



## Egyptian Journal of Animal Health

P-ISSN: 2735-4938 On Line-ISSN: 2735-4946  
Journal homepage: <https://ejah.journals.ekb.eg/>

### Effect of *Moringa olifera* seeds powder and Alum treatment on heavy metals levels and pesticides pollution in Ismailia channel water

Seif S. Elsayed and Howayda A. Zohree

Department of Biochemistry, Toxicology and Feed Deficiency,  
Animal Health Research Institute (AHRI), Agricultural  
Research Center (ARC), Giza, Egypt

#### Article History

Received in 13/10/2021  
Received in revised from  
31/10/2021  
Accepted in 2/12/2021

#### Keywords:

Wastewater  
*Moringa olifera*  
Alum  
Heavy metals  
pesticides

#### ABSTRACT

The objective of this study is to make a comparison between the effect of *Moringa olifera* seeds powder (M.O) and alum treatments on the level of some heavy metals and pesticides detected in collected water samples from Ismailia channel. Ten water samples were collected nearby and at distance of 500 m from the drainage of fertilizers factory at Ismailia channel (5 samples from each site). They were analyzed for heavy metals by atomic absorption and screened for pesticides by LC/MS/MS.

*Moringa olifera* seeds powder treatment showed a marked reduction in the concentration levels of iron (Fe), manganese (Mn), zinc (Zn), and cobalt (Co) meanwhile lead (Pb) and cadmium (Cd) levels were undetectable at both distances. Alum treatment showed a significant decrease in concentration levels of Pb, Cd, Fe, Mn, and Zn but non-significant reduction in Co levels in water samples near the drainage. Also, at distance of 500m far from the drainage a marked reduction of Pb and Cd levels was observed while Fe and Mn were undetectable. However, Zn and Co showed non-significant variations. Pesticides screening showed presence of deltamethrin, nicotine, diazinon and phoxim, in raw water samples. Alum treatment resulted in partial removal of these pesticides with nearly complete clearance by *M.olifera*.

In conclusion, M.O showed powerful effect more than alum on treatment of waste water by removal of the most common pollutants (heavy metals and pesticides) from water.

#### INTRODUCTION

The quality and accessibility of drinking water are of great importance to human health. Drinking water may contain disease causing agents and toxic chemicals. To control the risk to public health, systematic water quality monitoring and surveillance are required. Thou-

sands of chemicals have been identified in drinking water supplies around the world with potential hazard to human health at relative high concentrations. Heavy metals are the most harmful of the chemical pollutants with particular concern due to their toxicities to human (Ravikumar and Sheeja 2013).

Corresponding Author: Seif S. Elsayed, Department of chemistry, toxicology and feed deficiency, Animal health Research Institute, Agricultural Research Center Dokki, Giza, Egypt

E-mail address: [seif\\_salem2007@yahoo.com](mailto:seif_salem2007@yahoo.com)

They are either derived from direct sources such as sludge dumping, industrial effluents and mine tailing or indirectly through highway runoff. Therefore, researchers and industrialists aim to withdraw heavy metals in effluents and soil. Industrial effluents can seep into aquifer and pollute the underground water, where it is discharged without proper treatment. The pollutants cannot be confined within specific boundaries, so they can affect aquatic life especially metallic effluent which have ecological impacts on water bodies. (Sani et al. 2001).

Traditional methods for cleanup of pollutants usually involve the removal of unwanted materials through sedimentation, filtration, and subsequent treatment such as filtration, neutralization and electro dialysis before disposal. These processes, however may not guarantee adequate treatment of the effluents (Hardman et al. 1993). Moreover they are often laborious and expensive considering the volume of waste released during the industrial production processes.

Coagulation is one of the most important physicochemical operations used in water and wastewater treatment (Pernitsky and Edzwald 2006).

Chemical coagulants are either metallic salts such as alum or polymers. Polymers are manmade organic compounds made up of long chain of smaller molecules. Chemical coagulation have been in practice for several decades to precipitate the soluble heavy metals present in the wastewater as hydroxides and facilitate their removal by physical separation through the sedimentation process (Yilmaz et al. 2007).

Naturally occurring coagulants are usually presumed safe for human health.

Moringaolifera seed acts as natural coagulant, adsorbent and antimicrobial agent. Its seed is believed to be an organic natural polymer. The coagulation mechanism of the moringaolifera coagulant protein has been described as adsorption, charge neutralization and inter particle bridging. Its main characteristic is its high molecular weight polyelectrolyte. (Ravikumar and Sheeja . 2013)

Earlier studies have been found moringaolifera seeds are nontoxic coagulants used in water treatment in developing countries (Eman et al. 2010).

Moringaolifera is the best discovered natural coagulant that can replace aluminum sulfate (alum) which is widely used for water treatment around the world. The Moringaolifera is one of the natural coagulants that have been tested over the years as an alternative to inorganic and synthetic coagulants. Disadvantages of inorganic and synthetic coagulants are causing of Alzheimer disease and similar health related problems, reduction of pH, high costs, production of large sludge volume and low efficiency in coagulation in cold water (Gandiwa et al. 2020). Moringaolifera has potential effect on water treatment as a coagulant, water softener and bactericidal agent. Additional advantages are its low cost, less biodegradable volume and with pH change of water. Moringaolifera is a sustainable low cost, locally available, simple, reliable, acceptable, eco-friendly and household level point of use water treatment coagulant technology most suitable for developing countries where major population use contaminated water for drinking purposes. (Ravikumar and Sheeja 2013). Therefore, the present study was conducted to detect the efficiency of Moringaolifera seeds powder in purification of water from some heavy metals at an industrial area in comparison to alum treatment.

## MATERIALS AND METHODS

### MATERIALS:

Aluminum sulfate (Alum) obtained from Sigma-Aldrich Company 99.99%

-Moringaolifera seed obtained from the Crops Research Institute in Agricultural Research Center, Egypt.

-Nitric acid obtained from Nile Company, Giza, Egypt.

### METHODS:

#### 1-Sampling:

The technique of sampling was conducted according to APHA (1998). Clean glass bottles of one liter capacity were used for each sam-

ple. All cleaned glassware were soaked in 10% HNO<sub>3</sub> overnight for metal analysis and washed with distilled and deionized water before they were used. The bottle washed firstly with some water of River Nile then plunging it in an inverted position below the water surface. The bottle was turned until the neck points slightly upward and pushed forward horizontally in a manner away from the hand. Each sample was labelled and identified showing source, site and date of sampling.

The water samples were collected from the Ismailia channel, near to the drainage of fertilizers factory and at a distance about 500 m from the drainage. Ten samples were collected (5 from each site) and were transported on ice in polyethylene containers to the lab and stored at 4°C. Samples collected for heavy metals were preserved by adding concentrated nitric acid to pH<2 to avoid microbial growth.

## **2-Treatment of waste water by alum method:**

This method according to (Siriprapha et al 2011) where aluminum sulphate or alum was added at a dose 400 mg/l of water sample .pH was adjusted to 10 by adding sod. Hydroxide then start rapid mixing at 200 rpm for 10 min followed by slow mixing at 45 rpm for 30 min and left for settling for 60 min and filtrate. The filtrate was analyzed for heavy metals using atomic absorption.

## **3-Treatment of waste water by M.olifera seeds according to the method described by Ravikumar and Sheeja (2013):**

### **Preparation of M. olifera seeds:**

Healthy mature seeds were collected from the Crop Research Institute and transported in plastic bags at room temperature. The seeds were removed from their pods.

The seeds were dried, peeled by hand and grounded in a mortar until get a fine powder. Finally, the resulting powder was sieved through a dry test sieve and stored in plastic bags at room temperature

Aqueous Extract of M.olifera seeds was prepared by using 200ml of distilled water and 25 g of MO seed powder, mixed by a magnetic stirrer for 60 minutes and settled for 20

minutes. The solution was filtered through a muslin cloth to remove insoluble materials and into the water to be treated.

The treated water was stirred by a magnetic stirrer rapidly for at 1 minute then slowly (15-20 rpm) for 5-10 minutes.

The treated water was sit without disturbing for at least 1-2 hours.

When the particles and contaminates have settled to the bottom, the clean water was carefully poured off and filtered by Whatman filter paper. Then analyzed by atomic absorption.

## **4-Determination of heavy metals in water samples:**

Heavy metals lead (Pb), cadmium (Cd), iron (Fe), Manganese (Mn), zinc (Zn), cobalt (Co) and were determined using SensAAAtomic absorption device. The samples were filtered through membrane filter of pore size 45 nm before analysis (APHA 1998). The calibration standards of metals were prepared according to the standard methods for examination water and wastewater analysis (APHA 1998). The conditions for the Atomic Absorption Spectrophotometer (AAS) were optimized according to the recommended setting for each of the metals of interest with background correction using high purity grade acetylene fuel

## **5-Detection of pesticides in surface waste water samples :(APHA 1998)**

Water samples were filtered in laboratory using glass fiber filters.

1 L of the filtered water sample was pumped at a flow rate of 20 mL/min, through a Carbopak-B SPE cartridge containing 0.5 g of graphitized carbon sorbent. The compounds were eluted with 1.5 mL methanol.

The sample volume is reduced to near dryness and the final volume of the extract is 1 ml.

LC/MS/MS. (4000 Q TRAP AR25791008) is equipped with LC Column: ZORBAX Extend-C-18, RRHT, 2.1 mm × 100 mm, 1.8 µm- Column temperature: 40 °C and the mobile phases: A: 0.1% formic acid in water, add NH<sub>4</sub>OH buffer to pH 5.5, B: Acetonitrile (ACN) Flow rate: 0.3 mL/m.

## **6-Statistical analysis:**

Data are represented as the mean ± SE. Data were analyzed by one-way ANOVA using the

statistical software package SPSS for Windows (Version 21.0; SPSS Inc., Chicago, IL, USA), followed by Duncan's post hoc test for multiple group comparison. Statistical significance was accepted at  $P < 0.05$ .

The nonparametric tests were applied to confirm the results obtained by one-way ANOVA by using the independent sample Kruskal-Wallis test (the significance level is 0.05).

### RESULTS:

This study examined the levels of heavy metals in water samples collected from Ismailia channel near the drainage and at 500m from

the drainage of a fertilizer factory. The obtained results in water samples near the drainage detected a high levels of lead and cadmium above the permissible limits and iron level within this limit according to the Egyptian standard specifications. Treatment with alum and *M.olifera* seeds powder resulted in a significant decrease in lead, cadmium and iron as shown in table 1.

Table 1. Effect of *M.olifera* /alum treatment on some Heavy metals levels in water samples near the drainage of fertilizers factory at Ismailia Channel.

Element conc (ppm)	Lead	Cadmium	Iron
Treatment			
Raw water	0.13±0.01 <sup>a</sup>	0.15±0.02 <sup>a</sup>	0.43±0.07 <sup>a</sup>
Alum treated water	0.017±0.002 <sup>b</sup>	0.062±0.005 <sup>b</sup>	0.023±0.006 <sup>b</sup>
<i>M.olifera</i> treated water	0.00 <sup>b</sup>	0.00 <sup>b</sup>	0.045±0.004 <sup>b</sup>
Permissible limit (Egyptian Standard Specifications ) ppm	0.01	0.003	<1

a, b & c: There is a significant difference ( $P < 0.05$ ) between any two means, within the same column.

Also the results showed high levels of zinc and cobalt above the permissible limit of the Egyptian specifications and manganese level within this limit . Treatment with alum resulted in a significant decrease of manganese and zinc and non-significant decrease of cobalt as

shown in table 2. Treatment with *M.olifera* seeds powder resulted in a significant decrease in these elements.

Table 2: Effect of M.olifera /alum treatment on some Heavy metals levels in water samples near the drainage of fertilizers factory at Ismailia Channel:

Elementconc ppm	Manganese	Zinc	Cobalt
Treatment			
Raw water	0.15±0.02 <sup>a</sup>	0.35±0.015 <sup>a</sup>	0.24±0.04 <sup>a</sup>
Alum treated water	0.03±0.004 <sup>b</sup>	0.27±0.012 <sup>b</sup>	0.18±0.06 <sup>a</sup>
M.olifera treated water	0.078±0.015 <sup>b</sup>	0.12±0.006 <sup>c</sup>	0.015±0.005 <sup>b</sup>
Permissible limit (Egyptian Standard Specifications) ppm	0.2	<1	0.005

a, b & c: There is a significant difference ( $P>0.05$ ) between any two means, within the same column.

The obtained results in water samples 500 m from the drainage showed high levels of lead, cadmium, and iron above permissible limits according the Egyptian specifications . Treatment with alum resulted in a significant de-

crease in lead and cadmium levels and disappearance of iron. While treatment with M.olifera seeds powder resulted in disappearance of lead and cadmium and a significant decrease of iron as shown in table 3

Table 3: Effect of M.Olifera /alum treatment on some Heavy metals levels in water samples at distance 500 m from the drainage of fertilizers factory at Ismailia channel

Elementconc ppm	Lead	Cadmium	Iron
Treatment			
Raw water	0.08±0.004 <sup>a</sup>	0.062±0.006 <sup>a</sup>	0.28±0.02 <sup>a</sup>
Alum treated water	0.012±0.005 <sup>b</sup>	0.047±0.003 <sup>b</sup>	0.00 <sup>b</sup>
M.olifera treated water	0.00 <sup>b</sup>	0.00 <sup>c</sup>	0.032±0.01 <sup>b</sup>
Permissible limit (Egyptian Standard Specifications) ppm	0.01	0.003	<1

a, b & c: There is significant difference ( $P>0.05$ ) between any two means, within the same column

Also the results showed that cobalt level was above the permissible limits of the Egyptian specifications while zinc and manganese levels were within this limit. Treatment with alum resulted in a non-significant decrease of

zinc and cobalt with disappearance of manganese while treatment with M.olifera seeds powder resulted in a significant decrease in these metals as shown in table 4.

Table 4: Effect of M.Olifera /alum treatment on some Heavy metals levels in water samples at distance 500 m from the drainage of fertilizers factory at Ismailia channel

Elementconc ppm	Manganese	Zinc	Cobalt
Treatment			
Raw water	0.14±0.004 <sup>a</sup>	0.35±0.06 <sup>a</sup>	0.09±0.015 <sup>a</sup>
Alum treated water	0.00 <sup>c</sup>	0.32±0.014 <sup>a</sup>	0.047±0.006 <sup>ab</sup>
M.olifera treated water	0.042±0.004 <sup>b</sup>	0.13±0.009 <sup>b</sup>	0.005±0.001 <sup>b</sup>
Permissible limit (Egyptian Standard Specifications) ppm	0.2	<1	0.005

a, b & c: There is a significant difference ( $P>0.05$ ) between any two means, within the same column

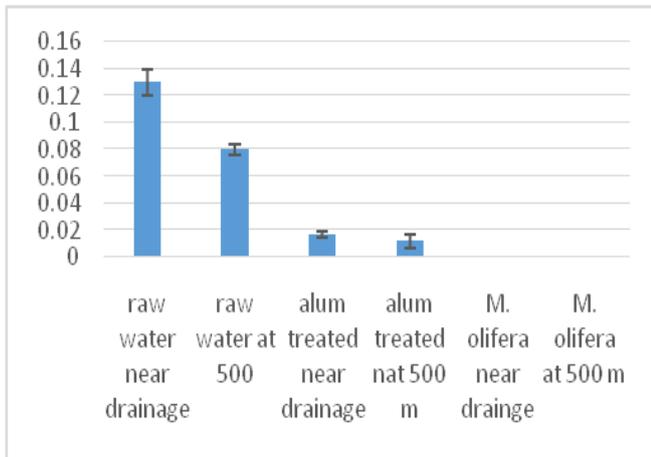


Fig (1): Lead levels in raw water and water treated with alum and M.olifera

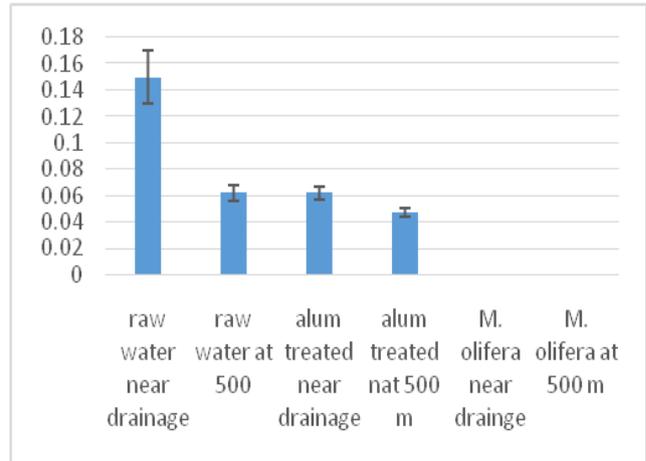


Fig (2): Cadmium levels in raw water and water treated with alum and M.olifera

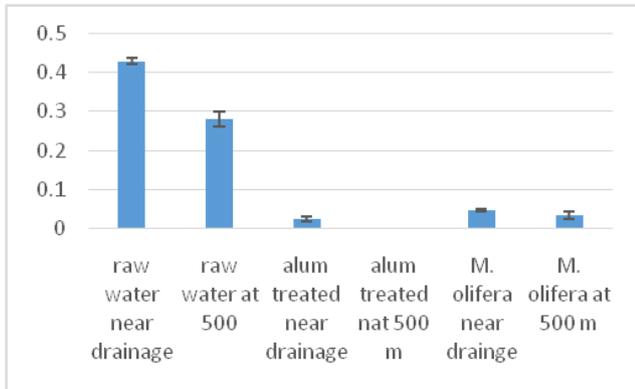


Fig (3): Iron levels in raw water and water treated with alum and M.olifera

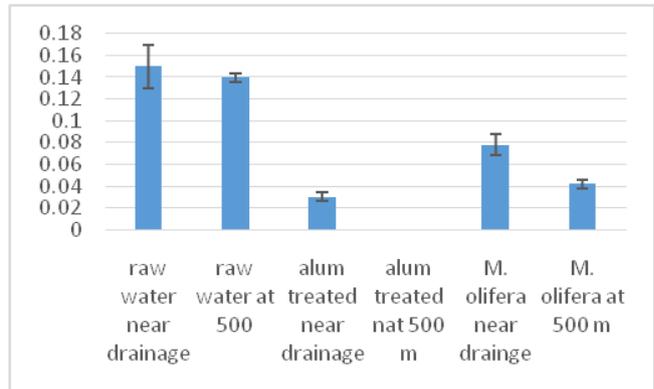


Fig (4):Manganese levels in raw water and water treated with alum and M.olifera

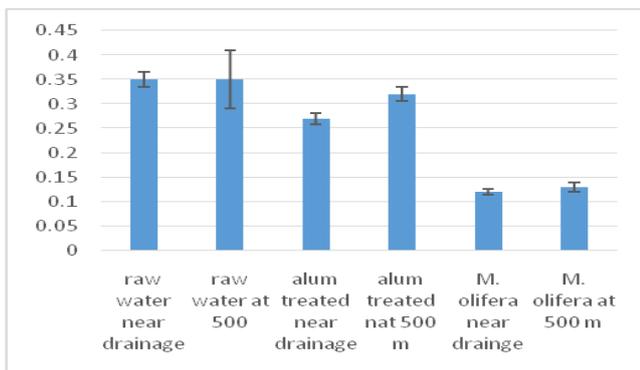


Fig (5): Zinc levels in raw water and water treated with alum and M.olifera

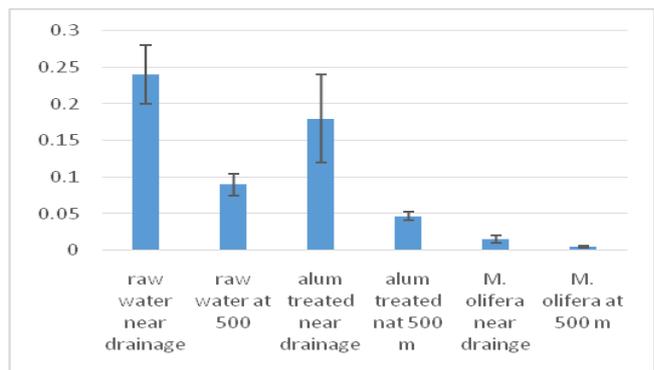


Fig (6): Cobalt levels in raw water and water treated with alum and M.olifera

Table (5): Detection of pesticides in water samples collected from Ismailia channel and effect of treatment by alum and *M.olifera* near the drainage:

	Type of pesticide	Raw surface water	Treatment by alum	Treatment by <i>M. olifera</i>
Sample 1	Deltamethrin	-ve	-ve	-ve
	Nicotine	-ve	-ve	-ve
	Diazinon	-ve	-ve	+ve
	Phoxim	+ve	-ve	-ve
Sample 2	Deltamethrin	+ve	+ve	-ve
	Nicotine	+ve	-ve	-ve
	Diazinon	+ve	-ve	-ve
	Phoxim	-ve	-ve	-ve
Sample 3	Deltamethrin	-ve	-ve	-ve
	Nicotine	+ve	-ve	-ve
	Diazinon	+ve	+ve	+ve
	Phoxim	+ve	+ve	-ve
Sample 4	Deltamethrin	+ve	-ve	-ve
	Nicotine	-ve	-ve	-ve
	Diazinon	+ve	-ve	-ve
	Phoxim	-ve	-ve	-ve
Sample 5	Deltamethrin	+ve	-ve	-ve
	Nicotine	+ve	+ve	-ve
	Diazinon	-ve	-ve	-ve
	Phoxim	+ve	+ve	-ve

Table (6): Detection of pesticides in water samples collected from Ismailia channel and effect of treatment by alum and *M.olifera* 500m from the drainage:

	Type of pesticide	Raw surface water	Treatment by alum	Treatment by <i>M. olifera</i>
Sample 1	Deltamethrin	-ve	-ve	-ve
	Nicotine	-ve	-ve	-ve
	Diazinon	-ve	-ve	-ve
	Phoxim	+ve	-ve	-ve
Sample 2	Deltamethrin	+ve	+ve	-ve
	Nicotine	+ve	-ve	-ve
	Diazinon	+ve	-ve	+ve
	Phoxim	-ve	-ve	-ve
Sample 3	Deltamethrin	+ve	+ve	-ve
	Nicotine	+ve	+ve	-ve
	Diazinon	-ve	-ve	-ve
	Phoxim	-ve	-ve	-ve
Sample 4	Deltamethrin	+ve	-ve	-ve
	Nicotine	-ve	-ve	-ve
	Diazinon	+ve	+ve	-ve
	Phoxim	+ve	+ve	-ve
Sample 5	Deltamethrin	-ve	-ve	-ve
	Nicotine	-ve	-ve	-ve
	Diazinon	-ve	-ve	-ve
	Phoxim	+ve	-ve	-ve

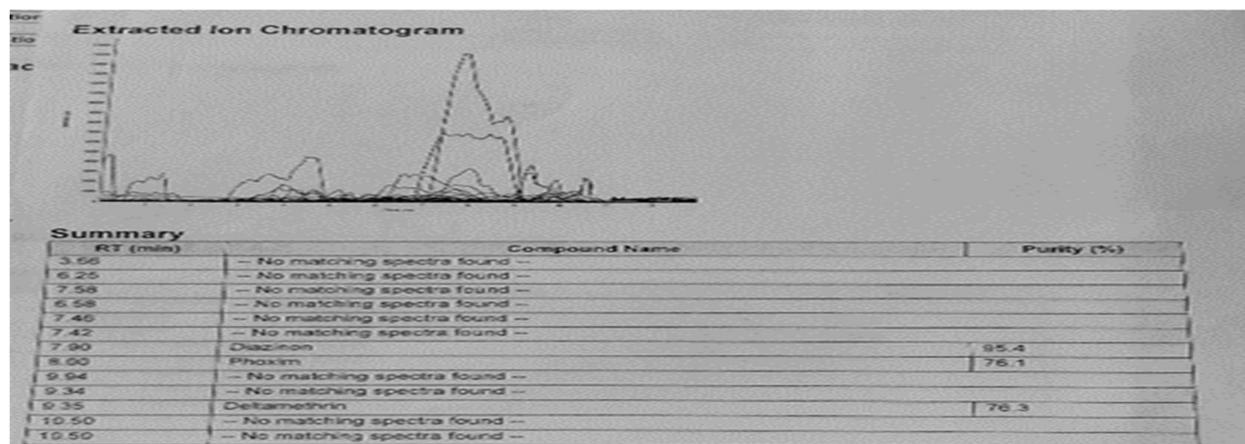


Fig (7): Pesticides screening by LC/MS/MS in raw water samples near the drainage

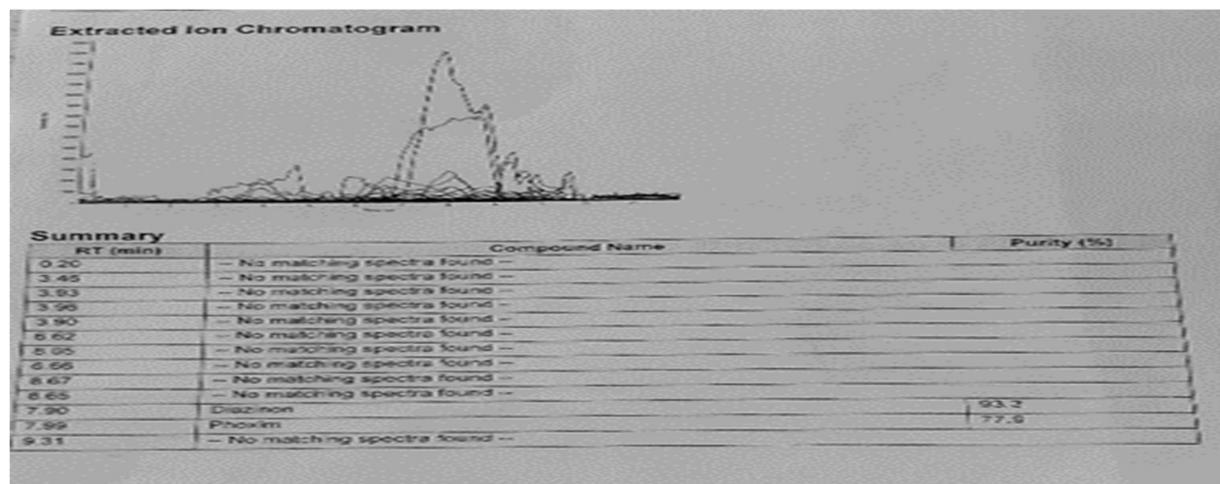


Fig (8): Pesticides screening by LC/MS/MS in raw water samples near the drainage

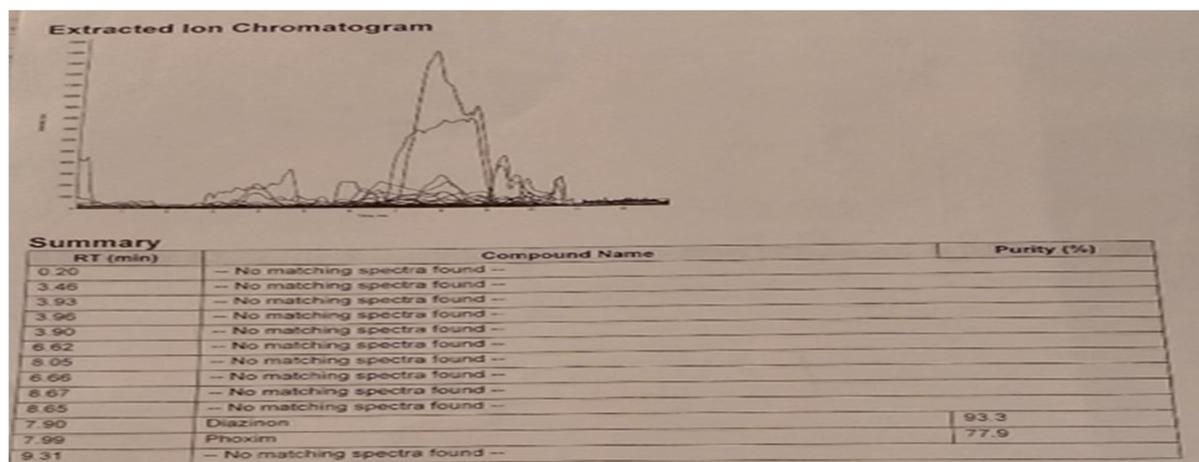


Fig (9):Pesticides screening by LC/MS/MS in water samples after treatment by Alum near the drainage

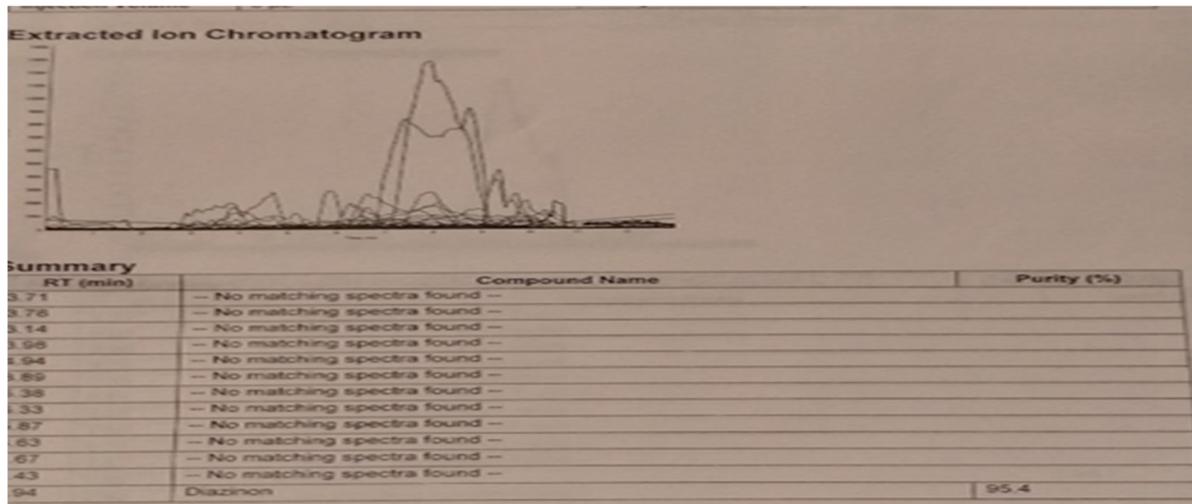


Fig (10): Pesticides screening by LC/MS/MS in water samples after treatment by *M.olifera* near the drainage

## DISCUSSION

Coagulation with moringa seeds according to **Eilert (1978)**, the seeds of *Moringa olifera* contains significant quantities of low molecular weight (water soluble proteins), which carries positive charge when the crushed seeds are added to raw water. The proteins produce positive charges acting like magnets and attracting the predominantly negatively charged particles (such as clay, slit bacteria, and other toxic particles in water) the flocculation process occurs when the proteins bind the negative charges forming flocs through the aggregation of particles which are present in water. These flocs are then easily removed by settling or filtration, a situation that give rise to this study on Moringa seed application. (**Muyibi et al. 2002a**)

The mechanism of alum in treatment of water include double layer compression, adsorption charge neutralization, sweep coagulation, and inter-particle bridging. The type of interaction between the chemical coagulant and contaminants determine the mechanism of coagulation. The predominance mechanisms observed during conventional coagulation with metal coagulants are adsorption, charge neutralization and sweep coagulation. (**Dennett et al. 1996**)

Regarding the level of lead and cadmium, our results showed that the lead level before treatments were above the permissible limits which is 0.01ppm according to Egyptian law

(**49/1982**) either in samples near the drainage or after 500 meters from the drainage. The elevated lead level in surface water correlated well with the industrial waste and leaded gasoline consumption (**Abd Elkader et al. 1993**). Treatment of water samples by aluminum sulfate (alum) resulted in a significant decrease in lead levels in samples near the drainage and in samples after 500 meters. These results agreed with **Mahmood et al. (2011)** while treatment with *M.olifera* showed disappearance of lead either in samples near the drainage of after 500 meters from the drainage and these results agreed with **Obsung et al. (2008)** who recorded the uptake of lead, copper, cadmium, nickel, and manganese from aqueous solution using the *M. olifera* seed biomass (MOSB) and amine-based ligand (ABL). Also agreed with **Reddy et al. (2010)** who reported the biosorption of  $Pb^{+2}$  from aqueous solutions by *M. olifera* bark. The elevated cadmium levels in surface water may be as a waste of electric batteries, and electronic components (**Friberg et al. 1986, Ros and Sloff 1987**) and they were above the permissible limit issued by the Egyptian Law (0.003 ppm). Treatment by alum resulted in a significant decrease in samples either near the drainage and at 500 m but still above the permissible limits (0.003 ppm) but the treatment by *M.olifera* resulted in the disappearance of cadmium form all samples at both sites and these results agreed with **Jamal et al. (2006)** who reported the sorption proper-

ties of the plant *M.olifera* for decontamination of Cd at laboratory scale which reached above 85.1% from the water samples.

Regarding the levels of iron, manganese, zinc and cobalt, our results showed the levels of manganese in the water samples at both sites were within the permissible limits issued by the Egyptian law (49/1982) which is 0.2 ppm and the cobalt level was above the permissible limit (0.005 ppm). The levels of iron and zinc were within the permissible limits <1 ppm. Alum treatment resulted in significant decrease of iron and manganese in samples near the drainage and don't be detected in samples after 500.

Zinc and cobalt levels showed a non-significant decrease than the raw samples, but cobalt was above the permissible limit. Heavy metals are usually present in water in diluted quantity and at acidic PH values. Metals enter system treatment are in stable, dissolved aqueous form, and are unable to form solids. The goal of metal treatment by precipitation is adjust the pH of water so that the metal will form insoluble precipitate, so they easily be removed from water (Citulski et al. 2009). Precipitation of metals is achieved by the addition of coagulants such as alum, lime, iron salts, and other organic polymer. The large amounts of sludge containing toxic compounds which produced during this process is the main disadvantage (Ahalya et al. 2003). *M.olifera* treatment resulted in a significant decrease of iron, zinc, manganese and cobalt below the raw water levels, either in samples near the drainage point or at 500 m. These results agreed partially with Pramanik et al. (2016) who reported that an initial dose of 5mg/l of alum or *M. oliferaseeds*, the removal efficiency for arsenic and iron was 63% and 58% respectively using alum, and 47% and 41% respectively using *M.olifera*.

#### **Determination of pesticides in surface water in Ismailia canal:**

The results showed that nicotine, deltamethrin, diazinon and phoxim pesticides were present in surface water samples table (5, 6). The results showed that deltamethrin present in samples 2, 4, and 5 near the drainage and 2, 3, and 4 at 500 m from the drainage and absent

in other samples. After treatment by alum it disappeared in samples 4 and 5 and sample 4 at 500m while treatment by *Moringa olifera* it disappeared in all samples in comparison with surface water before treatment. Nicotine result showed that it is present in samples 2, 3, 5 near the drainage and samples 2, 3 at 500 m. After treatment by alum it disappeared in samples 2 and 3 near the drainage and sample 2 at 500 m. Treatment with *moringaolifera* it disappeared in all samples in comparison with surface water. Before treatment, diazinon results showed that it is present in samples 2, 3, and 4 near the drainage and samples 2 and 4 at 500 m. After treatment by alum it disappeared in samples 2 and 4 near the drainage and in sample 2 at 500 m while treatment by *M.olifera*, it disappear in all samples except in samples 1, and 3 near the drainage and in sample 3 at 500m. Phoxim result showed that it is present at sites 1, 3, and 5 near the drainage and in samples 1,4 and, 5 at 500 m. After treatment by alum it is present only in samples 3 and 5 near the drainage and disappeared in samples 1 and 5 at 500 m. Treatment by *M.olifera* it disappeared from all sites when compared with result of surface water before treatment. These results agreed with result of Ivan and Vivitri (2019) who reported that *M. olifera* possess strong coagulation effectiveness in pesticides removal. Different pesticides washing solutions were used (mineral water, 2% salt solution, 10% salt solution, vegetable bath wash, and 10 g MO seed) for 10 min, followed by a 10 second mineral water bath rinse. Remaining profenofos residue concentrations on potatoes were quantified using High Performance Liquid Chromatography (HPLC). The experiments showed that the *M. olifera* seeds was the most effective at removing profenofos at 52.9% followed by the vegetable bath at 47.3%.

#### **CONCLUSION**

The present study demonstrates the effectiveness of the application of *M.olifera* seeds extract as primary coagulant in removal of heavy metals and pesticides from surface water more than the chemical coagulant as alum. Research should therefore continue in this area in order to develop new technologies for the treatment industrial or natural waters in developing countries.

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