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Occurrence and removal of metals in urban wastewater treatment plants

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ABSTRACT

In this study, nine metals (Al, Cd, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) found in urban wastewater treatment plants (WTPs) in Bursa (Turkey) were monitored for 23 months in 2002 and 2007. Metal influent and effluent concentrations of wastewater stabilization ponds (WSPs) and the activated sludge process (ASP) measured via 24-h composite samples were used to determine removal efficiencies. Average influent concentrations ranged between $2 \mu g/L$ (Cd) and 1975 $\mu g/L$ (Fe). In the stabilization ponds, the removal efficiency was 58% for Cr, while for Cd, Mn, and Pb, it was less than 20%. The activated sludge process yielded high removal efficiencies, ranging from 47% for Ni to 95% for Cr. The use of treated wastewaters for agricultural purposes was investigated, and it was determined that all metal concentrations met application limits, with the exception of Cr in wastewater stabilization pond effluent. Results showed that wastewater stabilization pond effluent reduced the receiving water quality with respect to Cr, Cu, Ni, and Pb. In addition, it was shown that effluent from the activated sludge process temporarily improved the receiving water quality with regard to the Cd, Cu, Mn, and Zn parameters. However, considering the periodic variations of the metals in both processes, water quality, and agricultural practices, it was determined that they should be monitored continuously.

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1. Introduction

Wastewater treatment plants (WTPs), serving both municipal and industrial districts, receive complex mixtures of nutrients and organic and inorganic micropollutants, which are treated to reduce their concentrations so that they do not impact the environment [1]. Most WTPs throughout the world are designed and regulated to remove nutrients from wastewaters, but it is also known that large amounts of potentially toxic elements, such as metals, enter the wastewater [2]. The presence of metals in industrial and urban wastewater is one of the main causes of water and soil pollution. Accumulation of these elements in wastewater depends on a number of local factors, including the industry type, peoples' way of life, and their awareness of the impacts to the environment by careless disposal of wastes [3,4]. Metals in urban wastewater originate mainly from domestic activities [5,6], industrial activities, and storm water runoff [7]. Metal discharges to the environment not only cause acute toxicity to aquatic organisms, microorganisms, and plants, but also strongly reduce microbial activity, which adversely affects biological WTPs [8]. Wastewater stabilization ponds (WSPs) [9,10] and the activated sludge process (ASP)[11,12] are among the biological processes used in wastewater metal removal. In addition to biological and physicochemical conditions, process operating conditions and design determine metal removal in biological wastewater treatment. However, in spite of the complexity of these factors, various metals can be removed in biological wastewater treatment processes [13,14]. WSPs are particularly efficient in removing metals. The anaerobic WSP has a higher resistance to toxic materials and shock loading [15]. Most removal occurs in the primary ponds (anaerobic or facultative) as a result of the sedimentation of solids to which the metals are adsorbed [16,17]. In biological WTPs, metal removal efficiency depends on the metal species and concentration, the reactivity of the available biopolymers or biomass, and the composition of other wastewater components [18,19]. ASP provides good removal of metals such as Cd, Cr, Cu, Zn, Ni, and Pb. Metal removal by ASP is due to sorption of the flocs [20].

In this study, weekly samples taken from wastewater entering urban WTPs in east Bursa (Turkey) were analyzed in 2002 and 2007 to determine influent/effluent metal characteristics. Removal efficiencies for metals in these WTPs were monitored for 1-year using monthly average values, and the WTP effluent suitability for agricultural irrigation was assessed. Additionally, metal variation was monitored in samples taken from the Nilüfer Stream during two periods to determine the effect of the treated wastewater on the receiving environment.

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WSPwaste stabilization pondASPactivated sludge processWTPwastewater treatment plantBEWTPsBursa east urban wastewater treatment plantsUupstreamDdownstream

2. Material and methods

2.1. Description of the Bursa city eastern urban wastewater treatment plants (BEWTPs)

The BEWTPs (40°13′N, 29°04′E) were established to control pollution into the Nilüfer Stream, which is the receiving water body. The plants were gradually installed over several years. The anaerobic WSPs were put into operation in 1998 and then replaced with an extended aeration ASP in 2006 [21]. Seven WSP units were built, having a total volume of 314.443 m³ and pond depth of 4 m [22]. The ASP, which has been in operation since 2006, has the capacity able to treat wastewater for a population of approximately 1,550,000 [23]. The plant consists of pre-treatment units containing screens, grit removal, screw pumps, a selector tank, anaerobic bio-phosphorus tanks, aeration tanks, a secondary sedimentation tank, and sludge dewatering units, and it treats approximately 160 ML/d.

2.2. Sample preparation

The 24-h composite samples were analyzed once per week from wastewater entering the plants to determine metal content. Samples from the WSPs between January and December of 2002, and those from the ASP between January and November of 2007 were collected as weekly 24-h composite samples using an ISCO 3700 portable sampling kit. The 2-h composite samples were collected from upstream and downstream sections of the Nilüfer Stream on dry days in 2002 (06/20/2002 and 09/19/2002) and 2007 (06/22/2007 and 09/27/2007), and the water quality of the receiving environment with respect to metals (except Al, Fe, and Mn) was analyzed. All samples were collected in polyethylene flasks and precleaned with 30% HNO₃ (Merck) and deionized water according to standard methods [24]. pH and temperature were measured with a Mettler Toledo pH meter.

Metal samples were prepared with a preliminary digesting process via the 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 HNO₃ (65% analytical grade) and 4 mL of HCl (37% analytical grade) were added to the cell. The cells were covered and a maximum pressure of 180 psi and a temperature of 160 °C were applied for 20 min. In the second step, the samples were allowed to cool for 10 min. After 30 min, 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 ICP-AES (Vista MPX, Varian) analysis.

2.3. Analysis

The metal concentrations in the digested samples were analyzed using ICP-AES. Nine metals were targeted: Al, Cd, Cr, Cu, Fe, 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 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 from the 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: $2 \mu g/L$ for Cd, $3 \mu g/L$ for Pb, $5 \mu g/L$ for Cr and Cu, $10 \mu g/L$ for Mn and Zn, $20 \mu g/L$ for Ni, $100 \mu g/L$ for Fe, and $200 \mu g/L$ for Al. Certified liquid samples were used to check analytical accuracy, which ranged between 1% and 10%.

2.4. Statistical analysis

All reagents used were of analytical grade or better. Multivariate analyses (element coefficient correlations) were used to determine the metal levels of the influent samples, which were performed using the SPSS statistical package program. A probability of 0.05 or less was considered as statistically significant.

3. Results and discussion

3.1. Untreated wastewater characterization

Variations in the metals analyzed from untreated wastewater in 2002 and 2007 are given in Table 1. The results illustrate that the wastewater metal composition is complex and guite variable. Similar results were obtained in other studies of untreated wastewaters [25–27]. The annual average values of 2007 show that the Al, Cd. Fe. Pb. and Zn metal concentrations increased by more than 20% compared with 2002. The variation in wastewater metal content was caused by the diversity in economic activities throughout the region. There are 80 textile, 90 leather, 26 metal plating and processing, 160 car maintenance, 6 auxiliary, 16 plastic rubber, 8 food, 13 laundry, 7 concrete, and 31 catering industries that are not included in the organized industrial district [28]. The textile industry is likely to produce Cr and Zn and the leather industry has Cr in its effluent, while Cr, Cu Fe, Ni, and Zn are attributed to the metal industry [29]. Al, Cr, Fe, and Zn generally entered the treatment plants at high concentrations. The reason these metals had higher concentrations than the others is that the textile and leather sectors were intensified in the region. In addition, groundwater used by these industrial companies increases the Fe and Mn values. The BEWTP influent includes household and industrial wastewaters. Industrial wastewater from the factories operating outside of the organized industrial zones and household wastewaters are treated by the BEWTP in the east side of the city and discharged into the Nilüfer Stream [30].

Table 1	
Range and mean values of metals in untreated wastewater ($\mu g/L$).

Metal	2002		2007	2007			
	Range	Mean \pm SD	Range	Mean \pm SD			
Al	849-1916	1302 ± 338	603-3753	1891 ± 872			
Cd	0-10	2 ± 3	0-137	19 ± 40			
Cr	742-1171	1009 ± 339	174-2120	1086 ± 509			
Cu	9-400	64 ± 108	12-179	60 ± 43			
Fe	994-2259	1499 ± 406	1038-3580	1975 ± 712			
Mn	42-139	104 ± 28	97-217	126 ± 33			
Ni	0-202	84 ± 57	59-202	100 ± 41			
Pb	1-47	16 ± 14	6-358	84 ± 100			
Zn	204-1036	387 ± 240	303-982	533 ± 209			

SD: standard deviation.



Fig. 1. Monthly variations in concentrations of the metals in the WSP influent and effluent ((**□**) influent, (**□**) effluent).

3.2. WSP influent and effluent metal concentrations and removal efficiencies

Monthly variations in the concentrations of the nine metals investigated in the WSP influent and effluent within the January–December 2002 period are presented in Fig. 1. Metals in particle form or adsorbed onto suspended solids in the wastewater settle in the pond, and are removed. Therefore, influent values are generally higher than effluent values. Moreover, changes were seen periodically in some of the metal concentrations (Cd, Cu, Mn, Pb, and Ni). This is because metals are released from the organic sludge during decomposition [31], or are replaced with H ions and released under acidic conditions [32].

The average removal efficiency in the WSPs is listed as Cr > Al > Fe > Zn > Cu > Ni > Pb, while Cd and Mn were not removed.

Metal removal is performed via settling in anaerobic WSPs. However the complex formation and dissolution values for each metal are different [33]. Metal removal efficiencies in WSPs vary by metal and type of WSP system, but in general, removal improves with the number of ponds in the WSP system, particularly if the final ponds are aerobic maturation ponds [8].

3.3. ASP influent and effluent metal concentrations and removal efficiencies

Fig. 2 shows monthly variations in concentrations of the metals in the ASP influent and effluent during the January–November 2007 period.

Periodic high influent metal values (\sim 4000 µg/L Al, \sim 3500 µg/L Fe, \sim 2400 µg/L Cr) were measured (Fig. 2). Effluent values were



Fig. 2. Monthly variations in concentrations of the metals in the ASP influent and effluent ((■) influent, (□) effluent).

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Table	2		
Correl	ations of metals influent water samples in 2	002 and	2007

	Al	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
Al	1								
Cd	0.09	1							
Cr	-0.11	0.06	1						
Cu	0.03	0.32	0.26	1					
Fe	0.82	0.24	0.22	0.01	1				
Mn	0.38	0.76	0.32	0.38	0.52	1			
Ni	0.11	0.53	0.23	0.45	0.34	0.51	1		
Pb	0.13	0.93	0.11	0.34	0.20	0.72	0.47	1	
Zn	-0.06	0.22	0.40	0.64	0.10	0.36	0.43	0.27	1

always less than influent values for all metals in all measurement periods, which indicate effective removal. It is possible that metal removal in the ASP occurs both in the primary treatment (where a portion of metals adsorb to the particles) and in secondary biological treatment (where metals are removed by biosorption) [27,34].

ASPs are chiefly designed for removal of organic matter by activated sludge microorganisms. Therefore, removal of metals by these systems may be regarded as a side benefit, and has been found to be quite variable [35,36]. Metal contents are listed as Cd < Cu < Pb < Ni < Mn < Zn < Cr < Al < Fe for the 2007 measurement period. Hence, wastewater metal removal may be influenced by their initial influent contents. The relationships between influent metal content and removal efficiency (Fig. 2) agree with other research findings [12,25-27,35,37-39], where it was observed that metal removal efficiencies were directly proportional to metal influent concentrations. Furthermore, metal removal efficiency is not only affected by metal ion species and concentration, but also by other conditions such as operating parameters, and physical, chemical, and biological factors [40]. For example, it is known that metal removal by ASP is dependent on dissolved organic matter [41] and pH [39,41], whereby the removal efficiency increases with pH until they precipitate as hydroxides. Biological wastewater treatment is normally conducted at pH 7-9. Thus, because of differing metal solubilities at these pH values, and since the composition of wastewater is complex and highly variable, the variability in metal removal is attributed to these factors [39]. In this study, pH values for untreated and treated wastewater samples ranged from 7.86 to 8.13 at 11.2-22.2 °C. This caused variations in removal efficiencies for metals (47-95%). Therefore, the level of metal removal from wastewater remains unpredictable.

3.4. Correlation analysis

Table 2 shows the correlation matrix for all observed influent metal values. The correlation analysis matrices for metals are obtained from samples taken in 2002 and 2007, and are shown in Table 2. There was a highly positive relation between Pb and Cd. A moderately positive correlation was found among Mn, Cd, and Pb, and for Al and Fe. We hypothesize that metals with a high positive correlation are possibly from the same pollution source. There was no highly or moderately negative correlation between any of the metals. Therefore, all metal pollution is attributed to industrial wastewaters.

3.5. Employing effluent to agricultural irrigation

Treated water from the treatment processes is discharged into the Nilüfer Stream, which is generally used for irrigation [23]. Metals in wastewaters significantly increase the metal content in soils irrigated by this water [42,43], and metals are transferred to the plants and through food chain [44]. Plants grown with high metal content soils pose a significant human health risk if consumed [45]. Applicable national and international standards for metals in effluents are presented in Table 3 to determine whether metal contents are suitable for agricultural irrigation. National guidelines [46] have been developed for metals and trace elements according to international guidelines, such as Ayers and Wescot's standards [47]. WSP effluent values are below national guidelines thresholds for shortterm irrigation with respect to metals. Cr is above the threshold for long-term irrigation. The WSP effluent is below threshold except Cr according to international guidelines. The ASP average effluent values meet irrigation standards according to both national and international guidelines. Although similar Cr influent values were measured in both processes (Table 1), high removal efficiency in ASP ensured that its effluent was suitable for agricultural irrigation. However, the high standard deviation values obtained in both processes for Cd are related to periodic high values measured in the effluent and influent (Figs. 1 and 2).

3.6. Effluent discharge impact on stream quality

Domestic, industrial, and agricultural wastes are discharged into the Nilüfer Stream as reported in several studies [48-50]. For example, Yılmaz et al. [50] found that Cr and Pb concentrations were above the standard limits given for the heavily polluted waters. Samples were taken from the upstream (40°14'N, 29°05'E) section of the treatment plants, and from the mouth section of the stream approximately 1500 m (40°14′N, 29°04′E) before it converges with other branches, in order to determine the effect of the metals in the treatment plant effluent on the Nilüfer Stream. Values of important metals (except Al, Fe, and Mn) and the quality criteria for national inland water resources [51] are presented in Table 4. Because metal values in WSP effluents are higher than metal values in the receiving environment, higher concentrations were typically measured downstream of the effluent outfall. Concentration of Cr, Cu, Ni, and Pb rose downstream of the effluent, while Cd and Zn remained unchanged. High metal concentrations in WSP effluents increased most metal concentrations in the receiving environment (Cr, Cu, Ni, Pb). Generally, lower metal values were measured downstream of the ASP. These lower metal values may be attributed to dilution caused by the treatment plant effluents (Table 3), the variable flow rate of the receiving water, and the difference between pH

Table 3

Comparison of treatment plant average effluent metal contents with applicable agricultural irrigation guidelines ($\mu g/L$).

Metal	WSP	ASP	National guidelines	International guidelines	
			Long-term use	Short-term use	
Al	593 ± 214	446 ± 285	5000	20,000	5000
Cd	4 ± 5	6 ± 8	10	50	10
Cr	423 ± 132	57 ± 40	100	1000	100
Cu	50 ± 77	17 ± 17	200	5000	200
Fe	838 ± 310	338 ± 172	5000	20,000	5000
Mn	112 ± 40	39 ± 24	200	10,000	200
Ni	67 ± 40	53 ± 24	200	2000	200
Pb	12 ± 9	30 ± 22	5000	10,000	5000
Zn	259 ± 196	150 ± 99	2000	10,000	2000

Metal	20/06/2	20/06/2002		19/09/2002		22/06/2007		27/09/2007		National water quality classes (WPCR, 2004)		
	U	D	U	D	U	D	U	D	I	II	III	IV
Cd	0.4	1.1	3.2	3.1	11	6	2	2	3	5	10	>10
Cr	59	82	45	96	52	51	84	53	20	50	200	>200
Cu	12	24	11	15	26	28	49	16	20	50	200	>200
Ni	37	35	40	57	44	43	49	57	20	50	200	>200
Pb	8	10	15	19	93	99	54	15	10	20	50	>50
Zn	91	121	43	79	165	173	647	215	200	500	2000	>2000

 Table 4

 Change in water quality of Nilüfer Stream and water quality criteria for inland water resources (μ g/L).

U: upstream, D: downstream.

values of the treated and the receiving waters [52]. One Cd value measured upstream exceeds the threshold specified for long-term agricultural irrigation in both the national and international guide-lines (Table 3). The Cd concentrations downstream of the treatment plant were reduced enough to meet the threshold standards for irrigation. In addition, Cd, Cu, and Zn values in the ASP effluent downstream of the treatment plant increased periodically. There were no changes in threshold limits observed with respect to Cr and Pb. Periodic decreases in water quality with respect to Ni was observed.

4. Conclusions

In this study, nine metals were measured over 23 months. Their contents in the treatment plant influent are shown to be guite variable. Al, Cr, Fe, and Zn exhibited the highest concentrations, as a result of intensive textile, metal, and leather industries in the region. Average values for 2007 showed that Al, Cd, Fe, Pb, and Zn concentrations increased by more than 20% over those measured in 2002. A high positive correlation was obtained for all influent metal values of Pb and Cd. A medium positive correlation was obtained for Mn, Cd, and Pb, and Al and Fe. The origin of the metals was attributed to industrial sources. Removal efficiencies greater than 50% were achieved for Al and Cr in WSPs, while Cd and Mn were not removed. Since there are few studies on metal removal in anaerobic WSPs, further investigation is required. For the ASPs, removal efficiencies were affected by influent metal contents and the pH of the process. Removal efficiencies ranged between 47% for (Ni) and 95% for (Cr). The suitability of effluents for agricultural irrigation was considered, and it was found that the Cr level in the WSP effluents does not meet national guidelines, while the ASP effluents do. Results showed that Cr, Cu, Ni, and Pb in the WSP effluents periodically reduced the Nilüfer Stream water quality, which is the receiving water body. However, ASP effluents were shown to periodically improve the Nilüfer Stream water quality with respect to Cd, Cu, and Zn. It is recommended that metals be monitored continuously for agricultural irrigation and water quality of the receiving environment, especially for Cd. The ASP process was shown to yield higher removal metal efficiencies compared to WSP in the treatment of urban wastewater, despite having higher metal concentrations in the influent. In addition, ASP effluents were suitable for agricultural irrigation and improved the water quality in the receiving environment.

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