INVESTIGATION OF HEAVY METALS CONCENTRATIONS IN *Ulva rigida* SAMPLES OF MERSIN COASTAL ZONE

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ABSTRACT

The area of our country where the Mediterranean is located, Mersin Bay is exposed to pollution allowing for increased population, industrial activities, agricultural activities and harbor traffic.As a result of these activities, pollutants reaching the marine environment accumulate in the aquatic ecosystem and reach high concentrations. In the marine ecosystem, the spread of pollutants, in particular, passes into the food chain and thus the food web. Macro algal species which are distributed in the region and reproduced naturally are used as suitable indicator organisms in the environmental pollution of the environment by trapping the pollutants in the water bodies.

In this study, heavy metal accumulation levels such as iron, cadmium, zinc, lead and copper were investigated in the specimens belonging to *Ulva rigida* species collected in four different stations determined in the coastal area of the Mersin Gulf. For this purpose, Fe, Cu, Pb, Zn and Cd levels were measured spectrophotometrically (ICP-MS) in macroalgae samples.It was determined that Fe> Zn> Cu> Pb> Cd with the order of heavy metal accumulation as a result of the studies made on *Ulva rigida* species.

Keywords: *Heavy metals, Macroalgae, Bioaccumulation, Mersin Gulf, Ulva rigida*

1. INTRODUCTION

Increasing pollution load with the increase of industrial activities; it affects nature and people negatively. Waste materials produced in the realization of vital activities and containing different contents which are later released after consumption are left uncontrolled to the environment and as a result the environment causes pollution of living areas (Sunlu, 1994). The water environment that makes up the majority of the world is the receiving zone for wastewater and many pollutants (Yarsan vd., 2000; Kayhan, 2006).

Metals are naturally exposed to aquatic environments by erosion of rocks, filtration from soil, and volcanic activity. At the same time, mining activities, fossil fuel use, maritime activities, urban and industrial activities reach the aquatic environment with the release of wastes as a result (Lobban ve Harrison, 1997; Kennish,1998).

Macroalgs, which can easily absorb metals dissolved in the sea, accumulate these metals in their structures and thus the indicator is evaluated as alive (Fytianos vd., 1999; Mohamed ve Khaled, 2005). Macroalgae such as *Fucus, Enteromorpha, Laminaria* and *Ulva* from indicator species are often used in the determination of heavy metal concentrations in marine environments (Fytianos vd., 1999). The species belonging to the genus Ulva are known to be a good indicator because they can easily collect and accumulate most metals (Haritonidis ve Malea, 1994).

Ulva spp. like other macroalga species, is intensely observed in the marine areas of our country and is a member of the Chlorophyceae with high biomass. Ulva spp., also known as marine marlin, continues its vitality in areas where pollution is intense in marine areas (Topcuoğlu vd., 2003).

In this study, we tried to determine the seasonal changes of some of the macroalga species distributed in Mersin Bay by analyzing heavy metal contents of *Ulva rigida*. Main sources of contamination of the Mersin Gulf, which has an important place in the Mediterranean, are thought to be caused by domestic wastes, pesticides and synthetic fertilizers that can be used in agricultural activities, industrial establishments and harbor activities. The presence of major industrial establishments such as glass, soda and oil refineries as well as fertilizer, chromium compounds and plastics production operating in the region can be shown as the reasons for the pollution (Kalay vd., 2004).

1.2. Heavy Metal Studies on Makroalga

Olgunoglu (2008) has carried out a study on accumulation of heavy metals such as Pb, Zn, Fe, Cd and Cu in Macroalg samples collected from the Eastern Mediterranean on the shores of the Iskenderun Gulf at three different stations.Gerçekleştirilen bu araştırmada makroalg örnekleri bünyesinde biriken ağır metallerin derişim sırasının Fe > Zn > Pb >Cu > Cd olduğu belirtilmiştir.

Alp et al. (2012), a study has been carried out to determine the accumulation levels of heavy metals in macroalg samples detected in four different stations used as beaches in summer in Mersin province. In this study, they have investigated heavy metal concentrations(*Ulva* sp. ve*Enteromorpha* sp.) of Mn, Al, Cr, Ni, Fe, Cu, Zn, Cd and Pb. As a result of the study; Ulva sp. Heavy metal accumulation levels were reported to be Fe> Al> Mn> Ni> Zn> Cr> Cu> Pb> Cd respectively.

Özden and Tunçer (2015) have been reported that *Ulva rigida* is consumed in the different regions of the world as being beneficial to human health, and also a good bioindicator in the measurement of metal pollution.

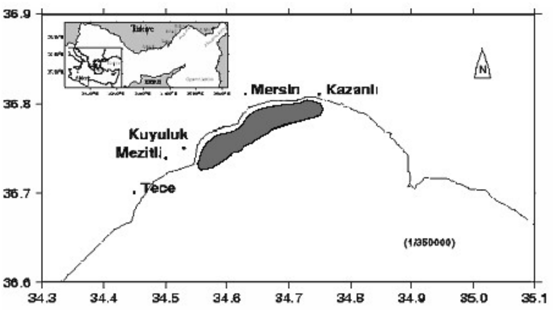
In this study heavy metal levels were investigated in Ulva rigida specimens taken from two different stations determined between 2015-2016 on the coast of Mersin Province.

2. MATERIALS AND METHODS

The study was carried out in four different locations determined from the Mediterranean Region of Karaduvar Fisherman's Bar in Mersin Province and the Viranşehir Coastal Offshore Facilities in Mezitli District between July 2015 and July 2016. The information on the stations where fishery and industrial activities are thought to be concentrated in heavy metal pollution and stations that are considered to be less concentrated are given below.

Tablo 2.1. Coordinates of the stations collected

İstasyon Adı	Koordinat (N)	Koordinat (E)
Karaduvar Balıkçı Barınağı	36°48'18.76"N	34°41'36.68"E
Çamlıbel Yat Limanı	36°47'20.03"N	34°37'46.44"E
Fenerbahçe Meydanı	36°46'21.53"N	34°34'23.00"E
Viranşehir Taşkıran Tesisi	36°44'18.47"N	34°32'28.02"E



Şekil. 2.1. Mersin Gulf Sampling Stations

Macroalg samples were checked and collected on a monthly basis during the study to ensure that the heavy metal accumulation can be determined in a timely manner.Specimens collected from the designated stations were labeled with polyethylene bags and transported to the laboratory for genetic diagnosis.

Macroalg specimens carried on the laboratory were washed with pure water and free from foreign bodies that could affect the results of sand and other work on their surfaces, leaving them to natural dryness. Then the sample was dried at 70ºC for 2 hours. After the drying process is completed, it is pulverized with the aid of a disintegrator. The blister was then placed in labeled polyethylene bags and stored in the refrigerator until analysis.

After pretreatment, 1 ml of H_2O_2 and 4 ml of water (1 mL of HCl and 3 mL of HNO_3) were added to the 0.5 g samples from *Ulva rigida* samples. The samples taken into the tubes belonging to the microwave device have been solubilized. The samples were treated with acid and lysed. It is dissolved in a microwave device so that it can be analyzed after shredding. The disassembly with the microwave device provides a significant advantage as it takes place in as little as 20 minutes. The solubilized samples were diluted to 50 mL with ultra distilled water. After labeling, the ICP-MS was kept under conditions suitable for heavy metal reading.

Macroalgae samples, which are ready to be analyzed, have been assigned to heavy metal levels after moving to Mersin University Advanced Technology Education, Research and Application Center.

3. RESULTS

In this study, Macroalg samples were collected from 4 different points determined between Karaduvar Fisherman's Shelter and Viranşehir Taşkıran Facility.

Six different macroalga species were found in four different strains: *Enteromorpha flexuosa, Hypnea musciformis, Scytosiphon lomentaria, Taonia atomaria, Ulva linza* and *Ulva rigida*. Sample collection studies continued throughout the year, but macroalga species were not found between August 2015 and January 2016 (Tablo 3.1). After collecting the samples, it was determined that the most collected macroalga species in the laboratory were *Ulva rigida*.

Tablo3.1. Macroalg detection status at stations

		Stations			
Number	Date	Karaduvar	Çamlıbel	Fenerbahçe Meydanı	Taşkıran
1	04.2015	-	UR, HM, SL	-	UL, HM
2	05.2015	-	-	-	-
3	06.2015	-	-	-	-
4	07.2015	UL	UR	UR	-
5	08.2015	-	-	-	-
6	09.2015	-	-	-	-
7	10.2015	-	-	-	-
8	11.2015	-	-	-	-
9	01.2016	-	-	-	-
10	01.2016	-	-	-	-
11	02.2016	-	-	EF	EF, UL
12	03.2016	UL	UR	UR, HM	-
13	04.2016	UL	UR, UL, HM	UR. HM	UL, EF
14	05.2016	-	-	-	UL, EF

EF (*Enteromorpha flexuosa*), **HM** (*Hypnea musciformis*), **SL** (*Scytosiphon lomentaria*), **TA** (*Taonia atomaria*), **UL** (*Ulva linza*), **UR** (*Ulva rigida*), (-) Not detected.

Heavy metal accumulation such as Fe, Pb, Cu, Zn and Cd in *Ulva rigida* species have been investigated. *Ulva rigida* species were observed only at Çamlıbel and Fenerbahçe Square stations. For *Ulva rigida* species samples could be collected at Çamlıbel station in March

2015, but macroalg could not be detected at Fenerbahçe Square station.

Tablo 3.2. Heavy metal accumulations of Ulva rigida at Çamlıbel and Fenerbahçe Square Stations (µg/g KA)

Metal (ppm)	Stations	Sample Collection Dates			
		2015.4	2015.7	2016.3	2016.4
Fe	Çamlıbel	657,30	579,54	2842,14	535,39
	Fenerbahçe Meydanı	-	1549,38	400,26	305,87
Pb	Çamlıbel	13,39	6,73	11,44	5,98
	Fenerbahçe Meydanı	-	6,91	10,96	2,72
Cu	Çamlıbel	7,01	5,76	18,04	1,81
	Fenerbahçe Meydanı	-	2,77	8,36	5,47
Zn	Çamlıbel	100,57	114,84	8,45	3,74
	Fenerbahçe Meydanı	-	80,21	21,67	53,35
Cd	Çamlıbel	1,77	1,63	1,68	0,96
	Fenerbahçe Meydanı	-	1,75	1,93	0,23

According to research findings, *Ulva rigida* is the highest place of Fe accumulation in Çamlıbel station. The highest metal accumulation [1549,38 µg / g dry matter (µg / g KA)] at Fenerbahçe Square station in July 2015 and [2842,14 µg / g dry matter (µg / g KA)] at the Çamlibel station in March 2016. The lowest Fe accumulation values for both stations were measured in April 2016 (Şekil 3.2). It has been determined that the accumulation of Fe in *Ulva rigida* samples collected is 1.83 times higher than the Fenerbahçe Square station at Çamlıbel station.

The results show that the accumulation of Pb in the *Ulva rigida* species is higher than the Fenerbahçe Square station at Çamlıbel station. The highest Pb accumulation occurred in the samples collected in April 2015.

Pb metal accumulation was highest at Çamlıbel station in April 2015 (13,39 µg / g KA) and at Fenerbahçe Square station in April 2016 (10,96 µg / g KA).It is determined that the accumulation of Pb in the samples collected at Çamlıbel station is 1.22 times more than the samples collected at Fenerbahçe Square station.

When the results of Cu accumulation in the *Ulva rigida* type are examined, it is determined that the most intensive history of accumulation is March 2016 in both stations. It was estimated that the highest accumulation occurred at Çamlıbel station (18.04 µg / g KA) was 2.15 times more than the Fenerbahçe Square station (8.36 µg / g KA).However, the lowest savings were found to have taken place in April 2016.

When the Zn data of *Ulva rigida* species are examined, it is determined that Çamlıbel station is the station where the heavy metal accumulation is the most intense. It is found that the accumulation (mean 107.71 µg / g KA) in 2015 is about 17.67 times higher than the year 2016 (mean 6.10 µg / g KA).It is determined that the metal accumulation in the samples taken from Fenerbahçe Square station is below the value determined in 2015 and the highest accumulation is in July 2015 (80,21 µg / g KA).

When the accumulation of Cd in *Ulva rigida* is examined, it is seen that Fenerbahçe Square is the station where heavy metal accumulation is most concentrated.It has been found that the accumulation of Cd in *Ulva rigida* samples collected at Çamlıbel and Fenerbahçe Square stations showed similar values.

It has been found that the accumulation of heavy metals in Ulva rigida species in the years between 2015 and 2016 is followed by Fe> Zn> Pb> Cu> Cd in Mersin Çamlıbel and Fenerbahçe Square stations.

4.DISCUSSION

Macroalg species are known to accumulate in heavy metals bodies and can be used for pollution detection due to these reasons. It has been reported that the formation of some metals (Fe, Zn, Pb) in macroalgae at high level is primarily due to the high accumulation of metal in the working area and secondly the high absorption capacity of these metals in algae (Haritonidis ve Malea, 1999).

Macroalg species were collected in 4 different regions of the Mersin Gulf. It has been determined that the collected species belong to 6 different macroalga species.The accumulation of Fe, Cu, Zn, Pb and Cd in heavy metal varieties of *Ulva rigida* of Ulva genus(Özden ve Tunçer, 2015; Haritonidis ve Malea, 1994) which is reported to be a good bioindicator in the detection of metal pollution has been investigated.Although the study was planned for four different stations, *Ulva rigida* was only found at Çamlıbel and Fenerbahçe Square stations.

It is well known that the Mersin Bay is an area where oil activities, such as oil filling, fertilizer production, production of chrome compounds, glass and soda industry and oil refinery, as well as fisheries and intensive ship traffic are frequent.It has been determined that the accumulation values of metals such as Fe, Cu, Pb and Cd in industrial wastes are high in Ulva rigida which is developed in the study area due to their high content.

As a result of the direct or indirect interactions with the pollutant sources in the workplace, the average accumulation values of heavy metals such as Fe, Zn, Pb, Cu and Cd for Ulva rigida species were determined.These values were 858,72 µg / g KA for Fe; 47.85 µg / g KA for Zn; 7.27 µg / g KA for Pb; For Cu 6.15 µg / g KA and for Cd 1.24 µg / g KA.It was determined that the order of Fe> Zn> Pb> Cu> Cd according to the determined average accumulation amounts.

A similar result has been reported in a study conducted in the area *Ulva rigida*, where the mining activities are concentrated in the Chilean northern center.In the report; for average accumulation, the order is Fe> Zn> Cu> Cd> Pb (Olivares vd.,2016). When compared to the heavy metal sequences in *Ulva rigida* species, it was determined that the order except Pb metal is the same.It is thought that the cause of this difference may be different depending on the pollutants in sampling areas, seasonal variations and algal species (Fytianos vd., 1999; Kalesh ve Nair, 2005; Türkmen vd., 2005). When the accumulations between metals are compared, it is stated that although the studied fields are different, the most concentrated accumulation is Fe and accumulates more than the other metals.

Tablo 4.4. Sequence of heavy metal accumulation in *Ulva rigida* species

Sampling		Watched Metals						Reference
Date	Station	Ranking	Fe	Zn	Pb	Cu	Cd	
2011	Şili Kuzey Merkezi	Fe>Zn>Cu >Cd>Pb	1346,14	15,24	0,24	10,31	0,87	Olivares vd. 2016
2015-2016	Mersin Körfezi	Fe>Zn>Cu >Pb>Cd	858,72	47,85	7,27	6,15	1,24	Bu çalışmada

In the study conducted in the Thermaikos Bay (Greece), the mean dry weight of Ulva rigida was 57.3 µg / g Zn; 97,2 µg / g Fe; 2.2 µg / g Cu; 14.7 µg / g Pb; 1.0 µg / g Cd, and reported that the order of heavy metal deposits was Fe> Zn> Pb> Cu> Cd. It was determined that the heavy metal results found in the specimens from the samples of 1982 were low between 1.02 and 12 times the metal accumulation in our study (Haritonidis ve Malea, 1999). It has been determined that the order of Fe> Zn> Pb> Cu> Cd is in spite of differences in the amounts of accumulation.

The accumulation of heavy metals such as Fe, Zn, Cu and Pb in Ulva rigida samples collected from Özden and Tunçer (2015) Çanakkale Bosphorus were investigated.It is known that shipyard construction, maintenance and repair are performed in this area as well as intensive harbor activities.In the work of Özden and Tunçer (2015), a dry weight of 10,87 µg / g Fe; 6.53 µg / g Zn; 0.60 µg / g Cu; 0.15 µg / g Pb and we have found that Fe> Zn> Pb> Cu in the order of heavy metal deposits.

Alp et al. (2012) carried out a study on Cu, Al, Ni, Cr, Zn, Pb, Mn, Cd and Fe in order to determine the heavy metal levels of the macroscopic green algae they have accumulated in their bodies in Mersin province. As a result of these studies; In the case of Ulva species, they reported metal accumulation as Fe> Al> Mn> Ni> Zn> Cr> Cu> Pb> Cd. It is seen that the sequence of metal accumulation is similar in this study made in the same region. However, the values in the study conducted between 2009 and 2010 were 209,8µg / g KA for iron; 4.6 µg / g KA for zinc; 1.0 µg / g KA for copper; 0.8 µg / g KA for lead and 0.03 µg / g KA for cadmium have been reported. Although the sorting is the same, the metal accumulation is 3-5 for iron; 8-12 for zinc; 4-8 for copper, 6-12 for lead and 33-50 times for cadmium.

When the values of heavy metal accumulation obtained by Alp vd from 2009-2010 are examined, it can be understood that pollution in the marine environment is increasing.

In this study, it was determined that heavy metal accumulation as a result of increasing industrial activities, marine traffic, harbor and fishing activities in the Mersin Gulf has been significantly increased.

5. CONCLUSION

The heavy metal deposits belonging to the Ulva rigida species, which are distributed in the Gulf of Mersin located on the Mediterranean coast of our country, were also investigated.

As a result of this study, it is also important to determine the effects of water pollution and climatic changes.At the same time, it was observed that the heavy metal values of the Ulva rigida species, which were collected from 6 different species collected in Mersin Bay, were Fe> Zn> Pb> Cu> Cd.

Especially because of heavy metal pollution accumulated in the receiving environment such as soil and water as a result of industrial and agricultural activities, natural life is put at great risk.Depending on the concentration given to the environment and the sensitivity of their bodies, living things in the receiving environment will be adversely affected.Sudden deaths can be encountered, as is the case in many instances, or there may be major problems in the form of passing through other foodstuffs (like human beings) through the food chain.For this reason, it is necessary to prevent the discharges of all factors, especially the industrial organizations, which may cause the receiving environment to increase the heavy metal concentration.

Tablo 4.5.*Ulva rigida* ve diğer *Ulva* spp. türlerinde ağır metal birikim seviyeleri(µg/g KA)

Sampling		**Metals Watched**						**References**
Date	**Location**	**Ranking**	**Fe**	**Zn**	**Pb**	**Cu**	**Cd**	
1982	Thermaikos Körfezi,	Fe>Zn>Pb>Cu>Cd	97,2	57,3	14,7	2,2	1	Haritonidis ve Malea, 1999.
2009-2010	Mersin Körfezi	Fe>Zn>Pb>Cu>Cd	209,78	4,59	0,81	1,02	0,03	Alp vd., 2012.
2009-2013	Çanakkale Boğazı	Fe>Zn>Cu>Pb	10,87	6,53	0,15	0,6	-	Özden ve Tunçer, 2015.
2011	Şili Kuzey Merkezi	Fe>Zn>Cu>Cd>Pb	1346,14	15,24	0,24	10,31	0,87	Olivares vd., 2016.
2015-2016	**Çamlıbel**	**Fe>Zn>Pb>Cu>Cd**	**1153,57**	**56,9**	**9,39**	**8,15**	**1,51**	**This study**
2015-2016	**Fenerbahçe Meydanı**		**563,88**	**38,81**	**5,15**	**4,15**	**0,98**	**This study**

We think that researches about heavy metal pollution in the Mersin coastal region, which is the receiving environment, should be carried out continuously and the necessary measures should be taken according to the results obtained.

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