plant and soil's chemical and physical properties. Experiments were carried out at 2015 and 2016. Pots were filled with 350 g of soil sample. Then, seeds were sown in pots. Non-treated and treated ADW have been added to the pots from stock water during planting. The pots were well arranged in a complete block with three replicates and placed on an open plastic mist bench. Plants were irrigated using the same volume of water at each time during the experiment. Throughout the harvest period; the vegetative growth, yield and concentrations of chlorophyll (a) and (b) were determined as outlined by Witham *et al.* (1971). The data collected as described elsewhere (Ghoneim and El-Araby, 2003) were subjected to analysis of variance (ANOVA) using statistical analysis system (SAS) and the means were separated using least significant difference (LSD 0.05).

3. Results and discussion

3.1. Properties of humic acid, kaolinite and ka - HA complex.

Elemental analysis is one of most simple and important means of characterizing HA (Heggy *et al.*, 2013). The elemental analysis result for the Aldrich HA was 40.06, 4.57, 0.79, 0.73 and 53.83 for carbon, hydrogen, nitrogen, sulphur and oxygen percentage respectively.

Energy Disperse Analysis X-ray (EDAX) results show chemical composition analysis of kaolinite (O 64.16%, Na 0.27%, Mg 0.09%, Al 16.94%, Si 17.61%, K 0.08%, Ca 0.03%, Ti 0.83%). Figure 1 displays the EDAX chart of kaolinite. The elemental analysis for kaolinite demonstrate that the Si:Al ratio is approximately 1:1 and the structure is comprised of an octahedral aluminum layer overlying a tetrahedral silicon sheet.

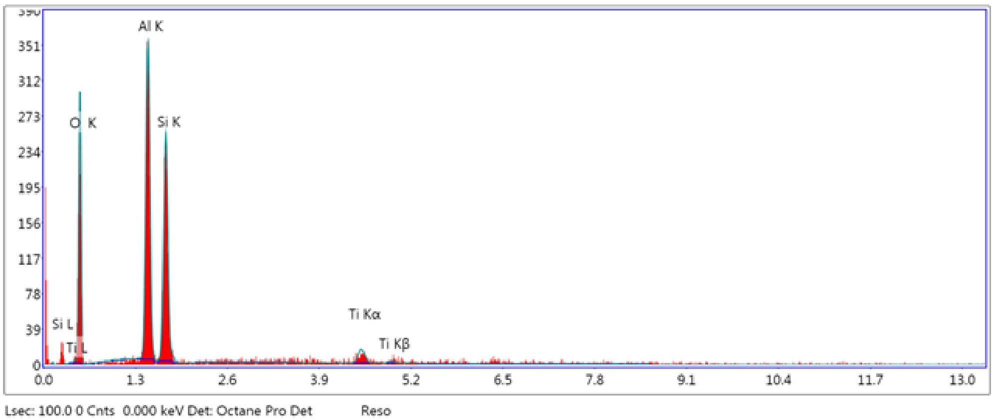

Fig. 1.Kaolinite elemental analysis by EDAX.

Figure 2 shows FT-IR spectra of humic acid, kaolinite and ka - HA. A large peak was observed at 1640−1588 cm^{-1}, associated to the carbonyl of the carboxylic acid group, and a broad peak at 3416.6 cm^{-1}, associated to carboxylates, phenols, and alcohols, for humic acid while the spectrum of kaolinite is a typical spectrum for well-crystallized kaolinite and matches the literature data very well (Chin *et al.*, 1994) as follows: a band around 3689 cm^{-1} and 3629 cm^{-1} due to OH stretching of inner hydroxyl groups and the strong bands at 1097 and 1026 cm^{-1}, indicates perpendicular and in-plane Si-O stretching. In addition, FT-IR spectroscopic analysis of kaolinite - humic acid complex shows major changes of the absorbance band at 1640-1588 and 1383 cm^{-1} as compared with the spectrum of the HA. These bands are characteristic of protonated, ionic and complexed carboxylic groups. These bands shift to lower frequency and broadening of the doublet that includes the latter feature, and changes in the region below 1020 cm^{-1}. These effects indicate that sorption on kaolinite is carried out through COO^- functional groups (Elsayed, 2013). This considers strong evidence for ligand-exchange mechanism.

The scanning electron microscopy was used to investigate the morphology changes with adsorption of HA. Figure 3 presents SEM micrograph of HA, kaolinite and ka - HA. The SEM of HA, kaolinite and ka - HA reveals numerous small discrete particles scattered among the micro aggregate. It was found that the ka - HA has rough surface. These indicate that HA aggregates on kaolinite particles.

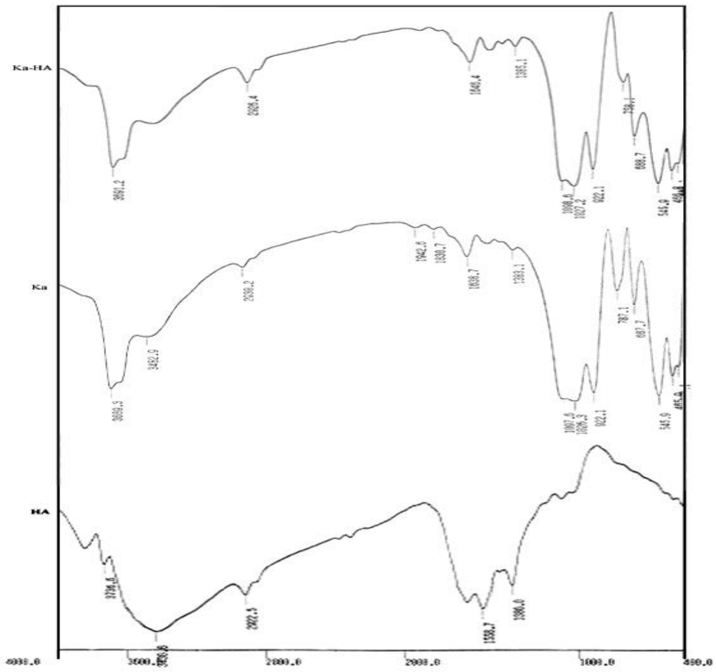

Fig. 2.FT-IR spectra of the humic acid, kaolinite and kaolinite - HA complex

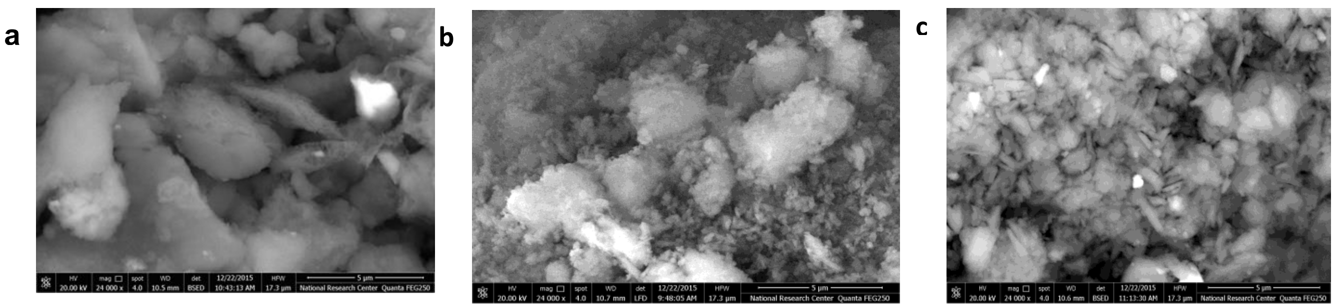

Fig. 3.SEM photograph of (a) HA, (b) kaolinite and (c) ka –HA complex

In order to investigate the influence of HA on the BET surface area for kaolinite and ka - HA was determined. BET surface area of kaolinite and kaolinite - humic acid complex were 10.7 and 12.2 m^2g^{-1} respectively. Therefore, HA enhanced the surface area of kaolinite.

The second way to evaluation the effect of humic acid on kaolinite surface, total active site for kaolinite and kaolinite - humic complex were determined by conductometric method (Komy *et al.*, 2014).Clearly, total active sites on kaolinite - humic complex were 4.32 meq g^{-1} while on kaolinite were 3.22 meq g^{-1}. This refers to a greater number of functional groups (–COOH, –C=O and –OH) on HA than that on kaolinite at the same conditions. Moreover, the results of total active sites on kaolinite - humic acid complexe have been matched with increasing BET surface area for kaolinite - humic acid complex than kaolinite.

3.2 Agriculture drainage water treatment

Treatment of ADW carried out by adsorption onto two sorbents "kaolinite and kaolinite - humic acid complex" by using fixed bed column model. The flow rate of this process was 70 ml/hour for each 10 grams kaolinite or kaolinite - humic acid to treat one liter. Data presented in Table 1, showed that TOC was completely removed from ADW by kaolinite and kaolinite - humic complex. In addition, cadmium, manganese, cobalt, chromium and lead were also found to be completely removed from ADW by kaolinite and kaolinite – humic complex while copper completely removed and removed by 50% ratio by kaolinite – humic complex and kaolinite respectively. Moreover, iron, zinc and selenium were removed by a ratio of 97%, 87.5% and 45% by kaolinite - humic complex while by ratio 15%, 50% and 10% by kaolinite from ADW respectively. From the results in Table 1, it is obvious that kaolinite - humic acid complex presents better activity compared to kaolinite in treating drainage water from heavy metals and organic contaminates. This refers to highly active site contents and BET surface area of kaolinite - humic complex rather than of kaolinite. Furthermore, HA generally contains both hydrophobic and hydrophilic moieties as well as many functional groups (−COOH, −C=O, and −OH) bonded to the aliphatic or aromatic carbons in the macromolecules. For that, HA can react with inorganic and organic contaminates. Thereafter, hydrophobic fraction of humic acid interacts with organic contaminates by hydrophobic – hydrophobic interaction mechanism while carboxylic and hydroxyl group interact with heavy metals by electrostatic and complexation mechanisms (Li *et al.*, 2003; Abate and Masini, 2005).

Moreover, kaolinite - humic complex enhanced the chemical analysis (EC, pH, major cations and major anions) of ADW rather than of kaolinite. The EC and pH values became much better to use in irrigation process. In addition, major cations and anions concentrations are highly affected with treatment by kaolinite - humic complex than kaolinite cf. table 2. This refers to total active sites contents of kaolinite - humic complex higher than kaolinite as described above and humic acid generally contains many functional groups (−COOH and −OH) can interact with cations by electrostatic interaction. Wheresoever, humic acid acts as cheating agent through its function groups.

The ability of water to expel calcium and magnesium by sodium can be estimated with the aid of sodium adsorption ratio "SAR". If water used for irrigation is high in Na^+ and low in Ca^{2+}, the cation exchange complex may become saturated with Na^+. This can destroy the soil structure owing to dispersion of the clay particles.

The main problem with high sodium concentration is its effect on soil permeability and water infiltration. Sodium also contributes directly to the total salinity of the water and may be toxic to sensitive crops. The sodium hazard of irrigation water is estimated by the sodium absorption ratio (SAR), which is calculated by the following formula (Richards 1954):

$$SAR = \frac{Na^+}{\sqrt{(Ca^{2+} + Mg^{2+})/2}}$$

Where Na^+, Mg^{2+} and Ca^{2+} are in meq/l.

Table 1: Heavy metals concentrations and organic matter percentage of non-treated and treated agricultural drainage water

Samples	Cd^{2+} ppm	Fe^{+2} ppm	Zn^{2+} ppm	Cu^{2+} ppm	Mn^{+2} ppm	Co^{+2} ppm	Cr^{+6} ppm	Se^{+4} ppm	Pb^{+2} ppm	TOC %
Non-treated ADW	0.0002	0.135	0.016	0.002	0.003	0.00003	0.0009	0.0304	0.011	0.05
ADW treated with kaolinite	0	0.115	0.008	0.001	0	0	0	0.0276	0	0
ADW treated with ka-HA complex	0	0.004	0.002	0	0	0	0	0.0165	0	0

Table 2: Major cations and anions concentrations of non-treated and treated agricultural drainage water

Sample no	pH	Ec ds/m	$[TDS_{640}]$ ppm	SAR	Na^+ ppm	K^+ Ppm	Ca^{2+} ppm	Mg^{2+} ppm	Cl^- ppm	SO_4^{2-} ppm	NO_3^- ppm	HCO_3^- ppm
Non-treated ADW	8.866	0.98	0.6272	1.023	50.87	31.65	50.23	80.79	101.3	60.3	48.65	124.29
ADW treated by kaolinite	8.294	0.761	0.487	0.83	32.88	18.03	30.37	52.88	72.71	49.71	36.41	98.32
ADW treated by ka-HA complex	7.81	0.645	0.413	0.74	22.80	17.36	22.17	30.2	44.5	35.8	18	79.54

Table 2 showed the SAR ratio values of non-treated and treated agricultural drainage water. The SAR value of non-treated ADW was 1.023 while 0.83 for ADW treated by kaolinite and decreased to 0.74 for agricultural drainage water treated by kaolinite - humic complex. Moreover the results indicating that the non-treated and treated agricultural drainage water from the study area are in range of excellent water class and can be used for irrigation on almost all soils, with little danger of the development of harmful levels of exchangeable sodium.

Pathogen analysis was determined for ADW before and after treatment. Pathogen analysis result showed that there was one type of fungi call aspergillassp, and free from bacteria. For that, drops of chloride were added to agriculture drainage water before treatment. Pathogen analysis has been repeated again after treatment and the results showed that treated drainage water became free from fungi.

3.3 Effect of applied treated and non-treated ADW in irrigation

In order to evaluate the influence of irrigation with treated and non-treated agriculture drainage water on some plant and soil properties under pots experiment. Jew's Mallow seeds were sown in pots and under different irrigation with non-treated and treated drainage water by kaolinite and kaolinite - humic complex in three replicated.

Soil properties

Data in table 3 showed the change in physical properties of soil before and after irrigation by treated and non-treated agriculture drainage water. The results illustrated that insignificant change in physical properties of soil under different irrigation source with little bite increasing in total organic carbon contents in case of using non-treated ADW than treated ADW. These results refer to EC values of non-treated and treated ADW are not high enough to change the physical properties of soil as described later in table 4. Furthermore, the little bite change in TOC refer to completely removing of organic contaminates during treatment process by kaolinite and kaolinite – humic complex from ADW. Moreover, Egyptian soil generally is poor in organic matter (OM) content, because the normal level of soil organic matter content is about 2-4%. It was 0.81% in the soil under investigation, this mean that this soil needs more organic fertilizer.

Data in table 4 showed the change in chemical properties of soil before and after irrigation by treated and non-treated agriculture drainage water. Soil pH and EC are important factors that affect nutrient availability and absorption by plant roots. Soil EC and pH had increased significantly by using non-treated ADW as compared to irrigated soil by treated ADW cf. Table 4. This is probably due to the alkaline nature and high concentrations of major cations.

Table 3: Physical Analysis for soil samples under investigation

Samples	SP mL/100g	OM %	Mechanical Analysis			Texture class
			Clay%	Silt%	Sand	
Blank before planting	48	0.81	20.6	57	22.4	Silt Loam
Non-treated ADW	45	0.81	20.6	57	22.4	Silt Loam
ADW treated with kaolinite	48	0.62	20.6	57	22.4	Silt Loam
ADW treated with ka-HA complex	45	0.63	20.6	57	22.4	Silt Loam

The same results were obtained by Alghobar *et al.*(2014) who reported that application of wastewater increased soil pH due to the alkalization effect of basic cations such as Na, Ca and Mg in the water. The highest values of EC, pH, major cations and major anions were after irrigation with non-treated ADW (Zein *et al.*, 2003) while the smallest values were after irrigation with treated ADW by kaolinite - humic complex. Moreover, data in table 4 explained that the chemical properties of soil such as EC, pH and major cations and anions decreased after irrigation by treated drainage water by kaolinite - humic complex than kaolinite. Hence, soil chemical properties became much better after irrigation with treated drainage water by kaolinite - humic complex than kaolinite. Accordingly, the irrigation with non - treated ADW seems to increase salinity, pH and accumulate inorganic and organic contaminates in soil while irrigation with treated ADW by kaolinite - humic complex gave a good soil chemical and physical properties.

Plant properties

Means of Vegetative Growth Characters of Jew's Mallow under treated and non-treated ADW are presented in Table (5). The results showed that plant height, Relative water content, Chlorophyll a, Chlorophyll b and Carotenoid parameters measured were significantly affected by the different water treatment, while No of leaves, Branches, Leaf width, Root length and Leaf area of plot were insignificantly affected. Furthermore, using treated ADW by kaolinite - humic complex in irrigation gave the highest values of all studied characters followed by using treatment ADW by kaolinite then non-treated ADW. The significantly depress in some plant growth characters under non-treated ADW irrigation due to the highest EC value where plant height, No of leaves, branches and root length decreased with increased EC values (Abdallah, 1998). Moreover, increasing EC values caused slower growth or led to stunted growth and plants mortality. In addition, toxicity resulting from excessive concentration of certain ions, principally Na^+ , Ca^{2+}, Mg^{2+}, Cl^- , $SO4^{2-}$, and $HCO3^-$as well as nutritional imbalances (Grattan and Grieve, 1999) may also play important roles in the response of plants in saline environments. Otherwise, the availability of some plant nutrients is greatly affected by soil and irrigation water pH. The "ideal" pH is close to neutral, and neutral soils are considered to fall within a range from a slightly acidic pH of 6.5 to slightly alkaline pH of 7.5. It has been determined that most plant nutrients are optimally available to plants within this 6.5 to 7.5 pH range, plus this range of pH is generally very compatible to plant root growth. The non-treated ADW pH was 8.86 while pH value was 8.2 and 7.81 of treated ADW by kaolinite and treated ADW by kaolinite - humic complex respectively, wherefore; the nutrients availability and plant growth were highly affected under irrigation with non-treated ADW than treated ADW by kaolinite and kaolinite - humic complex.

Table 4: Chemical Analysis for soil samples under study (in the peast extractable)

Soil Sample	pH 1:2.5	EC ds/m	Cations me/L				Anions me/L			
			Ca^{2+}	Mg^{2+}	Na^{+}	K^{+}	CO_3^{2-}	HCO_3^{-}	Cl^{-}	SO_4^{2-}
Blank before planting	7.90	1.02	8.69	0.78	0.82	0.25	-	4.25	5	2.30
Non-treated ADW	8	1.06	7.04	1.25	3.07	0.14	-	4.72	5	2.89
ADW treated with kaolinite	7.90	0.99	5.3	1.04	2.93	0.14	-	4.25	3.75	1.72
ADW treated with ka -HA complex	7.80	0.89	5.1	1.20	2.23	0.13	-	3.77	3.75	1.07

Accordingly, we believe that treatment agriculture drainage water by using kaolinite-humic acid complex is very economic and safe method to reuse agriculture drainage water again with highly significant effect on plant and soil properties.

Table 5: Means of Vegetative Growth Characters of Jew's Mallow

Treatment	Plant height (cm)	Relative water content %	Chlorophyll a (µg/ml)	Chlorophyll b (µg/ml)	Carotenoid (µg/ml)	No of leaves	Branches	Leaf width (cm)	Root length (cm)	Average leaf area of pot (cm)
Untreated ADW	11.00	66.25	6161.36	2427.37	1815.27	7.00	3.68	1.10	4.33	143.45
ADW treated with kaolinite	13.67	72.34	6757.09	2880.18	1911.13	7.67	3.33	1.17	4.17	165.14
ADW treated with ka – HA complex	14.50	73.95	8060.85	4228.86	1996.18	7.67	4.33	1.10	5.33	177.89
LSD $_{0.05}$	1.89	3.00	4.97	1.50	1.34	-	-	-	-	-

CONCLUSION

This study indicated that using agricultural drainage water in irrigation without future treatment caused problems for soil and plant on long term while treated ADW by kaolinite and kaolinite - humic complex had significant effect on soil and plant properties. Furthermore, the result explained that after using treated ADW by ka- HA complex in irrigation, soil properties became much better and gave the highest values of all studied characters of plant properties. Hence, treatment by kaolinite - humic acid complex consider better, safe and economic method can be used to re-use non-conventional water resources such as agriculture drainage water.

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