

Evaluation of Heavy Element Contamination Levels with the Use of Different Species of Hydrophilidae (Coleoptera) in Erzurum Province of Turkey

Zeynep AYDOGAN^{1*} Umit INCEKARA² Ali GUROL³

¹ *Narman Vocational School, Atatürk University, Narman, Erzurum/TURKEY*

² *Department of Biology, Faculty of Science, Atatürk University, Erzurum/TURKEY*

³ *Department of Physics, Faculty of Science, Atatürk University, Erzurum/TURKEY*

* Corresponding author (Sorumlu yazar): zeybionep@hotmail.com

Received (Geliş tarihi): 15.02.2018 Accepted (Kabul tarihi): 12.06.2018

ABSTRACT: *This study evaluates the level of some heavy elements (Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Sr, Pb) in Erzurum Province, and to observe whether Hydrophilidae (Coleoptera) is a useful candidate for biomonitoring studies. For this purpose, water, sediment and hydrophilid samples were collected from four sites on June, July, August 2014, Erzurum (Turkey). Heavy elements levels in insect, sediment and water samples which are at the same place were measured by Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer. The results have showed that the hydrophilids were contaminated by their environments, therefore accumulated some elements at higher concentration. The sediments heavy element analysis indicated that among the fourteen heavy elements Cr was maximally accumulated, followed by Ni and Fe. In all sampling sites, Ti, Fe, Ni, Cu, Br, Pb concentrations in water and sediment were measured, and every insect species accumulated Ti, Fe, Ni, Cr, Zn, Br, Sr, Pb in different levels. The water samples' results were compared with national water quality criteria. Some heavy elements' concentrations exceed the allowable limits.*

Keywords: *Aquatic insect, bio-monitor, EDXRF, pollution, wetland.*

Erzurum İlindeki Farklı Hydrophilidae (Coleoptera) Türlerini Kullanarak Ağır Element Kirliliğinin Değerlendirilmesi

ÖZ: *Bu çalışma, Erzurum civarındaki bazı ağır element (Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Br, Sr, Pb) miktarlarını ve Hydrophilidae (Coleoptera) türlerinin biyolojik izleme çalışmaları için uygun aday olup olmadığını değerlendirmektedir. Bu amaçla, su, sediment ve hydrophilid örnekleri Haziran, Temmuz, Ağustos 2014 aylarında Erzurum'daki dört istasyondan toplandı. Aynı lokalitelerden alınan böcek, su ve sediment örneklerindeki ağır element miktarları Enerji Dağılımlı X-Işınları Flüoresan (EDXRF) spektrometresi ile ölçüldü. Sonuçlar böceklerin buldukları ortamdan kontamine olduklarını, bu nedenle, bazı elementleri yüksek miktarda biriktirdiğini göstermiştir. Sediment ağır element analizleri, 14 element arasında en çok biriken elementin Cr olduğunu bunu Ni ve Fe elementlerinin takip ettiğini gösterdi. Tüm istasyonlarda Ti, Fe, Ni, Cu, Br, Pb konsantrasyonları su ve sedimentte ölçüldü; tüm böcek türleri Ti, Fe, Ni, Cr, Zn, Br, Sr, Pb elementlerini farklı seviyelerde biriktirdi. Su örnekleri ölçüm sonuçları ulusal su kalite parametreleri ile karşılaştırıldı. Bazı elementlerin seviyesi, izin verilen limitlerin üzerinde bulundu.*

Anahtar Kelimeler: *Sucul böcek, biyomonitör, EDXRF, kirlilik, sulak alan.*

INTRODUCTION

Environmental pollution with toxic elements has increased significantly in recent years as a result of rapid industrialization, entrances of the number and amounts of elements to the biotic and abiotic habitats continue to grow and cause serious

ecologic problems. Heavy elements are normally found in varying concentrations in all ecosystems because they are natural constituents of the Earth's crust and human activities have changed the biogeochemical balance of these heavy elements (Nriagu, 1996). These elements are critical

contaminants because they are toxic, persistent, non-biodegradable, accumulated in biota and biomagnified in food chain, thus this accumulation will damage human health when their concentrations are high level (Jiang *et al.*, 2015).

Aquatic insects are a vital component of both aquatic and terrestrial ecosystems due to their roles as predators, parasites, herbivores, saprophagous and pollinators and provide important source of protein for various organisms such as birds, fish and other in vertebrates (Rosenberg *et al.* 1998). During their life cycle, insects present in different feeding habits and positions in the food web. Aquatic insects are excellent biological monitors of heavy metal pollution because, they can accumulate elements whether or not these elements necessary to their metabolism. They can reflect element concentrations even the pollution ceases (Nehring, 1976). Elements enter their body via food, water, sediments or air and transfer to higher food web such as fish, birds (Duran *et al.*, 2007; Gall *et al.*, 2015). The toxic effects of elements can affect the insects in several ways, such as, changing the duration of growth, body weight, hatching success, number of eggs laid, reducing the pupation rate, increasing mortality, and declines in population size (Chen *et al.*, 2011). Recently, most researchers have been using aquatic insects to biomonitor the element pollution because they are present in almost every imaginable habitat, often abundant, easily sampled and easily identifiable (Nehring, 1976; Wahizatul *et al.*, 2011). Hydrophilidae, also known as water scavenger beetles, is large family of the order Coleoptera, zoo-geographically have a wide range of distribution. The adults are generally aquatic or semi-aquatic and most of their larvae are aquatic. The adults emerge during summer days and are mostly saprophagous, feeding on decaying organic matter, whereas larvae are predaceous, preying on invertebrates (Fikacek *et al.*, 2010). In adult stage hibernation usually occurs, besides sometimes the eggs also hibernate (Hansen, 1987). To assess anthropogenic impact, aquatic insects are the most used organisms in biomonitoring studies (Rosenberg *et al.*, 1998). But biomonitoring studies with aquatic insects have been often made with their larvae.

Many things can change the water's health quality. For protecting the wetlands, in accordance with EPA, they need to be monitored over the time (Anonymous, 2012). Biomonitoring studies can tell us how healthy the water in rivers, stream, creeks, swamp and wetlands is. Biomonitoring are commonly used to evaluate the levels of bioavailable pollution in the environment. Therefore, the objective of this study is to measure heavy element level of water, sediment, and adult hydrophilid and to certify the bio-monitor potential of them.

MATERIAL AND METHODS

Sampling sites

Erzurum is the biggest and developed city of Eastern Anatolia and surrounded by mountains, which have higher altitudes than the central and western part of Turkey. The photos of study area and location of the sampling sites are shown in Figure 1. Information about the sampling sites is given in Table 1. Industrialization in Erzurum has not reached the desired level but anthropogenically heavy element sources are transportation, industrial activities, fossil fuels, agriculture and other human activities. In this study, the samples were taken from industrial, roadside and suburban area.

Sampling site 1: This station is located in between 39°55'19 N/40°40'01 E coordinates (Figure 1.1), in the west of Aşkale Cement Plant. Contamination sources are traffic pollution and spread ash emissions from cement plant.

Sampling site 2: This station is located in between 39°55'39' N/40°40'41 E coordinates (Figure 1.2), in the east of Aşkale Cement Plant. Pollution sources are traffic and spread ash emissions from the cement plant.

Sampling site 3: This station located in between 40°02'32'N/41°20'04'E coordinates (Figure 1.3), on D 950 highway. Sources of pollution are traffic and domestic usage.

Sampling site 4: Akdağ village located at 20 km north of Erzurum, between 40°06'05" N/41°21'36" E coordinates (Figure 1.4). In this station, there is Akdağ hot spring. Because the local people engage

in livestock, the source of pollution is livestock. Map and photos of sampling sites in Erzurum is shown in Figure 1.

Collection of the samples

All samples were collected and analyzed as described in Aydoğan *et al.* (2016). Insects and their habitat's sediment and water were collected monthly from four sites in June to August 2014. The samples were collected three times in every station. Insect samples were collected via 1 mm pores sieve and killed 70% ethyl acetate and carried to the laboratory for identification. Insects' male genitalia were used for identification (Hansen, 1987). The species belong to three different genera *Anacaena* Thomson, 1859; *Hydrobius* Leach, 1815 and *Hydrochara* Berthold, 1827 were collected from four sites in Erzurum. Identified species were as follow; *Hydrobius fuscipes* (Linnaeus, 1758), *Anacaena limbata* (Fabricius, 1792), *Hydrochara caraboides* (Linnaeus, 1758).

Following to the APHA standarts (Anonymous, 2005), the sediments were taken from 30 cm depth benthic zone via plastic shovel and 500 ml water

samples were taken from 0.5 m depth then put in a bottle and added 5 ml HNO₃.

Elemental analysis

After identification, the samples were dried in an oven at 80 °C for 36 hours. An EDXRF spectrometer with an annular ²⁴¹Am radioactive source having 1 Ci activity and an HPGe detector having resolution 1~80 eV at 5.9 keV was used. Samples were excited by using 59.5 keV photons, emitted from ²⁴¹Am radioactive source. X-ray spectra were collected with HPGe detector which resolution is ~180 eV. Source/Sample distance was 35.5 mm. To eliminate elements and particles which found in the air, vacuum was used. All sample holder with Mylar films on both sides was used for water and sediment samples. Samples' measurement time was four hours. To measure heavy element content of insect samples, 13 mm diameter pellets of each species were made.

RESULTS

Results indicated that concentration of fourteen studied elements in all samples showed differences. The concentrations have some uncertainties due to EDXRF (maximum ~5%). These uncertainties of possible error sources are listed in Table 2.



Figure 1. Map and photos showing the sampling sites in Erzurum. (Sampling site 1; Sampling site 2; Sampling site 3; Sampling site 4).

Resim 1. Harita ve resimler Erzurum'daki örnekleme alanlarını göstermektedir. (Örnekleme noktası 1; Örnekleme noktası 2; Örnekleme noktası 3; Örnekleme noktası 4).

Table 1. Description of the four sampling sites.

Çizelge 1. Dört örnekleme istasyonu ile ilgili açıklama.

Sampling Site	Altitude (m)	Coordinates	Information about sampling sites	Notes on near environment
Örnekleme alanı	Rakım (m)	Koordinatlar	Örnekleme alanları hakkında bilgi	Yakın çevresi hakkında notlar
1	1636	39°55'19"N/40°40'01"E	Cement Factory(Western front)	Ash emission from cement factory, traffic
2	1669	39°55'39"N/40°40'41"E	Cement factory (Eastern front)	Ash emission from cement factory
3	1765	40°02'32"N/41°20'04"E	Erzurum-Tortum Road	Traffic and domestic
4	1826	40°06'05"N/41°21'36"E	Akdağ Village	Livestock pollution

Table 2. The error sources in the experimental results.

Çizelge 2. Deney sonuçlarındaki hata kaynakları.

Nature of uncertainty	Uncertainty (%)
Belirsizliğin doğal nedeni	Belirsizlik (%)
Counting Statistics (Sayım istatistikleri)	~1.00
Systematic Errors (Sistemik Hatalar)	~2.00
Peak Evaluation Procedure (Tepe Değerlendirme Prosedürü)	~3.00
Fundamental Parameter Methods (Temel Parametre Yöntemleri)	~3.00

Concentrations of Ti, Fe, Ni, Cu, Br and Pb were measured in all sampling sites' water and sediment samples. Concentrations of Ti, Cr, Zn, Fe, Ni, Br, Sr and Pb were measured in all insect species. According to the results, Fe had the highest concentration, followed by Cr, Ni. Among the rest of heavy elements Ti had the highest concentration in sediments of sampling site 4. V measured only sediment of sampling site 1. Mn had the highest concentration in only sediment of Sampling Site 2. Co had nearly same and the highest concentration in sediments of Sampling Site 3 and 4. Cu, Zn, As, Se, Br, Sr and Pb had the highest concentrations in sediment of Sampling Site 1. In a similar vein, in water, Ti had the highest concentration, followed by V, Cr. Se and Sr did not measured in all sampling site of waters. The rest of the heavy elements in water had nearly same concentration. The study results indicate that *Hydrobius fuscipes* is the best accumulator for certain metals. *H. fuscipes* is the most abundant and also most element accumulator specie in regard to Ti, V, Cr, Mn, Fe, Sr and Pb; also this insect found in all sampling sites. The highest element concentrations in *H. fuscipes* are Ti, V, Cr, Mn, Fe, Se, Sr and Pb; in *H. caraboides* are Co, Ni, Cu, Zn, As and Br. Except V, Mn and Cu all elements were determined in *H. fuscipes*; in *H. caraboides* only

Se was not determined. Fourteen elements were measured in *Anacaena limbata* but the level of these elements is not high. As it is seen in Table 3, *H. fuscipes* accumulated relatively more Ti, V, Cr, Fe, Mn, Sr and Pb, but Co, Cu, Ni, As, Zn and Br less accumulated as compared to *H. caraboides*. Variation in the element concentrations between species may be due to differences in prey choice, uptake, excretion and life history (Gall *et al.* 2015). The most abundant elements in the insects were Ti, Pb, Fe, Ni, Cr, Co, As, Zn, Sr and Br. In Table 4, water element concentration was lined up as; Ti>V>Cr>Mn>Fe>Pb>Ni>Co>Cu>As>Br>Zn, but Se and Sr concentration were not determined, and in sediment samples; Fe>Cr>Mn>Co>Ni>Sr>Se>As>Pb>Cu> Br>Zn>Ti>V. Mean concentrations and standart deviations were given in Tables 3 and 4.

The obtained water heavy element levels were compared with Turkish Water Pollution and Control Regulations (TWPCR) (Anonymous, 2004). There are 4 quality classes according to TWPCR: high quality water (I), weakly polluted water (II), polluted water (III) and highly polluted water (IV). The recommended values for Turkish Standards and studied water sample levels were given in Table 5.

Table 3. Average heavy element concentrations in insect samples (ppm).

Çizelge 3. Böceklerdeki ortalama ağır element konsantrasyonları (ppm).

Heavy Element Ağır Element	Average heavy element concentrations (ppm - Mean ± SD) Ortalama ağır element konsantrasyonları (ppm - Mean ± SD)							
	Sampling site 1 Örnekleme alanı 1		Sampling site 2 Örnekleme alanı 2		Sampling site 3 Örnekleme alanı 3		Sampling site 4 Örnekleme alanı 4	
	<i>H. fuscipennis</i> (ppm)	<i>H. fuscipennis</i> (ppm)	<i>A. limbata</i> (ppm)	<i>H. fuscipennis</i> (ppm)	<i>H. caraboides</i> (ppm)	<i>H. fuscipennis</i> (ppm)		
Ti	125 ± 6.24	0.29 ± 0.9	0.30 ± 0.07	0.40 ± 0.26	0.51 ± 0.04	352.3 ± 4.5		
V	19.3 ± 3.54	0 ± 0	0 ± 0	0 ± 0	0.32 ± 0.05	61.6 ± 5.03		
Cr	19.6 ± 2.51	152 ± 2.51	144.3 ± 6.8	249.0 ± 4.58	0.14 ± 0.03	12.6 ± 1.52		
Mn	6.73 ± 1.16	0 ± 0	0 ± 0	80.3 ± 13.01	0.13 ± 0.03	0 ± 0		
Fe	2.90 ± 0.36	89.6 ± 4.16	29.1 ± 2.56	55.8 ± 9.04	0.78 ± 0.05	7.46 ± 0.35		
Co	2.30 ± 0.20	8.23 ± 0.25	6.26 ± 1.25	0 ± 0	439.6 ± 17.24	0 ± 0		
Ni	0.36 ± 0.25	3.30 ± 0.20	3.30 ± 0.20	3.63 ± 1.40	260.0 ± 17.69	0.26 ± 0.15		
Cu	0.43 ± 0.23	0 ± 0	0 ± 0	2.70 ± 0.20	168.6 ± 14.6	0.13 ± 0.05		
Zn	0.40 ± 0.10	0.46 ± 0.40	1.40 ± 0.36	1.26 ± 0.20	369.3 ± 18.0	0.11 ± 0.01		
As	0 ± 0	1.46 ± 0.25	0.43 ± 0.35	2.46 ± 0.45	91.6 ± 9.07	0.30 ± 0.20		
Se	0.31 ± 0.18	1.46 ± 0.15	1.93 ± 0.20	2.96 ± 0.15	0 ± 0	0.40 ± 0.30		
Br	0.35 ± 0.21	4.0 ± 0.10	7.83 ± 0.58	11.2 ± 1.55	89.0 ± 5.56	0.23 ± 0.04		
Sr	327 ± 3.51	0.22 ± 0.02	0.53 ± 0.37	0.25 ± 0.04	51.3 ± 4.50	0.01 ± 0.02		
Pb	608 ± 7.02	55.0 ± 2.00	57.4 ± 5.50	84.8 ± 10.37	13.0 ± 2.00	2.76 ± 0.49		

Table 4. Average heavy element concentrations in water and sediment samples (ppm).

Çizelge 4. Su ve tortu örneklerindeki ortalama ağır element konsantrasyonları (ppm).

Heavy Element Ağır Element	Average heavy element concentrations in water and sediment samples (ppm - Mean ± SD) Su ve tortu örneklerindeki ortalama ağır element konsantrasyonları (ppm - Mean ± SD)							
	Sampling site 1 Örnekleme alanı 1		Sampling site 2 Örnekleme alanı 2		Sampling site 3 Örnekleme alanı 3		Sampling site 4 Örnekleme alanı 4	
	Water Su	Sediment Tortu	Water Su	Sediment Tortu	Water Su	Sediment Tortu	Water Su	Sediment Tortu
	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)
Ti	389.3 ± 59.7	1.80 ± 0.20	330 ± 8.2	1.50 ± 0.20	255.0 ± 8.0	0.43 ± 0.31	203.0 ± 3.6	69.8 ± 1.60
V	84.6 ± 2.51	0.16 ± 0.11	63.3 ± 2.7	0 ± 0	0 ± 0	0 ± 0	34.3 ± 4.50	0 ± 0
Cr	19.3 ± 4.04	844.6 ± 11.6	15.0 ± 2.0	672 ± 7	10.9 ± 1.49	0 ± 0	0.40 ± 0.30	320.0 ± 3.00
Mn	4.53 ± 2.44	0 ± 0	4.83 ± 0.37	234 ± 8.71	3.70 ± 0.30	0 ± 0	2.76 ± 0.49	0 ± 0
Fe	2.63 ± 0.40	0.23 ± 0.15	2.50 ± 0.20	246.3 ± 3.7	1.46 ± 0.25	844.3 ± 17.1	1.46 ± 0.25	864.6 ± 12.6
Co	1.03 ± 0.25	0 ± 0	0.04 ± 0.02	8.66 ± 0.75	0.50 ± 0.26	19.16 ± 2.02	0.33 ± 0.20	19.5 ± 1.80
Ni	0.43 ± 0.30	19.3 ± 2.08	0.5 ± 0.2	15.3 ± 2.51	0.53 ± 0.25	8.43 ± 0.20	0.43 ± 0.20	7.56 ± 0.40
Cu	0.33 ± 0.32	9.06 ± 0.40	0.23 ± 0.15	7.56 ± 0.40	0.233 ± 0.23	3.36 ± 0.35	0.20 ± 0.17	3.66 ± 0.85
Zn	0 ± 0	5.13 ± 0.32	0.13 ± 0.05	4.33 ± 0.35	0.33 ± 0.25	2.20 ± 0.43	0 ± 0	1.46 ± 0.55
As	1.10 ± 1.01	10.0 ± 2.0	0.33 ± .15	8.00 ± 0.10	0.33 ± 0.32	3.73 ± 0.25	0 ± 0	3.93 ± 0.90
Se	0 ± 0	10.56 ± 0.60	0 ± 0	7.46 ± 0.50	0 ± 0	4.16 ± 0.20	0 ± 0	3.20 ± 0.75
Br	0.20 ± 0.10	5.46 ± 0.15	0.14 ± 0.03	4.23 ± 0.25	0.68 ± 0.45	2.76 ± 0.30	0.05 ± 0.04	3.26 ± 0.25
Sr	0 ± 0	13.0 ± 2.64	0 ± 0	9.73 ± 0.25	0 ± 0	5.03 ± 0.25	0 ± 0	7.20 ± 0.26
Pb	2.16 ± 2.20	11.06 ± 1.0	2.36 ± 0.35	2.53 ± 0.72	1.26 ± 0.15	1.13 ± 0.35	0.60 ± 0.36	1.40 ± 0.40

Table 5. Results for the levels in water were compared with national regulation (ppm).

Çizelge 5. Sudaki ağır element sonuçlarının ulusal yönetmelikle karşılaştırılması (ppm).

Heavy Elements Ağır Element	Sampling site 1 Örnekleme alanı 1	Sampling site 2 Örnekleme alanı 2	Sampling site 3 Örnekleme alanı 3	Sampling site 4 Örnekleme alanı 4	TWPCR (µg/L)			
					I	II	III	IV
Pb	2.16 ± 2.20	2.36 ± 0.35	1.26 ± 0.15	0.60 ± 0.36	10	20	50	>50
As	1.10 ± 1.01	0.33 ± 0.15	0.33 ± 0.32	0 ± 0	20	50	100	>100
Cu	0.33 ± 0.32	0.23 ± 0.15	0.23 ± 0.21	0.20 ± 0.17	20	50	200	>200
Cr	19.3 ± 4.04	15.0 ± 2.0	10.9 ± 1.49	0.40 ± 0.30	20	50	200	>200
Co	1.03 ± 0.25	0.04 ± 0.02	0.50 ± 0.26	0.33 ± 0.20	10	20	200	>200
Ni	0.43 ± 0.30	0.5 ± 0.2	0.53 ± 0.25	0.43 ± 0.20	20	50	200	>200
Zn	0 ± 0	0.13 ± 0.05	0.33 ± 0.25	0 ± 0	200	500	2000	>2000
Fe	2.63 ± 0.40	2.50 ± 0.20	1.46 ± 0.25	1.46 ± 0.25	300	1000	5000	>5000
Mn	4.53 ± 2.44	4.83 ± 0.37	3.70 ± 0.30	2.76 ± 0.49	100	500	3000	>3000
Se	0 ± 0	0 ± 0	0 ± 0	0 ± 0	10	10	20	>20
Ba	0.0071	0.0053	0.0004	0.0031	1000	2000	2000	>2000

According to TWPCR (Anonymous, 2004), sampling waters have IV water quality and III water quality in regard to Co, Cr, Pb, Ni, As and Mn; I quality water in regard to Ba and Zn; II quality water in regard to Fe. Sampling Site 3 and 4 have II quality water in regard to Cu, however, Sampling Site 1 and 2 have III water quality. Sampling Site 1 and 2 have III and IV water quality in regard to Co, As, Cu, Pb, Cr, Mn and Ni, this can be due to cement factory ash emission and Aşkale-Trabzon highway traffic pollution. Sampling Site 3 has III and IV water quality in regard to Co, Pb, Cr, As, Ni and Mn this can be due to Tortum-Erzurum highway and also domestic wastes. These results showed that heavy element residues like Cr, Pb, Ni, Co and Mn in the Sampling Site 4 reveals the extent of the spread in Erzurum. Thus human activity is limited in this sampling site. These results belong to IV water quality and III water quality. However, Sampling Site 4 has I quality water in regard to Ba, which is below the detection limit and in regard to Fe and Cu it has II water quality. Ba has I quality water in all of the studied locations, but this metal was investigated below the detection limits. Like Ba there were some measured elements such as Ce, Pd, In, Sn, Nb, Pm and Mo below the detection limit of EDXRF spectrometry.

DISCUSSION

For healthy environment, pollution must be controlled. Contaminants residues in living and

non-living environments reflect environmental quality. Because aquatic beetles are in equilibrium with water body, thus they can reflect potential pollution level of water body in biologic side. The data presented in this paper provide information on heavy elements accumulated by hydrophilids. These elements are predominantly present in almost all sampling sites and have profound influence not only on the water quality but also on the life of the aquatic insects. When hydrophilid absorbs these elements from its field/habitat, they can bio-accumulate. Moreover, it can be transported from insects throughout the food chain. In the present study it is concluded that, high level of elements in an environment lead to high accumulation in insect samples. Therefore, the insects have ability to accumulate certain elements and they can be used in environmental monitoring studies. In this study among the hydrophilid species *H. fuscipes* fits for the biomonitor criteria such as; represent in large numbers all over the studying area, cosmopolitan, easily identifiable, larvae and adults are aquatic or semi-aquatic and its life cycle is known, have relatively long life cycles. *H. caraboides* and *A. limbata* also accumulate the elements in high concentration, but they found only in one sampling site.

In this study, the water quality and inorganic pollution of the sites were evaluated by using hydrophilid beetles. For more information on the assessment of health of environment, long-term biomonitoring must be regularly done. Because

long-term biomonitoring may provide insight into how human affects water quality, community and ecosystem structure. These aforementioned aquatic insects are possible biomonitor organisms that can be used as a useful tool for monitoring element contaminations studies.

ACKNOWLEDGEMENT

This study is a part of 1st author's Ph. D. Thesis which titled as "Erzurum ve Çevresindeki Sulak

Alanlarda Yaşayan Bazı Sucul Böcek (Hydrophilidae) Türlerindeki Ağır Element Birikimlerinin XRF Analiz Yöntemi ile Araştırılması".

The authors gratefully acknowledge the financial support of Ataturk University Scientific Research Project SRP-2012/162. We also thank to Dr. Buğrahan EMSEN in Department of Biology at the University of Karamanoğlu Mehmet Bey for statistical analysis of samples.

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