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The Assessment of Heavy Metal Contamination in the Waters of the Nilufer Stream in Bursa

Gokhan Ekrem USTUN

Uludag University, Faculty of Engineering and Architecture, Department of Environmental Engineering 16059 Gorukle, Bursa-TURKIYE

*Corresponding author: gokhaneu@uludag.edu.tr

Abstract
This study was conducted from 2002 through 2007 with no studies in 2004, to evaluate 8 metal contaminants (As(total), Cd, Cr (total), Cu, Mn, Ni, Pb and Zn) of the Nilufer Stream, where intensive industrialization, urbanization, and agricultural activities take place. The results were then compared with national and international water quality guidelines. The effect of the wastewater treatment facilities that were established during the measurement period on water quality was also taken into account. It was determined that the Nilüfer Stream water quality along the basin has declined rapidly year to year. Intensive wastewater discharge causes a waste dominating flow in the stream and has caused the water quality to steadily decline over time. The total chromium (Cr) and lead (Pb) levels in the basin outlet of the Nilufer Stream water were evaluated as "high polluted water" according to the national standards in the classification of the quality of the surface water. The mean metal concentrations in the Nilüfer stream water are generally higher than the international guidelines. As a result, metal contaminant pollution of the Nilüfer Stream was found to be connected to human activities in its catchments.
Keywords: Anthropogenic activities, guidelines, metal contaminants, Nilufer stream, water contamination.

Nilüfer Çayı'nda Ağır Metal Kirliliğinin Değerlendirilmesi

Özet
Bu çalışma kapsamında havzasında yoğun sanayileşme, kentleşme ve tarımsal faaliyetlerin yer aldığı Nilüfer çayında 8 metal kirleticinin (As(toplam), Cd, Cr(toplam), Cu, Mn, Ni, Pb, Zn) içeriği 2002 ve 2007 yılları arasında (2004 yılında çalışma yapılmamıştır) incelenmiştir. Elde edilen sonuçlar ulusal ve uluslararası su kalite yönergeleriyle kıyaslanmıştır. Ölçüm dönemi boyunca kurulan atıksu arıtma tesislerinin su kalitesine etkisi de dikkate alınmıştır. Nilüfer çayı su kalitesinin havza boyunca yıldan yıla kötüleştiği tespit edilmiştir. Yoğun atıksu deşarjı çayda atıksu ağırlıklı bir akış oluşturmuş ve su kalitesi zamanla kötüleşmiştir. Ulusal yüzeysel su kalite sınıflandırmasına göre Nilüfer Çayı havzası çıkış noktasında toplam krom (TCr) ve kurşun (Pb) seviyeleri açısından "çok kirlenmiş su" sınıfına girmektedir. Nilüfer çayı ortalama metal konsantrasyonları genellikle uluslararası standartlardan yüksektir. Sonuç olarak havzadaki insan aktiviteleri ile bağlantılı olarak Nilüfer Çayı'nda metal kirliliği tespit edilmiştir.
Anahtar Kelimeler: Antropojenik aktiviteler, metal kirleticiler, Nilüfer Çayı, su kirliliği, yönetmelikler.

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INTRODUCTION

Bursa is one of the most important industrialized and urbanized cities in Turkey and is experiencing rapid industrial development. Industrial and agricultural activities, along with a rapidly increasing population and unplanned urbanization processes, have challenged the ecological balance in Bursa. The Nilüfer Stream is the major riverine system in Bursa. The Nilüfer Stream basin covers 1540 km^2. More than 53.8% of the basin is used for agricultural purposes, with 33.9% of the area covered by forests, 5.0% covered by meadows, and 6.4% by settlements (Karaer and Küçükballı 2006). Not only does it supply drinking water to the Bursa inhabitants, but it also supplies irrigation water for the agricultural sites around the city through its tributaries (Fig. 1). The Nilüfer Stream and its tributaries are being polluted by organic and inorganic pollutants from the industrial and domestic wastes caused by the industrialization and urbanization activities in Bursa (Güleryüz et al. 2008, Kocaer and Baskaya 2004). The water quality in most parts of the stream do not meet the "National Inland Water Resources Quality Standards" (WQS) (Anonymous 2004).

Although several reports on the assessment of water quality based on physico-chemical and biological parameters have been published by several authors (Yılmaz et al. 1998, Karaer and Küçükballı 2006, Üstün 2009), very little information is available about the status of the metal

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contaminants in the Nilüfer Stream.

With this information, an investigation was initiated, with the primary objective to examine the present status of various metal contaminants in the Nilüfer Stream water samples were collected from six stations in 2007 and from one station between 2002 though 2007 with no collections in 2004.

MATERIAL AND METHODS

Study Area

The Nilüfer basin is located in the northwestern Anatolian Region. It includes the Nilüfer Stream (168 km) and the industrialized city of Bursa which lies at the intersection of 40°11' N latitude and 29°04' E longitude. The Nilüfer Stream passes through the city and supplies drinking water via the Dogancı dam which was built upstream of the city (Fig. 1). The Nilufer Stream's 2007 average flow rate was 16.77 m^3 s^{-1} (Anonymous 2007a). The main objective of the two Wastewater Treatment Plants (WTPs) that were established in recent years was to meet the WQS by decreasing the untreated domestic and industrial wastewater. The population of the basin in the east WTP area is 1.076.538 and about 61% of the population (656,713 people) are connected to the sewage system. In the west WTP area, the population is 215,196 and 60% of the population is connected to the sewage system. Therefore, a considerable amount of domestic waste water is not connected to the WTPs and is discharged directly into the Nilüfer Stream and its tributaries. Most of the existing industrial plants are located along the Nilüfer Stream's banks and its tributaries. The stream is also a convenient place to discharge industrial plant wastewaters (Anonymous 2007b). The agricultural activities performed in the basin (with the extensive use of fertilizers and pesticides) are non-point pollution sources that affect water quality (Aksoy and Ozsoy 2002).

Sampling Stations

The sampling stations are shown in Fig 1. The water quality problems experienced by the stream can be attributed to the direct discharges of domestic and industrial wastewaters, especially in the summer periods when the stream is mostly dominated by wastewater discharges. The largest WTPs in the basin are shown in Figure 1 and the characteristics of the WTPs are given in Table 1. Sampling station one is lasted in a region where there are many textile dying facilities. Whereas sampling station two, is located in an area of metal plating industries, leather industries, and textile dying facilities. The eastern WTP is in the third sampling station with the Demirtas Organized Industrial District (DOID) WTP as the fourth sampling station. In the fifth sampling station, there are industrial areas and the western WTP is where the solid waste treatment of the city's waste water is performed (Ustun and Akal Solmaz 2007). Finally, the sixth sampling station contains plating and leather facilities and is the main stream into which all of the waters from the basin are collected.

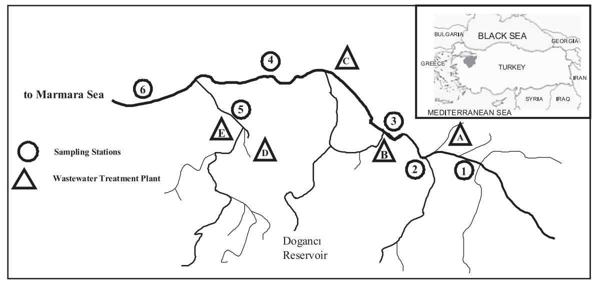

Fig 1. Sampling stations and WTPs in the catchment area of Nilüfer Stream
WTPs: A: Yesil Cevre, B: East WTP, C: Demirtas OID, D: Bursa OID, E: West WTP.

Water samples were collected from mid stream at a depth of 15-20 cm in 1000-mL polyethylene bottles, which had previously been cleaned by soaking in 10% nitric acid and rinsed with distilled water. At the sampling site, the bottles were rinsed twice with the water to be sampled prior to filling. Grab samples of water were collected in triplicate and mixed to get a composite sample for each sampling station. The water samples were acidified on site to a pH less than 2 with 5 mL of analytical grade concentrated HNO_3. After collection the samples were placed in coolers with ice bags while being transported to the laboratory and kept at about 4°C until being analyzed (Fianko et al 2007). Samples were collected between 2002 through 2007 (for except 2004 when no samples were collected) and composite samples of two hours were taken in dry weather from the six measuring stations in the months representing each season (March, June, September, and December).

Laboratory Analyses

Metal samples were prepared with a preliminary digesting process via a CEM MARS-5 model microwave instrument. The sample preparation procedure was as follows: a 40-mL sample was placed into the cell and then 6 mL of HNO_3 (65% analytical grade) and 4 mL of HCl (37% analytical

Table 1. The properties of WTPs in the Nilufer basin

WTPs- Construction Year	Wastewater Type ⋆	Wastewater Flow ($m^3 day^{-1}$)
Yesil Cevre-2006	I +U	55,000
East WTP-2006	U	154,777
Demirtas OID-2007	I	70000
Bursa OID-2006	I	96000
West WTP-2005	U	40,539

⋆ I: Industrial, U: Urban

grade) were added to the cell. The cells were covered and a maximum pressure of 180 psi and a temperature of 160°C was applied for 20 minutes. In the second step, the samples were allowed to cool for 10 minutes. After 30 minutes, the samples were cooled to room temperature and transferred into a 100-mL flask. The digested samples were filled with distilled water to the 100-mL mark, and used in the ICP-AES (Vista MPX, Varian) analysis.

The metal concentrations in the digested samples were analyzed using the ICP-AES. Eight metals were targeted: As, Cd, Cr, Cu, Mn, Ni, Pb, and Zn. The blanks, standard calibration solutions, and digested samples were put into tubes in an automatic sampler and the analysis was started. The standard calibration solutions employed in the analyses were prepared at concentrations of 0.05, 0.1, 0.25, 0.5, and 1 mg/L. For the sample concentrations higher than 1 mg/L, calibration solution concentrations were prepared at 1, 2, 5, and 10 mg/L. The blanks were prepared by adding concentrated 5% HNO_3 into ultra pure water that was produced by Milli-Q (Millipore Co.).

Quality controls were performed with certified liquid samples (multi-elements standard, catalogue number 900-Q30-002, lot number SC0019251, SCP Science, Lasalle, Quebec) to ensure the accuracy of the measurements. Quantification limits were: 2 μg/L for Cd, 3 μg/L for Pb, 5 μg/L for As, Cr, and Cu, 10 μg/L for Mn and Zn, and 20 μg/L for Ni. Certified liquid samples were used to check the analytical accuracy, which ranged between 1% and 10%. All reagents used were of analytical grade or better.

Statistical Analysis

A Multivariate analysis (element coefficient correlations) was used to determine the metal levels of the water samples, which were performed using the SPSS statistical package program. A probability of 0.05 or less was considered as statistically significant. Additionally variations in metal concentrations were assessed for statistical significance using a two tailed paired t test (Anderson 1987).

Table 2. Annual values (2007) of the metal contaminants for different sampling stations of the Nilüfer Stream (μg L^{-1})

Metals	Stations					
	1	2	3	4	5	6
As	17±12	201±124	62±26	56±21	49±14	81±23
Cd	6±2	14±4	11±2	9±4	10±5	7±3
Cr	15±6	48±6	49±26	319±45	181±89	511±302
Cu	25±13	53±18	34±4	50±15	68±48	69±37
Mn	35±21	103±27	110±43	177±45	285±235	274±37
Ni	14±3	37±11	20±3	51±22	114±98	73±10
Pb	76±57	130±52	92±7	108±30	119±91	119±52
Zn	32±18	248±117	187±142	245±130	1079±659	373±116

RESULTS AND DISCUSSION

Spatial Distribution of Metal Contaminants

Annual values (2007) of the metal contaminants for each station of the Nilüfer Stream are given in Table 2.

To determine the average metal concentration levels at the measuring stations for similarity, the t-test was used. Statistical results of the calculation for Cr, As, and Zn respectively, for stations 6, 2, and 5 show different levels of metal concentrations found outside the stations. This is the result of anthropogenic activities thought to arise about the stations.

The results of the metal contaminant values were evaluated according to the national WPCR (Water Pollution Control Regulation 2004), international guidelines (Anonymous (2006)), and the industrial WTPs discharge limits as seen in Table 3. According to the criteria stipulated in the WPCR, inland surface water resources were classified into four categories, each having distinct utilization purposes. For example, Class I waters were classified as high quality waters that could be used for drinking water supplies with simple disinfection. Class II waters, on the other hand, were considered to be medium quality waters that could be used for drinking water supplies only after appropriate treatment. Low quality Class III waters could be used for industrial and agricultural purposes but could not be used for drinking purposes under any conditions. Class IV waters were highly polluted and could not be used for either municipal or industrial purposes. For OIDs, the standards given in the water pollution control regulation are applied (Anonymous 2004). Anonymous (2006), for water quality criteria described; the criteria maximum concentrations (CMC) and criterion continuous concentration (CCC).

The occurrence of arsenic (As), a commonly-

Table 3. Some guidelines and industrial WTPs discharge limits (μg L^{-1})
a:(Anonymous 2004), b: (Anonymous 2006), c: Cr^{+6}

	As (total)	Cd	Cr (total)	Cu	Mn	Ni	Pb	Zn
Turkish Environmental Guidelines[a]								
Class I	20	3	-	20	100	20	10	200
Class II	50	5	20	50	500	50	20	500
Class III	100	10	50	200	3000	200	50	2000
Class IV	> 100	> 10	> 50	> 200	> 3000	> 200	> 50	> 2000
Industrial WTPs discharge limits[a]	-	100	2000	3000	-	-	2000	5000
Water quality criteria[b]								
CMC	340	2	16[c]	13	-	470	65	120
CCC	150	0.25	11[c]	9	-	52	2.5	120

occurring toxic metal in natural ecosystems can be associated with natural conditions or industrial practices (Korte and Fernando 1991). The parameters vary between 17 and 201 μg L^{-1} throughout the stream. Because of the presence of metal plating industries in the region highest values were obtanined at station two. As seen in Table 2, the water quality (Class I) at station one becomes Class III at the basin outlet.

The CMC and CCC values of Cd in the water are 2 and 0.25 μg L^{-1} respectively (Anonymous 2006). The concentration of Cd in the water samples ranged from 6 to 14 μg L^{-1}. According to the WQS, this water quality is Class III and IV.

The total chromium (Cr) concentration measurement interval was 15-511 μg L^{-1} and the highest level of Cr was found in station six due to the tannery facilities in the region. In the fourth, fifth and sixth stations, in the western part of the basin, the water quality in terms of total Cr decreased to Class IV. The normal range of chromium in water is 100 μg L^{-1} (Anonymous 2003).

Although the concentration of copper (Cu) did not change much in the Nilüfer Stream, the mean levels of Cu ranged from 25 to 69 μg L^{-1}. According to the WQS, the water quality in all the stations was Class II or III. The CMC and CCC values of Cu in the water were 13 and 9 μg L^{-1} respectively (Anonymous 2006). The Cu accumulation in the water may have been due to the plastics industry, blast-furnace, steel industry, and the application of agrochemicals of the agro-based industry. Copper is, however, characterized by so-called point sources of contamination, which are uncontrolled, active, or untended waste dumps (Gowd and Govil 2008).

Manganese (Mn) values ranged from 35 and 285 μg L^{-1} in the Nilüfer Stream. The water quality is Class I in station one, while it is Class II at other stations. Ground water used by industrial companies caused an increase in the Mn in the region.

The concentration of nickel (Ni) ranged from 14 to 114 μg L-1 at the stations. The CMC and CCC values of Ni in water are 470 and 52 μg L^{-1} respectively (Anonymous 2006). The sources of Ni in the water include contamination from municipal sewage sludge, wastewater from sewage treatment plants, and groundwater near landfill sites (Gowd and Govil 2008). The urban WTP and landfill leachate treatment plant in the region of the fifth station caused the highest Ni values in this region (114 μg L^{-1}).

The lead (Pb) level ranged from 76 and 119 μg L^{-1}, and the water quality was Class IV in all of the stations. The CMC and CCC values of Pb in water are 65 and 2.5 μg L^{-1} respectively (Anonymous 2006).

The mean levels of Zinc (Zn) ranged from 32 to 1079 μg L^{-1}. Zinc in water can present a problem in the aquatic ecosystem. Zinc in the Nilüfer Stream was Class I in the first station and the highest concentration was found at station five. In station six, the water quality was Class II. High concentrations of some metals were thought to have resulted from anthropogenic influences, particularly from nearby industries and pesticides used in agriculture that present a pollution risk (Tuna et al. 2007).

Temporal Variations of Metal Concentrations

In order to determine the change of the metal concentrations of the Nilüfer Stream over a period of time, the water quality was monitored between 2002 and 2007 (except for 2004 when no samples were taken) at station six, where all of the branches in the basin were collected. The change in the parameters from year to year is given in Table 4. When Table 4 is examined, it is observed that there has not been a significant change in the amount of As, Cd, and Mn. However, Cr, Cu, Ni, Pb, and Zn values increased with fluctuation.

Measurement of the metal levels over the years to detect the differences in metal concentrations of the statistical evaluation levels showed no difference from each other over the years. This is the result of anthropogenic activities in the region over the years, despite the established treatment plants and increase of untreated wastewater into the stream which the data reflects.

The total discharge flow rate for the largest five WTPs discharging their wastewater into the stream is 29% of the 2007 average stream flow rate. When the WTPs with smaller capacities and wastewater discharges without treatment are added, a wastewater dominant flow occurs in the stream, especially in the summer season. In developing countries, the rapid increase of the population and industry can increase the loads of wastewater treatment plants and can reduce surface water quality (Kamal et al. 1999, Tsagarakis et al. 2001, Ma et al. 2009).

Domestic, industrial, and agricultural activities are partly responsible for the higher concentrations of metal contaminants in the Nilüfer Stream. Untreated stream waters are used to irrigate vegetable crops in the agricultural areas, especially in the dry summer season. The domestic and industrial pollutants contained in the untreated wastewaters are transferred to the soil and eventually may enter the food chain (Moon et al. 1994, Lehman and Mills 1994).

Correlation Analysis

The correlation analysis matrix for metal contaminants were obtained from samples taken between 2002 and 2007 and are shown in Table 5. Table 5 shows a high positive relation between Cu and Zn. A moderately positive correlation was found among Cd, Cr, and Mn, and Cr and Mn. The metals which had a high positive correlation were possibly connected to the same pollution source. There was no high or moderately negative correlation between any of the metals. Therefore, the only source of pollution by metal contaminants was thought to be the industrial wastewaters.

CONCLUSIONS

The point and non-point pollutant sources due to intensive industrial activities, residential areas, and agricultural activities in the Nilüfer Stream Basin are causing pollution and decrease the water quality of the basin. Urban WTPs cannot collect all of the waste water of the basin population. The discharge criteria applied to industrial WTPs and a high effluent amount creates a pollution load for the stream. The high positive correlation obtained of the metal contaminants Cu and Zn, and medium positive correlation obtained for Cd, Cr, and Mn metal contaminants shows that the origin of their pollution sources is heavy industry. Domestic, industrial, and agricultural activities are partly responsible for the higher concentrations of metal contaminants in the Nilüfer Stream. It was determined that the Nilüfer Stream was polluted by point and non-point pollution sources and that the water quality along the stream basin has steadily decreased from year to year. Intensive wastewater discharge causes a waste dominating flow in the stream and causes the water quality to steadily decrease over time.

Table 4. Sampling station six (between 2002 and 2007) the change in the water metal contaminant values ($\mu g\ L^{-1}$)

Metals	2002	2003	2005	2006	2007
T As	98±67	52±21	101±49	73±24	81±23
Cd	6±2	9±4	3±1	5±3	7±3
T Cr	88±36	119±46	260±107	198±76	511±202
Cu	11±6	26±15	30±16	40±24	69±37
Mn	255±82	160±61	153 ±69	151±15	274±37
Ni	27±15	155±44	29±11	49±13	73±10
Pb	13±11	134±41	27±10	52±36	119±52
Zn	114±16	249±78	207±70	302±82	373±186

Table 5. Correlation coefficient matrix of metal contaminant in water.

	As	Cd	Cr	Cu	Mn	Ni	Pb	Zn
As	1,00							
Cd	0,51	1,00						
Cr	0,39	0,64*	1,00					
Cu	0,04	0,52	0,67	1,00				
Mn	0,46	0,66*	0,66*	0,57	1,00			
Ni	0,05	0,04	-0,07	0,22	-0,15	1,00		
Pb	-0,08	-0,21	-0,11	-0,02	-0,25	0,30	1,00	
Zn	0,10	0,40	0,55	0,88*	0,36	0,58	0,17	1,00

Note: *Significant at 0.05 level.

In the study by Aydinalp et al. (2005) water being extensively used for irrigation in the Bursa plain is seriously polluted by industrial wastewater and that its use for irrigation is causing soil pollution. However, these water resources are affected by the industrial development of the province and are polluted with wastewater from industry and the city. The Kahramanmaraş textile industry is located mainly in the areas where the plains are concentrated mostly at the rivers. The Aksu River and its tributaries show the highest levels of metal contamination. According to the WQS the Aksu River Cu, Fe, Zn, Mn, Ni, and Pb metal pollutants levels are Class IV (Toroğlu et al., 2006).

The River Gediz, is the second largest river of the Aegean Region and is under the threat of pollution caused by institutions of the region, domestic waste, agricultural chemicals, and artificial fertilizers. In water samples, the metals of high levels are; Pb: 27.0±%0.8 μg/L in the Nif River, Cr: 48.9±%0.9 μg/L at the Muradiye Bridge, Cd: 12.1±%0.6 μg/L at the Istanbul Bridge, Cu:

90.2±%0.4 μg/L at Muradiye Bridge, Ni: 309.8±%0.7 μg/L, Fe: 914.1±%0.3 μg/L, Zn, and 208.3±%0.5 μg/L in Karaçay. The quality of water is at level four, according to the WQS (Öner and Çelik 2011).

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