Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2020.26078

198 (2020) 314–322
September

The polyaromatic hydrocarbons types, concentrations, and sources in particulate matter and aquatic plant in Tigris River, Baghdad, Iraq

F.M. Hassan[a,*], A.H.M.J. Al-Obaidy[b], S.M. Khalaf[c]

[a]*Deptmentof Biology, College of Science for Women, University of Baghdad, Baghdad, Iraq, email: fikrat@csw.uobaghdad.edu.iq (F.M. Hassan)*
[b]*Environmental Research Center, University of Technology, Baghdad, Iraq, email: jawaddhy@yahoo.co.in (A.H.M.J. Al-Obaidy)*
[c]*Ministry of Education, Second Karkh Educational Directorate, Baghdad, Iraq, email: shathamohammad1978@gmail.com (S.M. Khalaf)*

Received 24 August 2019; Accepted 6 May 2020

ABSTRACT

In recent years, was focused on the leaks out of oil into the Tigris River in the north of Baghdad Province, and what is their fate and the environmental impacts; therefore this study was investigated on the concentrations, origin and fate of polycyclic aromatic hydrocarbons (PAHs) compound in the river from the periods between July 2017 to April 2018. The results showed that twelve PAHs compounds were detected in suspended particulate matter and an aquatic plant (*Ceratophyllum demersum*) of the river and the highest concentrations of these compounds were found in suspended particulate matter samples. The highest value of PAHs in particulate was 8391.37 μg kg^{-1} dry weight of ovalene in March 2018 and the most abundant compound was naphthalene, the origin of PAHs in particulate was pyrogenic and petrogenic. The maximum value of PAHs in the aquatic plant was 8,809.98 μg kg^{-1} dry weight of acenaphthylene in January 2018, while the most abundant compound was chrysene, which is one of the carcinogenic compounds; the origin of PAHs in *C. demersum* was pyrogenic. According to these results, it can conclude that particulate has large concentrations of PAH compounds due to their hydrophobic nature. High accumulation of PAHs was observed in *C. demersum*, which indicated the possibility to use for bioremediation for PAHs. Based on the results, the superoxide dismutase enzyme could be considered as a biomarker of PAHs in an aquatic plant.

Keywords: Origin; Pollution; Polycyclic aromatic hydrocarbons (PAHs); SPM; Superoxide dismutase (SOD) enzyme

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1. Introduction

A lot of attention has been raised due to the environmental properties of persistent organic pollutants (POPs). Polycyclic aromatic hydrocarbons (PAHs) are a ubiquitous group of POPs causing carcinogenic, toxic and mutagenic damages and enter the food chain in an ecosystem. Most PAHs compound introduced into the environment by different natural and other agro-industrial activities [1].

PAHs are a class of diverse organic compounds consisting of two or more aromatic rings combined with carbon and hydrogen atoms in linear, angular, or cluster arrangements. Pure PAHs are usually colored, crystalline solids at ambient temperature. Depending on the physical properties of PAHs, their compounds tend to bind to soil particles, the high molecular weight (HMW) PAHs (PAHs with more than four rings) are less water-soluble, less volatile and more lipophilic than lower molecular weight (LMW) ones [2].

PAHs are important contaminants that can appear with high concentrations due to the growing industrialization and needs for oil exploration and transportation. Large oil spills can cause irreversible damage to the aquatic environment. Two major examples of such damages to the Iraqi water surfaces are the oil leak out into the Tigris River in

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* Corresponding author.

2014 when the pipeline was bombed [3], and the oil leak out into the Arabian Gulf during the Gulf War in 1991 [4].

The PAHs have a hydrophobic property and tend to adsorption on particulate matters or to sink into the sediment of aquatic systems, these pollutants enter into aquatic systems by different natural and anthropogenic sources [5,6].

PAHs are compounds with two or more fused rings forming a large group of organic pollutants. The occurrence of PAHs has been detected in both aquatic and terrestrial environments. These compounds have a high affinity to exist in organisms, which have high fatty acids contents [7].

Albers [8] revealed that PAHs were highly toxic in the aquatic system due to it influenced by physiological processes and natural photooxidation. Fate and behavior of PAHs are affected by a number of their physical and chemical properties such as volatilization, dissolution, adsorption onto suspended solids and subsequent sediments in aquatic ecosystems [9].

High molecular weight PAHs are difficult to uptake by macrophytes due to their lipophilic nature and cell wall structure [10]. Still, the thin cuticle for macrophytes gives the plant the ability to uptake the bioactive components [11].

Several studies conducted in China to evaluate PAH concentrations, Qiu et al. [12] showed that average concentrations ∑15PAHs in Deep Bay, South China while the maximum values (69.4 ng L^{-1}, 429.1 ng g^{-1} and 353.8 ng g^{-1} dry weight) were recorded in seawater, suspended particulate matter (SPM), and surface sediment, respectively. Yang et al. [13] found that the PAHs in the Jinjiang River reported that higher PAHs concentrations recorded in suspended particulate matter in comparison with the dissolved phase.

Haiyan et al. [14] found the three and four rings of PAH compounds in water and the suspended particulate matter in Pearl River, Dongjiang River, and the Pearl River Estuary, while the study reported a high PAHs concentration found in fish collected from the estuary and low concentration in freshwater fish. Wang et al. [15] illustrated the role of suspended particulate matter in the transportation of these PAH pollutants. Zheng et al. [16] reported that the high concentration of total concentrations was in suspended particulate matter and followed by sediments while a low concentration recorded in water in the Daliao River estuary and the adjacent area, China. Another study on seawater recorded different concentrations of PAH compounds in water (high concentration) and suspended phase (low concentration) throughout the 12 months of the study [1].

The PAH compounds in macrophytes were recorded in different aquatic systems. Al-Khatib [17] found in the study in Hor Al-Howaiza, the concentrations of PAHs in two species (*Ceratophyllum demersum* and *Paspalum paspaloides*) were higher in summer than autumn. Latif et al. [18] showed that an average concentration of PAHs in three types of plants in marshland was lower than the guideline value. Hassan et al. [19] reported the different concentrations of PAH compounds in four aquatic plants in the Hilla River. They found in the *Phragmites australis*, PAH concentrations were ranged from 0.425 μg g^{-1} dry weight (DW) for B(ghi)P to 299.424 μg g^{-1} DW for B(b)F, while in *Potamogeton perfoliatus* was 0.354 to 235.84 μg g^{-1} DW for B(b)F and B(ghi)P, respectively, *C. demersum* 0.996 to 162.942 μg g^{-1} DW for Ant and B(ghi)P, respectively, and *P. pectinatus* 0.383 to 99.87 μg g^{-1} DW for Ant and Nap, respectively. Jazza et al. [20] showed that two species of macrophytes (*C. demersum* and *Paspalum paspaloides*) in the Kahlaa River could be used as bioindicators of PAHs compounds because they have the capability of accumulating PAHs composites.

The lipids content and its structures in the plant are affected by the presence of petroleum hydrocarbons and also reduce the plant biomass through the distribution of metabolic reaction or kill the embryo of the plant seed, this damage ability of these pollutants caused by the ability of low molecules of PAH to penetrate through cell membrane [21–23].

Four aquatic habitats were selected in a study according to the distance of the pollution sources (industrial and urban) in Turkey. This study recorded eight PAHs compounds in the leaves of an aquatic plant (*Alisma plantago*), the highest of total PAH concentration recorded in station affected by industrial, urban and highway in comparison with distance from the pollution sources [24].

The authors reported that origins and sources (petrogenic or pyrogenic) of PAHs from various environmental media could be identified using the diagnostic ratio as a widely used technique. These ratios values depend on P/Ant, Ant/(Ant + P) and LMW/HMW if there values as follows: <10, >0.1 and <1, respectively so the origin of PAHs is pyrogenic while if their values >10, <0.1 and >1, respectively is petrogenic origin [25,26].

PAHs were associated with elevated cancer in organs of the human body [27]. According to US Environmental Protection Agency (EPA) classification, seven compounds of PAHs were identified as carcinogens benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, chrysene, dibenzo[ah]anthracene, and indeno[1,2,3cd]pyrene. The mutagenic carcinogenic properties of these compounds resulted in their inclusion as measurements in different environmental metrics such as those related to the quality of air, water, particulate, plant, sediment [28].

The objectives of the current study were to investigate concentrations and compositions of PAHs and to elucidate their potential sources and determined carcinogenic composites of PAHs in SPM and *C. demersum* in Tigris River within Baghdad City.

2. Material and methods

2.1. Sampling area

Tigris River and Euphrates River are the primary water resources for different uses since they pass through the most important cities in the country [29]. Tigris River divides Baghdad City (33°14′-33°25′ N, 44°31′-44°17′ E) into two sections (Karkh and Risafa) with a flow direction from north to south. The Iraqi climate in Baghdad City is arid to semi-arid climate, with a 151.8 mm annual rainfall [30]. Three sites along the Tigris River were chosen in order to estimate concentrations of PAHs. Site 1 (Alkreaat) was located upstream. In contrast, site 2 (Jadriya) was situated in the midstream and the site 3 (General company of vegetable oil/Rasheed plant) was located at the downstream (Fig. 1, Table 1), according to global position system locations.

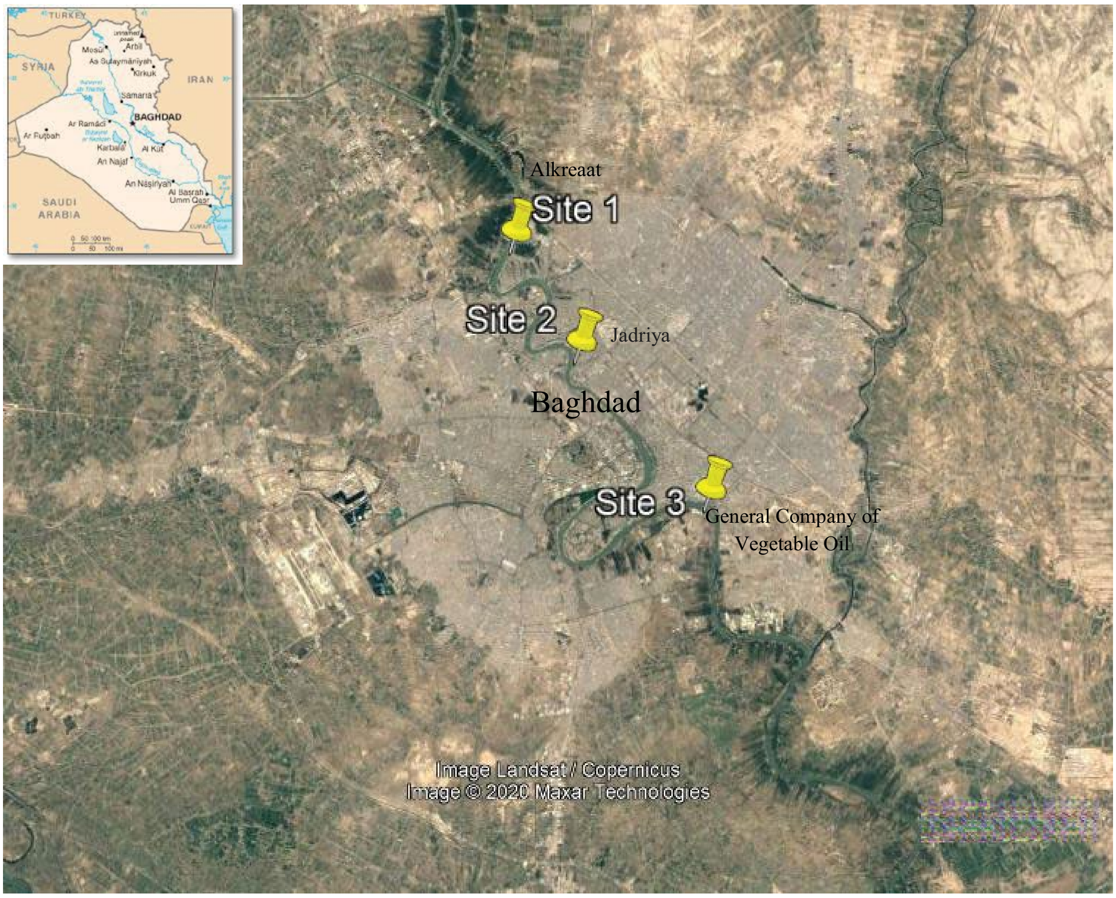

Fig. 1. Map of the study sites.

Table 1
Geographical positioning system (GPS) of the selected sites

Sites	Catchment area	Longitude (eastwards)	Latitude (northwards)	Activities of catchment area
S1	Kreaat	″20.28′44°20	″12.36′33°24	Agriculture
S2	Jaderiya	″30.42′44°22	″12.62′33°21	Urban
S3	Rasheed plant	″48.20′44°26	″10.78′33°17	Industrial

2.2. Sampling

The samples were collected monthly from July 2017 to April 2018, and then considered for two seasons; dry season (July–November 2017) and wet season (December 2017–April 2018). The season type (dry/wet) was adopted to the values of relative humidity when its value in season ≤50% is considered as a dry season. In comparison, >50% was a wet season provided by the Ministry of Transportation, Iraqi Meteorological Organization and Seismology [31].

Water samples were collected from the depth of 50 cm by a pre-cleaned dark glass bottle in a metallic holder around the bottle connected with a rope that was lowered into the water and allowed resting briefly, ensuring that it is filled with water. Firstly, macrophytes were washed with tap water followed by distilled water, dried under 15°C, covered in aluminum papers, labeled and stored until analysis [32]. Procedures for selected water quality constituents followed for analysis have been by [32].

2.3. PAHs analysis

The extraction of PAHs from particulate was described by Li et al. [14], Kafilzadeh et al. [33] and Yuan [34]. Plants were dried well, sieved through a 63 mm mesh sieve, and then 10 g was weighed and placed in a flask before being grinded well in a metal blender. Then 50 ml of acetone was added and mixed by hands for 5 min, followed by a step of soaking in a dark-cold glass container three times. After that, all steps that were followed in the procedure of extracting PAHs from particulate were applied to extract PAHs from the soaked plant mixture [34].

The analysis of PAHs from particulate and macrophytes extracts was performed by gas chromatography (Shimadzu, 2014, Japan) (column oven (SE-30 m) = 150°C (hold 1 min) –290°C (10°C/min) temperature of injector = 280°C, temperature of detector (FID) = 310°C, pressure100KPa, injection volume = 1u l) [35]. The percentage recoveries showed that the uncertainties in the methods of analyses used were within the acceptable limit and the procedure adopted was effective. The median percent recovery for all PAHs reported as 96.7%.

2.4. Extraction of super oxide dismutase

A fresh plant sample was taken (0.5 g) after washing with tap water and DW. Then the liquid nitrogen was added to the sample, which was grinded in the mortar and suspended in 1.5 ml of homogenization buffer solution. The suspension was centrifuged next with a cooling centrifuge (14,000 rpm in 30 min at 4°C). The supernatant was then used for enzyme assay [36]. The specific activity of the enzyme was estimated depending on the riboflavin/NBT method [37]. The measurements of the activity of superoxide dismutase (SOD) have been described in Rasheed and Aziz [38]. The protein concentrations were determined according to the Bradford method [37]. The protein concentration in the samples was calculated using the standard curved [38,39]. The present study results were analyzed statistically by Canoco for windows 4.5 Canonical correspondence analysis (CCA).

3. Results and discussion

3.1. PAHs in particulate

The concentrations of PAHs in particulate are shown in Table 2.

The highest concentration of total PAHs was recorded in particulate samples; The PAHs compound is characterized by its tendency to be adsorbed on colloidal material in the water column and for that, the highest value of PAHs was recorded in particulate parts in this study [40]. PAH concentrations in the water column were higher in the dry season than that in the wet season. The PAHs tend to adsorb on colloidal particles in the water column due to their low solubility. They are eventually settled into the sediment [11,28]. The highest value of PAHs in particulate was 8,391.37 $\mu g\ kg^{-1}$ dry weight that observed for Ova in March 2018 and the most abundant compound was N. According to CCA results (Fig. 2), that's due to water flow (WF), total suspended solids (TSS), and turbidity (TUR). The origin of PAHs in particulate, according to Table 3, was pyrogenic in S1, while it was pyrogenic and petrogenic at S2 and S3, respectively. The values of the LMW/HMW ratio for sites 2 and 3 were reported as 1.67 and 1.06, respectively, which suggests that the PAHs are from a combustion source. A possible contribution source of PAH in these sites is the effect of petroleum activities around these sites [7]. The carcinogenic compounds of PAHs in particulate were B[a]Ant, B[a]Fl, Chry and dibenzo(a,h)anthracene (DAH), and their concentrations have exceeded the limit values.

3.2. PAHs in macrophytes

The mean concentrations of PAHs in macrophytes are shown in Table 4.

PAHs concentrations in macrophytes samples ($\mu g\ kg^{-1}$ dry weight) were 205.96 in the dry season and 884.39 in the wet season. Hassan et al. [19] explained that summer in Iraq is characterized by long daylight, which led to increasing the photo-degradation of PAHs; moreover, the less ability to uptake these compounds were by the plant. Therefore, this study recorded a higher concentration of PAHs in the wet season compared with the dry season [41]. This is in agreement with the present results because of the high molecule weight characteristics of PAHs whose uptake problematic by the plant. These may be available if the plant has a thin cuticle cover and other cell wall prosperities [16,30]. The maximum value of PAH compounds was 8,809.98 $\mu g\ kg^{-1}$ of Acy in January 2018, while the most abundant compound was Chry, which is one of the carcinogenic compounds.

According to CCA results (Fig. 3), PAH's high concentrations in macrophytes are possibly due to total dissolved

Table 2
Descriptive data of PAHs in particulate

Variables	Mean	Median	Minimum	Maximum	Standard deviation
Acy	437.08	536.79	ND	614.50	216.70
Ant	809.72	987.44	ND	1,177.37	490.49
B[a]Ant	69.33	69.33	ND	69.33	
B[a]Fl	ND	ND	ND		
Chry	946.78	933.04	ND	1,272.33	242.66
DAH	560.30	560.30	ND	560.30	
F	3,146.19	3,146.19	ND	6,168.00	4,273.49
N	415.48	294.41	ND	830.51	251.54
Ova	6,176.66	6,176.66	ND	8,391.37	3,132.08
P	2,142.20	2,142.20	ND	2,142.20	
Pyr	85.81	85.81	ND	102.29	23.31
Tetr	ND	ND	ND		

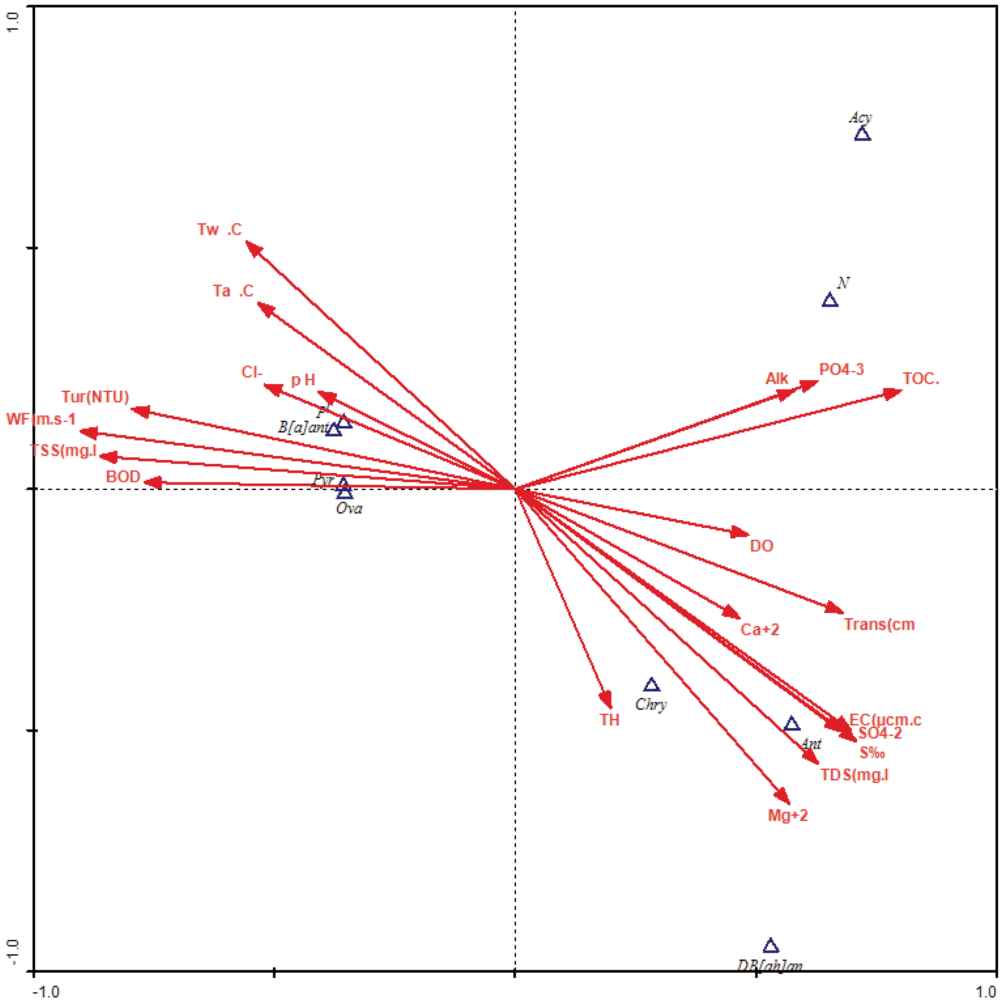

Fig. 2. CCA of PAHs in particulate.

solids, Mg, dissolved oxygen, total organic carbon, and pH. PAHs origin was pyrogenic in macrophytes at all sites.

A *t*-test revealed (significant differences) *P*-value < 0.05 of some PAHs composites such as Acy, Chry, N, Pyr, and specific activity of SOD, a significant relation was found between N and Protein concentration of SOD. Depending on the result of the PAHs compounds ratios, illustrated in Table 5, PAHs origin in macrophytes was pyrogenic.

The carcinogenic compounds of PAHs in macrophytes were B[a]Ant, B[a]Fl, Chry, and DAH, and their concentrations have exceeded limit values.

3.3. Super oxide dismutase results

Table 6 shows inhibition ratio (IR), enzyme activity (EA), protein concentration (P con.), and specific activity (SA) of SOD in macrophytes for 6 months, while Table 7 shows *T*-Test of IR, EA, P con., and SA of SOD enzyme with PAHs concentration compounds in macrophytes, and Table 8 shows a correlation of PAHs concentrations in macrophytes and SA of SOD enzyme.

The test of SOD activity is to measure its ability to prevent the chemical reduction of nitroblue tetrazolium. It has been shown that different species accumulate and tolerate PAHs [42]. The relationships between exposure to pollutants and alterations in several enzymes have increased their use as environmental biomarkers in aquatic ecosystems [43].

Environmental stresses such as PAHs pollution might be induced by the activity of SOD as shown in this study [44]. A few studies showed that an increase in SOD activity when exposing the Paraquate treatments on maize

Table 3
Characteristic values of selected molecular ratios for pyrogenic and petrogenic origins of PAHs in particulate during study period

	P/Ant	Ant/(Ant + P)	LMW/HMW
Pyrogenic origin	<10	>0.1	<1
Petrogenic origin	>10	<0.1	>1
S1	ND	1.00	0.56
S2	2.13	0.32	1.67
S3	2.50	0.28	1.06

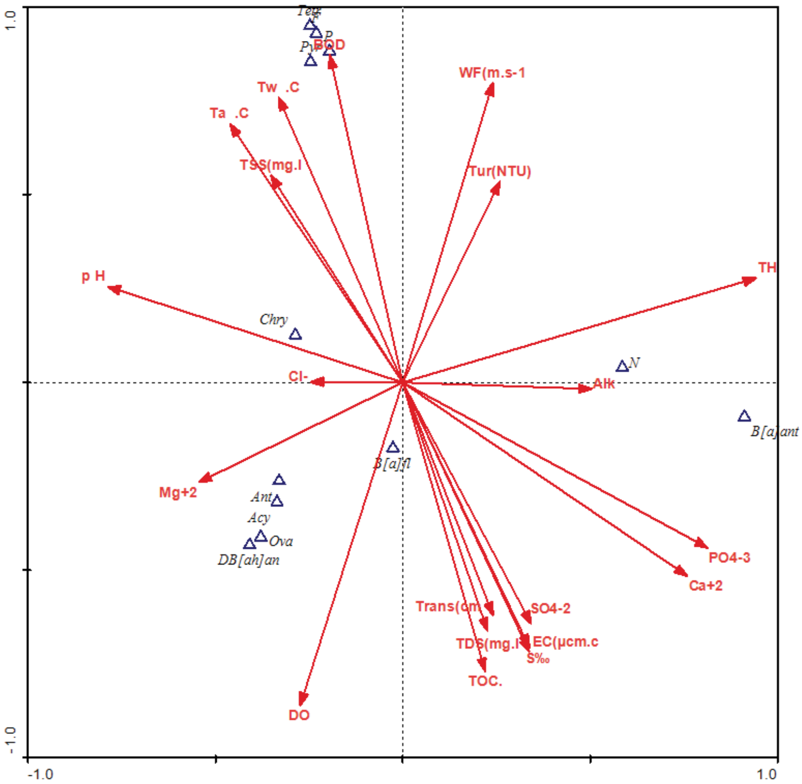

Fig. 3. CCA of PAHs in macrophyte.

Table 4
Descriptive data of PAHs in macrophyte

Variable	Mean	Median	Minimum	Maximum	Standard deviation
Acy	1,290.26	255.66	ND	8,809.98	3,042.78
Ant	94.80	25.60	ND	616.51	211.25
B[a]Ant	6,478.42	6,478.42	ND	6,478.42	
B[a]Fl	234.42	203.70	ND	493.98	245.64
Chry	755.26	307.85	6.66	4,167.93	1,254.94
DAH	1,070.46	1,070.46	ND	1,070.46	
F	402.16	267.22	ND	1,661.40	535.27
N	552.82	129.67	2.67	2,847.47	982.55
Ova	664.89	76.38	ND	2,504.94	1,228.42
P	136.74	93.09	ND	448.21	169.81
Pyr	5.83	7.10	ND	9.47	3.95
Tetr	193.93	165.42	ND	327.49	127.83

leaves, these results were interpreted by the higher levels of specific SOD isozymes [44,45]. The current study has been concluded that is possible for the relation between PAHs pollution and SOD activity in aquatic plants.

4. Conclusions

The carcinogenic compounds such as (PAHs) were detected in different metrics in the Tigris River. Particulate matter has large concentrations of PAH compounds due to their hydrophobic nature. High accumulation of PAHs was observed in macrophytes, which can be used, it as

Table 5
Characteristic values of selected molecular ratios for pyrogenic and petrogenic origins of PAHs in macrophyte during study period

	P/Ant	Ant/(Ant + P)	LMW/HMW
Pyrogenic origin	<10	>0.1	<1
Petrogenic origin	>10	<0.1	>1
S1	0.57	0.64	0.73
S2	0.75	0.57	0.52
S3	0.75	0.57	0.17

Table 6
IR, EA, Pcon, and SA of SOD enzyme

Months	Inhibition ratio	Enzyme activity (u ml^{-1})	Protein concentration (mg ml^{-1})	Specific activity (u mg^{-1})
November	3.17	1.90	0.11	20.49
December	14.67	8.80	1.22	6.04
January	72.11	43.26	0.16	272.46
February	8.40	5.04	0.15	32.98
March	18.50	11.10	0.10	110.32
April	14.40	8.64	0.11	87.39

Table 7
T-test of PAHs and SOD enzyme in macrophyte

	Mean	Standard deviation	Standard Error	*t*-value	*df*	*p*
Acy	891.10	284.07	164.01	5.43	2.00	0.03
Ant	338.78	156.56	90.39	3.75	2.00	0.06
B[a]Ant	22.89	14.38	10.17	2.25	1.00	0.27
B[a]Flu	348.84					
Chry^{-}	2,274.94	863.58	498.59	4.56	2.00	0.04
DAH	1,070.46					
F	815.17	433.24	250.13	3.26	2.00	0.08
N	947.96	317.82	183.49	5.17	2.00	0.04
Ova	1,091.94	610.48	352.46	3.10	2.00	0.09
P	237.88	130.57	75.38	3.16	2.00	0.09
Pyr	7.13	0.96	0.56	12.82	2.00	0.01
Tetr	138.79	108.80	62.82	2.21	2.00	0.16
IR	131.24	49.59	28.63	4.58	2.00	0.04
EA (u ml^{-1})	78.74	29.75	17.18	4.58	2.00	0.04
P con. (mg ml^{-1})	1.85	0.78	0.45	4.12	2.00	0.05
SA (u mg^{-1})	529.68	155.58	89.82	5.90	2.00	0.03

Table 8
Correlations of PAHs conc. in macrophyte and specif. act. SOD enzyme

Variable	Correlations												
	Acy	Ant	Chry	F	N	Ova	P	Pyr	Tetr	IR	EA (u ml^{-1})	P con. (mg ml^{-1})	SA (u mg^{-1})
Acy	1.00												
Ant	–0.24	1.00											
Chry	0.84	–0.73	1.00										
F	0.97	0.02	0.67	1.00									
N	–0.80	0.78	–1.00	–0.61	1.00								
Ova	0.81	–0.77	1.00	0.63	–1.00	1.00							
P	–0.44	0.98	–0.86	–0.20	0.90	–0.89	1.00						
Pyr	–0.86	0.70	–1.00	–0.70	0.99	–1.00	0.84	1.00					
Tetr	–0.49	–0.73	0.06	–0.70	-0.13	0.12	–0.56	–0.02	1.00				
IR	–0.13	0.99	–0.65	0.13	0.70	–0.69	0.95	0.62	–0.80	1.00			
EA (u ml^{-1})	–0.13	0.99	–0.65	0.13	0.70	–0.69	0.95	0.62	–0.80	1.00	1.00		
P con. (mg ml^{-1})	0.47	–0.97	0.87	0.22	–0.91	0.90	–1.00	–0.86	0.54	–0.94	–0.94	1.00	
SA (u mg^{-1})	–0.96	0.50	–0.96	–0.85	0.93	–0.94	0.68	0.97	0.23	0.40	0.40	–0.70	1.00

bioremediation of PAHs in river water. However, it is clear that particulate matter has higher PAHs concentrations because of the resuspension of sediments due to water flow. Furthermore, based on the results, the SOD enzyme could be considered as a biomarker of PAHs in macrophytes.

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