Research Article

Study of Water Pollutants in El-Mahmoudia Agricultural Irrigation Stream at El-Beheira Governorate, Egypt

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Abstract

Background: Diverse types of agricultural, domestic, industrial, and municipal pollutants are discarded in agricultural water streams. These contaminants affect water quality extensively, therefore, current study aimed to scrutinize types of water pollutants in El-Mahmoudia water canal during winter, spring, summer, and autumn seasons.

Materials and Methods: Organic pollutants and pesticide residues were analyzed using the gas-chromatography-mass spectrometry (GC-MS). Heavy metals were determined by the atomic absorption spectrometry (AAS). Also, the physicochemical parameters of water samples were examined.

Results: Results showed that residues of DDD, DDE, and dieldrin insecticides were detected in all seasons, while chlorpyrifos-methyl residues were detected during the summer and autumn seasons only. Moreover, non-targeted metabolomic analysis revealed the presence of phthalates, amino and fatty acids, and plant secondary metabolites residues. In addition, significant deterioration of the physicochemical parameters and heavy metal contents were reported compared to the safety guidelines of the Egyptian Irrigation Ministry, WHO, and FAO organizations.

Conclusion: Current results found that agricultural and domestic pollutants were dominant in the water canal, which urge the need for more water treatment facilities to be established.

Keywords: Water Quality; Solid Phase Extraction (SPE); GC-MS; Persistent Organic Pollutants; Pesticide Residue Analysis

1. Introduction

Water pollutants affect directly and/or indirectly consumers' health and should not exceed international allowable limits [1]. In Egypt, the agricultural sector consumes about 67000 million m^3 (about 86%) of water resources [2]. Irrigation water is provided mainly from the River Nile with fewer amounts from groundwater and rain [2]. Along with water shortages in Egypt, its quality is degraded with waste from agricultural, industrial, and municipal effluents [3]. Industrial and municipal wastewaters are considered important pollutants, especially for drinking and irrigation water [4]. Contamination of water canals by industry- and public-originated wastes is due to irresponsible or negligent practices. Such activities intensify pollution problems of water, such as impaired biological oxygen demand (BOD), suspended matter, bacteria, metals, and chemicals [5]. Also, agrochemicals including pesticides and fertilizers exert short- and long-term effects on water quality [6-8]. Persistent (POPs) and volatile (VOPs) organic pollutants and pesticides might be leached from agricultural or urban areas causing contamination of the aquatic systems [6-13].

Pesticides, including the organochlorines might accumulate in food chains; for example, DDT was found in shrimp and fish at 1,000 to 10,000-fold of its concentration in water [14-22]. Monitoring pesticides and other pollutants in agricultural irrigation streams would enlighten officials about possible intervention methods to lessen potential adverse effects and type of environmental pollutants. With a limited number of environmental screening studies about sources and types of water pollutants in Egypt, the objective of the current study was to evaluate the water quality of El-Mahmoudia stream at El-Beheira Governorate, Egypt as a main irrigation canal in Northern Egypt via the estimation of physicochemical characteristics, persistent and/or volatile organic pollutants, plasticizers, plant residues, and pesticide residues.

2. Materials and Methods

2.1 Materials

Nitric acid, MacConky broth w/Natural Red medium, sodium sulfate (anhydrous), acetonitrile, ethyl acetate, hexane, and methylene chloride were HPLC-grade and were obtained from local reputed chemical distributors. Pesticides reference standards (Table 1) were purchased from ULTRA Scientific Analytical Solutions, RI, USA.

2.2 Location of study area

El-Mahmoudia canal (77 km long) starts from the Westside of Rosetta branch and ends at Alexandria harbor. It provides irrigation water for an area of 285,714 Feddans (120,000 Hectares) and provides water for municipality and industrial activities in both Alexandria and El-Beheira Governorates. Moreover, it provides water to 67 branch canals that provide water to more than 90 villages [3].

Acaricides, Insecticides, and Nematicides
α-HCH (25)*, β-HCH (25), γ-HCH (25), p,p-DDE (10), p,p-DDD (10), Aldrin (20), Azinophos-methyl (20), Bifenazate (50), Cadusafos (50), Chlorpyrifos (10), Chlorpyrifos-methyl, (10), Cyhalothrin (10), Cypermethrin (15), Deltamethrin (10), Dicofol (100), Dieldrin (10), Dimethoate (50), Diazinon (20), Dichlorvos (10), Endosulfan (15), Endosulfan Sulfate (15), Endrin (50), Endrin Aldehyde (50), Endrin Ketone (50), Esfenvalerate (15), Heptachlor (10), Heptachlor epoxide (10), Hexythoazox (25), Malathion (10), Pirimiphos-methyl (10), Profenofos (15), Quinalphos (15), Thiocyclam (25), and Thiamethoxam (50)
Fungicides
Chlorothalonil (25), Difenoconazole (20), Diniconazole (20), Fluazinam (20), Propioconazole (15), and Penconazole (30)
Herbicides
Atrazine (10), Butralin (20), and Pendimethalin (50)

*Numbers between brackets represent the limits of quantification (LOQ; µg L^{-1})

Table 1: List of scanned for pesticides in water samples that were collected in Autumn (A), Winter (W), Spring (SP), and Summer (SU) seasons using the GC-MS.

2.3 Collection of water samples

Water samples were collected over one year starting in December 2016 to August 2017 representing Winter, Spring, Summer, and Fall of 2017. Samples of 2.5 L each were collected from a mid-point of El-Mahmoudia canal in clean Pyrex borosilicate brown glass bottles at 50 cm below the water surface. The bottles were transported to the laboratory and filtered, then analyzed for water pollutants, including plant residues (secondary metabolites), pesticide residues, and domestic and industrial effluent pollutants within 3 hrs of collection. The physicochemical, microbiological characteristics and mineral contents were estimated on water samples collected during the Autumn season due to elevated contamination levels compared to other seasons.

2.4 Gas chromatography-mass spectrometry (GC-MS) analysis of pollutants and pesticides residues extraction

A modified solid phase extraction (SPE) technique using the Empore disc technology, according to EPA 353523 was used. The disks were washed with 5 ml of a 1:1 mixture of ethyl acetate and methylene chloride (ETAC:MeCL2). Water samples were extracted under vacuum for about 10 min, then disks were rinsed with two 3 ml portions of 1:1 ETAC:MeCL2. Combined eluates were passed over sodium sulfate (anhydrous). Gently, the extract was dried over a water bath under a stream of nitrogen to a volume of 0.5 to 1 mL. The concentrated extract was injected into the GC-MS. To check the recovery of the internal standards, aliquots of the standard were added to the blank (distilled) water and is passed through the same extraction process.

2.5 Separation and estimation conditions

Water extracts and recovered samples (2 µL) were analyzed using GC-MS equipped with Varian 8200 autosampler, which was operated in splitless mode. An HP-5MS capillary column (30 m × 0.53 mm i.d. 0.25 um film thickness) was used to separate the components. Helium was used as the carrier gas. Separation conditions were as follows: initial column temperature set at 80°C for 6 min. It was increased to 215°C at 15°C min^{-1} (hold for 1 min), then to 230°C at 5°C min^{-1} and finally to 290°C at 5°C min^{-1} (hold for 2 min). The carrier gas was at a constant flow rate of 1.1 mL min^{-1}. The GC-MS was controlled by a computer system, which has EI-MS libraries. The target compounds were identified by their full mass spectra scans and retention time using the total ion current as a monitor to give a total ion chromatogram (TIC). The use of the full scan mode allowed the contrast of the spectrum of obtained compounds with the EI-MS libraries. Pollutants and pesticide compounds identified on NIST 14, Willey 9, and PEST database libraries. Then individually listed compounds were re-confirmed through the online databases: AMDIS, PUBCHEM, ChemSpider, and Chinese Chemical database.

2.6 Method and machine precision

The precision of the analytical method was confirmed through the repeatability (intra-day assay) and intermediate precision (inter-day assay) [24]. The precision was expressed as coefficient of variation percentages ((mean/standard deviation) × 100). The limits of quantification (LOQs) were calculated from the signal-to-noise (S/N) ratios of the samples with the lowest concentration level of the same studied pesticides. Fortified water samples (0.1 and 1 µg L^{-1} of pesticide standards) were extracted and analyzed as previously mentioned. The average recovery percentages and the percent relative standard deviation (%RSD) for recoveries were calculated. All data of pesticide residue analysis were corrected according to these obtained recovery percentage values.

2.7 Heavy metals measurements

Determination of heavy metals: copper (Cu), ferrous (Fe), manganese (Mn), potassium (K), sodium (Na), calcium (Ca), chlorine (Cl), lead (Pb), and cadmium (Cd) were done following the method of APHA [25, 26] using the Atomic Absorption Spectrophotometer (Model Thermo Scientific ICE 3000 series).

2.8 Physicochemical analyses

Temperature, pH value, electric conductivity (EC), total dissolved solids (TDS), total solids (TS), turbidity, dissolved oxygen (DO), biological oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS), ammonium (NH_4), nitrate (NO_3^-), chloride, carbonate, and bicarbonate were measured according to the reference methodology reported by APHA [25]. Data were expressed as mg L^{-1} and compared to the listed permissible limits of APHA, FAO, and WHO [25, 27, 28].

2.9 Microbiological examination

The multiple tube fermentation technique was carried out to estimate the most portable number (MPN) of the total microbial count according to APHA [25] using MacConky broth w/natural red (HIMEDIA M007) medium. Aliquots of 1 mL of each of serial dilution of the water samples were added to each of 3 tubes containing 9 mL of McConkey broth. Cultural characteristics observed after incubation at 35-37°C for 18-24 hrs.

2.10 Statistical analysis

Data were analyzed using the Statistical Analysis System (SAS, Cary, USA, version 9.3) software. The physicochemical parameters, heavy metals, and pesticide residues were presented as mean ± SD. Significant means were compared using Tukey's Studentized Range (HSD) *post-hoc* Test ($P \leq 0.05$). Plant residues and plasticizers were reported in tables when the probability of a match with the reference database libraries exceeds 70% probability.

3. Results

3.1 Accuracy and precision of GC-MS method

The results of precision and repeatability (intra-day assay and inter-day assay) of the employed method were presented in Table 2. Also, the recovery percentages of standard pesticides ranged from 92 to 102 % (Table 2). Data showed that the developed method was precise as the coefficient of variation (CV%) values were less than 10% [24, 29]. These results demonstrate that the analytical method is reliable and effective.

Pesticide	**CV (%)***		**Recovery (%) ± RSD**	
	Intra-Assay	**Inter-Assay**	**0.1 µg L^{-1}**	**1 µg L^{-1}**
Heptachlor	5.18	7.62	91.7 ± 4.6	92.8 ± 6.2
Dieldrin	4.45	6.54	96.7 ± 2.0	99.1 ± 1.2
p,p-DDD	4.50	7.69	93.5 ± 2.1	95.6 ± 1.5
p,p-DDT	5.36	6.34	98.2 ± 3.5	102.3 ± 4.0
Methoxychlor	5.53	4.93	97.1 ± 2.9	99.3 ± 4.0
Chlorpyrifos-methyl	4.83	6.84	97.5 ± 3.1	99.7 ± 3.7

*Inter-assay and intra-assay precision data obtained from the analysis of the concentrations of each standard pesticide in fortified laboratory blank water samples.

Table 2: Coefficients of variation (CV%) and recovery (%) percentages ± RSD for the pesticides that were extracted from spiked blank water (distilled) samples.

3.2 GC-MS analysis of water pollutants

Data in Figure 1 and Tables 3, 4, 5, and 6 showed the combined results of the GC-MS analysis of pollutants in water samples during winter, spring, summer, and autumn seasons. Various organic chemicals, pesticides, oils, fatty acids, carboxylic acids, plant metabolites, and plasticizers were identified and confirmed using NIST 14, WILEY 9, and PEST mass spectral libraries.

3.3 Pesticide residues

Results in Table 3 and Figure 1 lists the detected pesticides' residues and limits of quantification. Out of the organochlorine insecticides (OC) screened for, only p,p-DDE, p,p-DDD, and dieldrin were detected at different concentrations at the four seasons but at lower levels than the ADI permissible limits. The first two compounds were adducts of DDT that was banned worldwide in the previous century. Also, out of the pesticides mix (insecticides, fungicides, nematicides, acaricides, and herbicides) that were studied, the organophosphate insecticide chlorpyrifos-methyl was detected only during Summer and Autumn seasons. Noticeably, levels of chlorpyrifos-methyl were greater during the Summer compared to autumn.

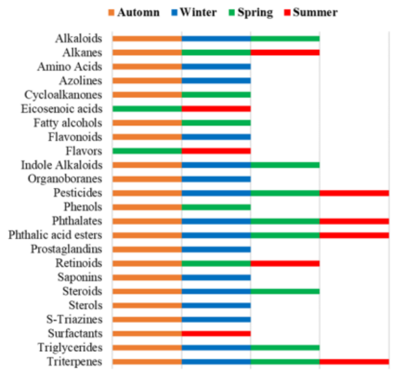

Figure 1: List of groups of plant metabolites, pesticides, plasticizers, saponins, surfactants, and triglycerides detected in water samples (n=4) collected on Autumn, Winter, Spring, and Summer seasons and verified in the Willey 9, NIST 14, and AMIDS 32 metabolite databases.

Pesticide	**Residue (μgL^{-1})**				**LOQ (ngL^{-1})**
	A	**W**	**SP**	**SU**	
p,p-DDE	0.062^{b}	0.065^{b}	0.085^{a}	0.048^{c}	10
p,p-DDD	0.045^{c}	0.075^{a}	0.065^{ab}	0.047^{c}	10
Dieldrin	0.080^{b}	0.114^{a}	0.109^{ab}	0.050^{c}	10
Chlorpyrifos-methyl	0.137^{b}	-	-	0.227^{a}	10

n=4, Means were compared using Tukey's Studentized Range (HSD) *post-hoc* Test ($P \leq 0.05$). Means with the same superscript letter are not significantly different.

Table 3: List of the detected pesticides in water samples that were collected from all-Mahmoudia canal in Autumn (A), Winter (W), Spring (SP), and Summer (SU) seasons using the GC-MS.

3.4 Triglycerides, plant residues, and phthalates (domestic waste)

Results in Figure 1 and Table 4 showed the presence of 2',6'-dihydroxyacetophenone, bis(trimethylsilyl) ether, 9,12-octadecadienoic acid (Z,Z)-, 2,3-bis[(trimethylsilyl) oxy]propyl ester, n-hexadecanoic acid, 2,3-bis[(trimethylsilyl) oxy]propyl (9E,12E,15E)-9,12,15-octadecatrienoate, 6,9,12,15-docosatetraenoic acid, methyl ester, cyclopropaneoctanoic acid, 2-octyl-, methyl ester, 16-octadecenoic acid, methyl ester, pentadecanoic acid, methyl ester, 9,12,15-octadecatrienoic acid, 2,3-bis[(trimethylsilyl)oxy]propyl ester, (Z,Z,Z), oleic acid, 3-(octadecyloxy)propyl ester, olean-12-ene-3,15,16,21,22,28-hexol, (3β,15α,16α,21β,22α), oleic acid, eicosyl ester as the major compounds detected mostly during Winter, Spring, and Autumn. Also, results in Figure 1 and Table 5 showed the presence of various residues of plants: hydrocarbon alkanes, flavonoids, prostaglandins, steroids, alkaloids, sterols, retinoids, amino acids, eicosenoic acids, triterpene, and terpenoids. Moreover, most of the organic pollutants detected using the GC-MS data analysis were identified as endocrine disrupting phthalate esters, fatty acids, phenolic acids, and plasticizers (Figure 1 and Table 6) including decamethylcyclopentasiloxane, 1-(2-acetoxyethyl)-3,6-diazahomoadamantan-9-one oxime, nonadecane, 2',6'-dihydroxyacetophenone, bis(trimethylsilyl) ether, 4H-1-benzopyran-4-one,2-(3,4-dimethoxyphenyl)-3,7-dimethoxy-,11,16-bis(acetyloxy)-3,20-dioxopregn-4-en-21-yl acetate, 9,12-octadecadienoic acid (Z,Z)-, 2,3-bis[(trimethylsilyl) oxy]propyl ester, m-dioxane, 5-(hexadecyloxy)-2-pentadecyl-trans-dodecamethylcyclo hexasiloxane, 2-(9-bora bicyclo[3.3.1]non-9-yloxy)-3-([2-(9-borabicyclo[3.3.1]phenylnon-9-yloxy)ethyl]sulfanyl) propyl ether.

Rt	Chemical	Season			
		A	W	SP	SU
6.10	2',6'-Dihydroxyacetophenone, bis(trimethylsilyl) ether	D[1]	D	-[2]	-
6.57	9,12-Octadecadienoic acid (Z,Z)-, 2,3-bis[(trimethylsilyl)oxy]propyl ester	D	D	-	-
14.24	n-Hexadecanoic acid	D	D	D	-
18.18	2,3-Bis[(trimethylsilyl)oxy]propyl (9E,12E,15E)-9,12,15-octadecatrienoate	D	D	-	-
19.83	6,9,12,15-Docosatetraenoic acid, methyl ester	D	-	D	-
22.08	Cyclopropaneoctanoic acid, 2-octyl-, methyl ester	D	-	D	-
26.61	16-Octadecenoic acid, methyl ester	D	-	D	-
27.06	Pentadecanoic acid, methyl ester	D	-	D	-
32.19	9,12,15-Octadecatrienoic acid, 2,3-bis[(trimethylsilyl)oxy]propyl ester, (Z,Z,Z)-	D	-	D	-
33.24	Oleic acid, 3-(octadecyloxy)propyl ester	D	-	D	-
35.05	Olean-12-ene-3,15,16,21,22,28-hexol, (3β,15α,16α,21β,22α)-	D	-	D	-
35.21	Oleic acid, eicosyl ester	D	-	D	-

[1]Detected and [2]Not detected

Table 4: List of triglycerides detected in water samples (n=4) collected from El-Mahmoudia canal on Summer (SU), Autumn (A), Winter (W), and Spring (SP) seasons and verified in the Willey 9, NIST 14, and AMIDS 32 metabolites databases.

3.5 Physicochemical characteristics of water samples

Results in Table 7 showed that the turbidity value was 84% greater than of the permissible limits of the guidelines of WHO with 9.17 NTU. Such elevated levels of turbidity correspond to increased suspended materials that reduce light penetration and restrict plant growth and hence food resources and habitat for water organisms. Also, suspended solids concentration in El-Mahmoudia stream was 51 mS cm^{-1}, which was within the permissible limits of Egyptian law 48/1982 (<100 mg L^{-1}). The pH value was found to be low compared to the reference values and that might be due to the drainage of organic matter [60-63]. The BOD was significantly increased (138% greater than the reference limits) with 23.75 mg L^{-1}. The mean value of COD was 24.75 mg L^{-1}, which was 65% greater than the standard levels (Egyptian Law 48/1982), which was <10-15 mg L^{-1}. All previously mentioned results significantly affected microbiological level (MPN) in El-Mahmodia stream to a level (12075 cm^{-3}) that exceeded the limits of Egyptian Law 48/1982 (5000 100 cm^{-3}).

Rt	Chemical	Chemical Class	Season			
			A	W	SP	SU
5.76	Nonadecane	Alkane	-[2]	-	D[1]	D
6.30	4H-1-Benzopyran-4-one,2-(3,4-dimethoxyphenyl)-3,7-dimethoxy-	Flavonoids	D	D	-	-
6.41	11,16-Bis(acetyloxy)-3,20-dioxopregn-4-en-21-yl acetate	Prostaglandin	D	D	-	-
8.00	1-Tridecene	Alkane	-	-	D	D
8.04	((5LPregnane- 3,20 L diol14 ,à,18à-[4-methyl-3-oxo-(1-oxa-4-azabutane-1,4-diyl)]-, diacetate	Steroids	D	D	-	-
8.21	2,7-Diphenyl-1,6-dioxopyridazino[4,5:2',3'] pyrrolo[4',5'-d]pyridazine	Alkaloids	D	D	-	-
10.05	Phenol, p-tert-butyl-	Flavor	-	-	D	D
10.91	3-Hydroxyspirost-8-en-11-one	Sterol	D	D	-	-
12.18	Dasycarpidan-1-methanol, acetate (ester)	Alkaloids	D	D	D	-
19.83	Fenretinide	Retinoids	D	-	D	D
20.45	Methyl((24-oxo-3,7,12 tris[(trimethylsilyl) oxy]cholan-24-yl)amino)acetate	Amino Acid	D	D	-	-
21.22	cis-13-Eicosenoic acid	Eicosenoic acids	-	-	D	D
21.44	Propanoic acid, 2-(3-acetoxy-4,4,14-trimethylandrost-8-en-17-yl)-	Triterpene	D	D	-	-
24.00	Estra-1,3,5(10)-trien-17β-ol	Steroids	D	-	D	-
25.59	Dihydroxanthin	Alkaloids	D	-	D	-
26.50	Corynan-17-ol, 18,19-didehydro-10-methoxy-, acetate (ester)	Indole Alkaloids	D	-	D	-
31.46	Tricyclo[20.8.0.0(7,16)]triacontane, 1(22),7(16)-diepoxy-	Alkane	D	-	D	-
32.52	1H-Cyclopropa[3,4]benz[1,2-e]azulene-5,7b,9,9a-tetrol, 1a,1b,4,4a,5,7a,8,9-octahydro-3-(hydroxymethyl)-1,1,6,	Terpenoids	D	-	D	-
34.59	9-Desoxo-9-x-acetoxy-3,8,12-tri-O-acetylingol	Terpenoids	-	-	D	D
36.81	Pregnane, 3,11,17,20,21-pentamethoxy-, (3α,5β,11β,17α,20β)-	Steroids	D	-	D	-

[1]Detected and [2]Not detected

Table 5: List of plant residues detected in water samples (n=4) of El-Mahmoudia canal on Summer (SU), Autumn (A), Winter (W), and Spring (SP) seasons and verified in the Willey9, NIST14, and AMIDS32 metabolites databases.

Rt	Chemical	Class	Season			
			A	W	SP	SU
5.37	Decamethylcyclopentasiloxane	Saponin	D[1]	D	-[2]	-
5.65	1-(2-Acetoxyethyl)-3,6-diazahomoadamantan-9-one oxime	Azolines	D	D	-	-
6.41	11,16-Bis(acetyloxy)-3,20-dioxopregn-4-en-21-yl acetate	Prostaglandin	D	D	-	-
7.03	m-Dioxane, 5-(hexadecyloxy)-2-pentadecyl-, trans-	S-Triazine	D	D	-	-
7.13	Dodecamethylcyclohexasiloxane	Saponin (Cyclic Siloxane)	D	D	-	-
7.28	2-(9-Borabicyclo[3.3.1]non-9-yloxy)-3-([2-(9-borabicyclo[3.3.1] phenyl non-9-yloxy)ethyl]sulfanyl)propyl ether	Organoborane	D	D	-	-
7.95	Sulfurous acid, butyl hexyl ester	Surfactants	D	-	-	D
9.15	Phthalic acid, butyl tetradecyl ester	Phthalic acid ester	D	D	D	-
10.05	Phenol, *p*-tert-butyl-	Flavor	-	-	D	D
11.1	Hexadecamethyl-cyclooctasioxane	Saponin	D	D	-	-
13.3	Phthalic acid, isobutyl octadecyl ester	Phthalate	D	D	D	-
13.37	Phthalic acid, butyl 2-ethylbutyl ester	Phthalate	D	D	-	-
14.32	1,2-Benzenedicarboxylic acid, dibutyl ester	Phthalate	D	D	-	D
15.95	Phenol, 3,5-bis(1,1-dimethylethyl)-	Phenol	D	-	D	-
17.89	1,2-Benzenedicarboxylic acid, butyl phenylmethyl ester	Phthalate	D	D	-	-
26.23	1-Hexadecanol, 2-methyl-	Saponin, Fatty alcohol	D	-	D	-
27.12	1,2,4-Trioxolane-2-octanoic acid, 5-octyl-, methyl ester	Saponin	D	-	D	-
33.88	Phthalic acid, di(2-propylpentyl) ester	Phthalate	D	-	D	-
33.92	Bis(2-ethylhexyl) phthalate	Phthalate	D	-	D	D
34.41	Benzeneacetonitrile, α-[[4-(dimethylamino)-2,5-dimethoxyphenyl]methylene]-4-nitro-	Cycloalkanone	D	-	D	-

[1]Detected and [2]Not detected

Table 6: List of saponins, plasticizers, and surfactants detected in water samples (n=4) collected from El-Mahmoudia canal on Summer (SU), Autumn (A), Winter (W), and Spring (SP) seasons and verified in the Willey 9, NIST 14, and AMIDS 32 metabolites databases.

Parameter	Mean ± SD	95% Confidence Limits		Reference values & Assoc.
		Lower	Upper	
[1]EC (mS cm^{-1})	0.50 ± 0.01	0.48	0.59	0.31-1.87 A[2]
pH	7.68 ± 0.05	7.60	8.99	6.5-8.4 A 7.94-8.5 B 6-8.5 C
TDS (mg L^{-1})	249 ± 14.16	226.5	291.5	500 B
Turbidity (NTU)	9.17 ± 2.66	4.92	13.41	5 D
TSS (mg L^{-1})	51.00 ± 3.36	45.64	56.35	<100 B
TS (mg L^{-1})	293.00 ± 27.83	249.00	337.5	<500 E
DO (mg L^{-1})	4.35 ± 0.10	4.19	4.51	<5 B
BOD (mg L^{-1})	23.75 ± 2.50	19.77	27.72	6-10 B
COD (mg L^{-1})	24.75 ± 3.86	18.60	30.89	10-15 B
MPN (cm^{-3})	12075 ± 3380	669	17452	5000/100 B

[1]EC-Electrical Conductivity; TDS-Total Dissolved Solids; TSS-Total Suspended Solids; TS-Total Solids; DO-Dissolved Oxygen; BOD-Biological Oxygen Deman; COD-Chemical Oxygen Demand; and MPN-Most Probable Number (concentration of viable microorganisms). Capital letters after each value corresponding to the referencing association: A-FAO (1985); B-Law 48/1982; C-WHO (1993); D-the guidelines of WHO (Chapman 1992); and E-APHA (1992).

Table 7: Physicochemical characteristics of water samples (n=4) of El-Mahmoudia stream that were collected during the autumn season of 2017 and compared to the reference recommended limits.

3.6 Mineral content of water samples

Mineral contents of Na, K, Ca, Mg, Fe, Mn, Cd, and Cl in water samples were less compared to the standard permissible limits with averages of 49.1, 7.99, 31.80, 56.40, 0.65, 0.09, and 0.002 mg L^{-1}, respectively (Table 7). The total alkalinity (HCO_3 content) in water samples of El-Mahmoudia stream was 253.3 mg L^{-1}. HCO_3 exceeded the permissible limits of the Egyptian Law (48/1982), which is <200 mg L^{-1}. Ammonia levels were elevated (NH_4; 1.42 mg L^{-1}) compared to the set limits (Table 8). But phosphates (PO_4) were decreased to about 0.01 mg L^{-1} (10 times less than the permissible values).

4. Discussions

The results reported herein showed the presence of organic chemicals, pesticides, oils, fatty acids, carboxylic acids, plant metabolites, and plasticizers in El-Mahmoudia water stream. These results were in agreement with results of Fishbein [30], APRP [31], and Saeed and Shaker [32]. The various types of pollutants might alter the aquatic environment and affect the aquatic organisms [33, 34]. Industrial wastewater was considered as a major source of pollution of Nile. There are about 129 factories discharging wastewater into the River Nile. However, wastewater effluents are partially treated [35]. In 2000, it was estimated that the total amount of reused treated wastewater in Egypt was about 1.4 billion m^3 [36]. Pesticides might leach to the water stream from irrigation water. Presence of pesticide residues, that were reported herein, were reported in other water resources in Egypt [37, 38]. About 16 OC pesticides were reported in the drains including BHC and DDT [6, 12]. Sometimes the amounts that have been detected were higher than the allowable limits for healthy water streams [39-41]. Additionally, the elevated levels of chlorpyrifos-methyl in Summer compared to autumn might be due to the application of great amounts of the insecticide to control various insects on summer crops (tomato, melons, corn, and potatoes) [42]. It was noticed that season has some effects on the levels of pesticide residues. Water samples collected in Summer had increased concentrations of pesticide residues compared to the Autumn, Spring, and Winter. These results were consistent with what was found by Azab et al. [43]. They reported that in Summer, organochlorines were significantly higher in water samples.

Parameter (mg L^{-1})	**Mean ± SD**	**95 % Confidence Limits**		**Reference Value and Association**
		Lower	**Upper**	
Sodium	49.91 ± 0.57	42.51	55.31	200 A*
Potassium	7.99 ± 0.43	5.09	10.21	12 C
Calcium	31.80 ± 4.54	24.57	39.05	<150 E
Magnesium	16.38 ± 5.30	7.94	24.82	100C
Chloride	56.40 ± 3.28	51.18	61.63	<200 A, B
Bicarbonates	253.30 ± 11.71	234.60	271.90	<200 A
Ammonia	1.42 ± 0.14	1.18	1.66	<0.5 A
Nitrate	5.89 ± 0.93	4.40	7.37	<45 A
Phosphates	0.01 ± 0.004	0.009	0.02	1 A
Ferrous	0.65 ± 0.24	0.04	1.26	<1 A, B
Copper	10.91 ± 2.59	6.79	15.03	<1.0 A, B, D
Manganese	0.09 ± 0.01	0.06	0.13	<0.5 A, B
Cadmium	0.002 ± 0.001	0.0003	0.05	0.003 B

*Capital letters after each reference value corresponds with referencing association: A-Law 48/1982; B-WHO (1993); C-BIS (2012); D-USEPA (2001); and E-Biernbaum (1995).

Table 8: Mean ± SD values of heavy metals and mineral salts in water samples (n=4) of El-Mahmoudia canal that were collected during the autumn season of 2017 and compared to assigned reference values.

Triglycerides, plant residues, and phthalates result from domestic and industrial effluents and significantly affect the quality of water. Triglycerides such as grease, oil, and fatty acids were described as major pollutants from such effluents [37, 38, 44, 45, 46]. Kandil and Abu-Taleb [47] reported that food industry discharges had significant effects on water quality. High levels of oil and grease were reported at Assiut and El-Menia [35]. Most of the organic pollutants were identified as endocrine disrupting phthalate esters, fatty acids, phenolic acids, and plasticizers that were classified as "priority pollutants" due to their severe toxicity [48]. Perceptibly, phthalates, common plasticizers, are used to impart flexibility to plastics, e.g. polyvinylchloride (PVC). It reaches the environment through volatilization, degradation, and/or leaching from plastics and drainage of industrial wastewater. Their widespread usage and stability marked them as major environmental contaminants [49]. Bioaccumulation of such compounds causes reproductive, developmental, mutagenic, genotoxic and endocrine disrupting effects to humans, animals, and aquatic organisms [50-54]. The 2,6-dihydroxybenzoic acids, that were detected in the water samples, are key metabolites of the polyaromatic hydrocarbons during wastewater treatment [55]. Moreover, it has been added to personal care products including shampoos, skin creams, cosmetics, and deodorants [56, 57]. Benzene acetonitriles and other benzyl compounds are considered as moderate to low toxicants to aquatic animals and were classified as Group 2A carcinogen [55, 58].

The physicochemical parameters of water samples were altered due to various charges of pollutants into the water stream. It was clear that various physicochemical parameters including EC, pH, TDS, turbidity, TSS, TS, DO, BOD, COD, and MPN of viable microorganisms indicate the stream water quality [27]. pH value was found to be low compared to the reference values and that might be due to the drainage of organic matter [60-63]. Chapman [61] reported that clean and wastewater had BOD values of 2 and 10 mg L^{-1}, respectively. The BOD values of waters of El-Mahmoudia stream exceeded the limits of the Egyptian Law (48/1982). The COD is an indicator of oxidation of the organic and inorganic materials in water and sewage [66]. Total suspended particles were the main source of turbidity in water that interferes with the penetration of light and affects the water salinity [59]. High inorganic matter and nutrient waste discharges might decrease the DO concentrations because of the increased microbial activity (respiration) [64, 65]. In another study, levels of BOD and COD were reported to be higher than the permissible levels at El-Gharbia, Dakahleya, and Damietta, which was an indication of pollution with non-degradable chemicals [35]. The increased values oxygen-related characteristics indicate the excessive content of the biodegradable organic matter that increases de-oxygenation of water to the level that aquatic life cannot survive [67]. However, fecal coliform was reported as a major pollutant in rivers and water streams, where it originates mainly from human sources [68].

Heavy metals concentrations were reduced to less than 33% for all studied elements. Only, the Cu element was increased compared to the standard value. Its content was 10 times greater than the permissible limit of the Egyptian Law (48/1982), WHO [28], and USEPA [48]. The elevated concentration of HCO_3 might be attributed to the decomposition of the dead phytoplankton leading to the release of CO_2. It exceeded the permissible limits of the Egyptian Law (48/1982), which is <200 mg L^{-1}. Also, elevation levels of ammonia as a result of the agricultural practices and municipal or industrial discharges [59].

Clearly, the results reported herein showed low levels of heavy metals including Cd but not for Cu. Such low values of the heavy metals (except for Cu) might be due to their deposition with sediments (silt) on the stream's bottom [69, 70, 71]. Also, contamination with Cu in El-Mahmoudia stream might be from the discharge of domestic wastewater, manufacturing processes, steam, electrical production, the dumping of sewage sludge, and Cu-fungicides application [72]. Also, elevated levels of nitrite and total alkalinity that exceeded the standard values were reported at El-Gharbia governorate [35], which might be a result of over-use of plant fertilizers. But phosphates (PO_4) were decreased to about 0.01 mg L^{-1} (10 times less than the permissible values).

5. Conclusions

Organic and inorganic pollutants: pesticides, plasticizers, plant residues were detected. Turbidity, BOD, COD, NH_4, HCO_3, MPN, and Cu were at levels above the standard safety guidelines of the Egyptian irrigation ministry laws, WHO, and FAO. Moreover, water pollutants such as heavy metals and organic pollutants occurred due to mixing domestic and industrial wastewater with agricultural water. However, all drains of Upper Egypt flow back to the Nile and many irrigation canals are contaminated with pollutants from domestic sources. The Ministry of the Environment in Egypt is observing the enforcement of the legislation regarding the treatment of industrial and domestic wastewater. It is also advocating organic farming and limiting the use of chemical fertilizers and pesticides to reduce water pollution. Moreover, specific wastewater treatment projects were established, such as the wastewater treatment plant at El-Gabal El-Asfar and the sludge conversion plants in Nawaq and El-Minia. Yet, the current study highlights important findings that would be of great help for the planning to implement more plants of wastewater treatment along the river Nile, especially in the Northern parts of Egypt.

Significance Statements

The current study revealed that the water quality of El-Mahmoudia canal is contaminated with elevated levels of organic and inorganic pollutants: pesticides, plasticizers, plant residues compared to standard safety permissible guidelines. Apparently, mixing of domestic and industrial wastes with agricultural water causes serious pollution problems.

Conflict of Interest

The authors declare no conflict of interest

Co-authors Contributions

E.R.: proposed the research problem, conducted the physicochemical and microbiological analyses and revised the manuscript; **E.E.:** proposed the research problem and revised the manuscript; **A.N.:** conducted GC/MS analyses, run statistical analysis of results, and wrote and submitted the manuscript; **Y.S.:** proposed the research problem, run the elemental analysis and revised the manuscript; **H.H.:** proposed the research problem and revised the manuscript; **K.A.A.:** proposed the research problem and revised the manuscript; and **N.A.H.:** conducted the experiments and wrote the first draft of the manuscript.

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