

Original Article

Heavy metals and polycyclic aromatic hydrocarbons in sludge from three industrial wastewater treatment plants of the industrial parks of Isfahan province

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ABSTRACT

Aims: In this study, the characterization of heavy metals (HMs) and polycyclic aromatic hydrocarbons (PAHs) were investigated in three different industrial sewage sludges in industrial parks of Isfahan province, Isfahan, Iran.

Materials and Methods: Sludge samples were collected from three dry bed wastewater treatment plants (WWTPs) within the province during of four seasons of 2011-2012. Gas chromatography-mass spectrometry and inductively coupled plasma were used to measure PAHs and HMs contents in sludge.

Results: The average concentrations of Cd, Cr, Cu, Mo, Ni, Pb, As, Hg, and Se in sewage sludge were 305.67, 514, 1019, 6.46, 102.33, 1181.33, 277.33, 226.83, and 6.35 mg/kg dry weight (d.w.), respectively. The concentrations of HMs in most of the sludge samples were above the regulatory limits for the sludge class 1 and 2 to be used in agriculture in Iran. The levels of Σ_8 PAHs varied from 92.33 $\mu\text{g}/\text{kg}$ to 257.87 $\mu\text{g}/\text{kg}$ d.w. in the sludge, that lower than the limitation value (6 mg/kg) recommended by the Europe Union and US Environmental Protection Agency for land application. Phenanthrene, pyrene, and fluorene were predominant isomers of PAH in sludge samples. The highest value was found in the Morchehort WWTP.

Conclusion: The concentration and composition of PAHs and HMs in sewage sludge varied and depended mainly on the quantity and type of industrial wastewater accepted by the WWTPs. There is a pyrene in sewage samples that is a carcinogenic. The concentration of HMs in sewage sludge was above of standard levels. Finally, we are not recommended the using of industrial sewage sludge in agriculture because of the high value of HMs was found in samples.

Key words: Heavy metals, industrial parks, Isfahan province, polycyclic aromatic hydrocarbons

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INTRODUCTION

The industries are one of the major issues of global concern for the environment because the release of smoke, gases, effluents, and solid wastes.^[1] Wastewater treatment plants (WWTPs), especially those serving industrial areas, continuously receive very complex mixtures. Sewage sludge

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is an undesirable but unavoidable by-product of wastewater treatment process. The presence of hazardous materials in this sludge is becoming an important topic.^[2,3] By the way, it is reported that industrial sludge from WWTPs potentially transport chemical pollutants such as heavy metals (HMs) and persistent organic pollutants.^[4,5] Actually, pollution adversities may arise from toxic HMs that are taken up by plants are either mobilized into the soil solution or transported in drainage waters. Detrimental organic compounds in industrial sewage sludge may potentially get into other environmental media to which humans may be exposed. Due to their accumulation with frequency use of industrial sludge in soil and their toxic, mutagenic, and carcinogenic properties, US Environmental Protection Agency has listed 16 polycyclic aromatic hydrocarbons (PAHs) as the priority pollutants for remediation.^[6-9] Accumulation of these pollutants in plants, soils and animals pastures and subsequent entry into the food chain has happened.^[10,11] Therefore, any form of disposal need to be controlled in order to protect human health and the environment.^[3] The increasing amount of industrial sludge produced as a consequence of the rising number of industrial WWTPs in Iran, hazardous substances in industrial sludge have become an important environmental issue. Isfahan located in the Iran Center, Isfahan province is one of the most developed regions in Iran. It is a Metropolis of Iran and also harbors many industrial parks. There are 63 industrial parks that about nine have WWTPs. We are studied three of them, Morchekhort, Mobarake, and Oshtorjan with the treatment capacity of about 1,100,600 and 500 m³ wastewater/day. Approximately, >80 tons of sewage sludge are produced; most of the industrial sludge are piled in temporary places that become other potential pollution sources. We must be to adopt a practical, economic and acceptable approach in managing and disposing of sewage sludge. Currently, incineration and land application are proposed as the main alternatives for the disposal of sludge. Considering the organic matter and plant-available are rich in the sludge, land application is thought to be an inexpensive and better method.^[8,12] Many studies have shown that PAHs are the most significant organic pollutant in industrial sludge of different countries such as Norway, Spain, Germany, the United Kingdom, and France.^[10,13,14] The study of Ozcan *et al.* on the levels of HMs, PAHs, and polychlorinated biphenyls in sewage sludge in Turkey has shown, the concentrations

of HMs, including Cd, Cr, Cu, Ni, Pb, and Zn in sludge samples did not exceed the limits regulations. However, in some samples the ΣPAHs concentration ranging from 1077 to 17,509 µg/kg dry matter (DM) and was not suitable for using in agriculture.^[15] In 2011, the total amounts of PAHs in sewage sludges ranged from 1.9645 to 6.5752 mg/kg, which did not exceed the projected European Union (EU) cutoff limits (6 mg/kg) for sludge found in farmland.^[16] In 2014, survey of HM concentrations of sewage sludge samples from 107 municipal sewage treatment plants located in 48 cities in China, shows, 85 of the 107 municipal sludges analyzed would be considered suitable for land application.^[17]

This study was to investigate the concentrations and characterization of the PAHs and HMs in industrial sludge of three WWTPs in industrial parks of Isfahan province. The results of the research are useful for the provincial policy makers to make proper decisions and suitable application of sewage sludge in to agriculture soils.

MATERIALS AND METHODS

Sampling method

Sewage sludge samples were taken from three WWTPs within Isfahan province, Isfahan, Iran. These WWTPs were selected to be big industrial parks. The namely of these WWTPs are Morchekhort, Mobarake, and Oshtorjan. The characteristics and wastewater quality and quantity of three WWTPs are shown in Table 1. Most of the WWTPs mainly receive of industrial and sanitary wastewater of factories. Distributions of the factories are shown in Table 2. Sampling and analysis of sludge were carried out from December 2011 to September 2012. Five replicated samples with 1 liter were taken from each WWTPs, Samples were collected from four points of dry beds and then mixed.^[18]

Sampling preparation and examination

All sludge samples were stored at freezing condition for further analysis. All samples were freeze-dried, sieved in an agate mortar to obtain a fraction <0.1 mm. Series Optima Perkin Elmer 4000 ICP-OES was used to detect the concentration of Cr, Cd, Cu, Pb, Ni, Se, and Mo, Perkin Elmer 4100 equipped with graphite furnace for arsenic and Perkin Elmer flow injection mercury system

Table 1: The characteristics and wastewater quality and quantity of 3 WWTPs are in industrial parks

Samples	Parameters							
	COD	BOD ₅	TSS	pH	Wastewater treatment methods	Sludge processing methods	Flows (m ³ /day)	Industrial parks
Influent	1046	407	357	7.36	AC + extended aeration	Gravity thickened + filter	500	Oshtorjan
Effluent	127	24	75	7.22	activated sludge	press + dry bed sludge		
Influent	3000	1500	1400	7.2	UAFB + extended aeration	Gravity thickened + filter	600	Mobarake
Effluent	70	20	70	7.5	activated sludge	press + dry bed sludge		
Influent	1400	500	320	7.1	AC + SBR	Gravity thickened + filter	1100	Morchekhort
Effluent	100	40	50	7.6		press + dry bed sludge		

The unit of parameters is mg/l except pH, AC: Anaerobic contact, UAFB: Up flow anaerobic fixed bed, SBR: Sequencing batch reactor

for measurement of mercury. The total content of each HMs determined by 0.5 g of sample was digested in a 20 mL mixture of nitric, hydrochloric, and hydrofluoric acid (in the ratio 1:1:2).^[18,19]

Polycyclic aromatic hydrocarbons were carried out with 20 mL mixture of organic solvents from freeze-dried samples by a dichloromethane-methanol (2:1) mixture in a bath with sonication, acetone-dichloromethane (1:1 v/v) for three times of sonication (20 min each time). The extracts were evaporated to 1 mL, and the remained was then dissolved in 2 mL n-hexane. A clean-up and separation chromatography column was used to remove impurity such as lipids and sulfur from the extracts. The layers of silica gel (200-300 mesh) used for the column packed, neutral aluminum oxide (100-200 mesh), activated copper powder and anhydrous sodium sulfate, and other chemicals were used.^[20,21] An alumina column was used for cleaned-up of the sludge extracts. Four surrogate standards ($[^2\text{H}_8]$ naphthalene, $[^2\text{H}_{10}]$ anthracene, $[^2\text{H}_{12}]$ benzo[a]anthracene, and $[^2\text{H}_{12}]$ benzo[ghi]perylene) were used for quantitation by gas chromatography (GC)-mass spectrometry (MS) model of GC 6890N Agilent and MS 5975C, MODE EI, operating in the electron impact mode (70 eV). A 30 m HP-5 column (0.25 mm ID, 0.25 μm treated film thickness) was used to separate the conventional PAHs. High-grade helium gas was used as the carrier's gas. Injection was made in splitless mode. The oven temperature was increased from 80°C to 255°C at 15°C/min, then increased at a rate of 1°C/min to 26°C and then increased at a rate of 2.5°C/min to 295°C, and finally held at 295°C for 1 min. Injector and transfer line temperatures were 2800°C and 3000°C, respectively.^[13]

General parameters such as pH, electrical conductivity (EC), total Kjeldahl nitrogen (TKN), total organic matter (TOM), ash content, phosphorous, and potassium were measurement according to Institute of Standards and Industrial Research of Iran (ISIRI) guideline.^[22]

Finally, statistical analysis *t*-test and ANOVA were used for analysis and comparison between data and diagrams were drawing by Excel software.

RESULTS

General properties of sewage sludge

Some general properties the sludge samples collected from the three WWTPs are listed in Table 3. Fluctuations in pH, EC, ash content, TOM, TKN, phosphorous, and potassium, could be clearly observed. pH value ranged from 7.3 to 7.61, EC from 2.48 to 6.47 dS/m, ash content from 41.17% to 66.08%, TOM from 33.95% to 58.85%, TKN from 1.98% to 2.33%, phosphorous from 0.54% to 0.63%, and potassium from 0.8% to 1.71% dry weight (d.w.), respectively. The lowest concentrations of main nutrients were observed in sewage sludge from Oshtorjan WWTPs.

Heavy metals in sewage sludge

The concentrations of HMs expressed on a dry mass basis in sludge are given in Table 4. Iran legislation (ISIRI 1071) prohibits the use of sewage sludge as fertilizer in agriculture that exceeds the maximum allowed values for concentrations of HMs. It is evident from the data of Table 4 that concentrations of Cd, Cr, Pb, As and Hg from 253.33 to 305.67, 171 to 514, 278.33 to 1181.33, 75.67 to 277.33, 84.17 to 226.83 mg/kg d.w. respectively. In all of the sewage sludge exceed the limit values for basic soil in Iran legislation. Between HMs was mentioned Pb in the sewage sludge from Morchehort with 1181.33 mg/kg d.w. has a highest concentration.

However, concentration of Cu, Ni, Se, and Mo in most of sewage sludge can be used for basic soil according to Iran legislation standard. The highest concentrations of HMs are evident from Morchehort sewage sludge, and lowest concentration of them observed in Oshtorjan sewage sludge.

Table 2: Distributions of the factories are in industrial parks

Industrial parks	Type of industry							Total
	Metal	Chemical	Textile	Nutrition	Nonmetal mineral	Cellulose	Electrical and electronics	
Morchehort	75	68	30	54	15	20	15	277
Mobarake	129	106	35	31	25	16	5	347
Oshtorjan	102	25	5	—	10	4	8	154

Table 3: General properties of sludge

Parameters	Units	Oshtorjan	Mobarake	Morchehort
pH	—	7.61 ± 0.28	7.30 ± 0.1	7.36 ± 0.32
EC	dS/m	2.48 ± 0.82	7.23 ± 0.87	6.47 ± 0.91
Ash content	%	66.08 ± 10.38	41.17 ± 3.76	45.53 ± 7.92
Organic matter	% d.w.	33.95 ± 10.43	58.85 ± 3.78	54.45 ± 7.94
TKN	% d.w.	1.98 ± 1.55	2.33 ± 1.17	2.09 ± 0.36
Phosphorous (P ₂ O ₅)	% d.w.	0.54 ± 0.29	0.63 ± 0.38	0.63 ± 0.48
Potassium (K ₂ O)	% d.w.	0.80 ± 0.92	1.04 ± 0.43	1.71 ± 1.3

TKN: Total Kjeldahl nitrogen, EC: Electrical conductivity, d.w.: Dry weight

Table 4: The average concentration of heavy metals in industrial sludge samples (mg/kg d.w.)

Parameters	Industrial parks				
	Oshtorjan	Mobarake	Morchehort	Standards of Iran*	
				Class 1	Class 2
Cd	277.67 ± 73.85	253.33 ± 51.86	305.67 ± 131.33	10	25
Cr	171 ± 60.31	224.33 ± 42.67	514 ± 179.64	—	—
Cu	152 ± 15.39	325 ± 141.45	1019 ± 278.91	500	1000
Mo	4.33 ± 0.57	5.23 ± 1.52	6.46 ± 2.02	—	—
Ni	32.73 ± 14.43	42.30 ± 6.85	102.3 ± 29.69	100	200
Pb	496 ± 147.62	278.33 ± 191.09	1181.33 ± 625.46	250	500
As	75.67 ± 74.47	154.67 ± 127.22	277.33 ± 239.42	—	—
Hg	84.17 ± 49.48	106.67 ± 129.71	226.83 ± 193.77	10	10
Se	2.23 ± 1.86	4.37 ± 3.59	6.35 ± 5.22	—	—

*Limitation values for agriculture application of compost in Iran, d.w.: Dry weight

Concentration of polycyclic aromatic hydrocarbons in sewage sludge

The concentration of PAHs in industrial sludge collected from the three WWTPs is shown in Table 5. The total content of PAHs in the examined sludge ranged from 92.33 to 257.87 µg/kg d.w. The distributions of PAHs with different rings showed that phenanthrene, fluorine, and pyrene with concentration of 91.67, 80.67, and 31.33 µg/kg appeared to be the primary components in most of the tested sludge samples, respectively. The content of PAHs in Morchehort sludge samples is higher than others.

Table 5: The Concentration of PAHs in industrial sludge samples (µg/kg d.w.)

Parameters	Industrial parks		
	Oshtorjan	Mobarake	Morchehort
Naphthalene	9.33 ± 1.53	9.50 ± 3.04	11.67 ± 2.75
Acenaphthylene	7.50 ± 1.5	7.80 ± 1.93	9.67 ± 2.25
Acenaphthene	5.87 ± 1.52	5.17 ± 2.47	7.70 ± 1.75
Fluorene	38.67 ± 20.03	11.97 ± 3.05	80.67 ± 31.09
Phenanthrene	70.33 ± 40.07	21.23 ± 11.17	91.67 ± 62.92
Anthracene	7.27 ± 2.41	8.00 ± 3.00	11.67 ± 4.16
Fluoranthene	8.43 ± 1.41	9.17 ± 2.75	13.50 ± 5.07
Pyrene	21.93 ± 11.49	19.50 ± 4.5	31.33 ± 7.09
Total	169.33 ± 63.91	92.33 ± 20.34	257.87 ± 89.95

PAHs: Polycyclic aromatic hydrocarbons, d.w.: Dry weight

DISCUSSION

In this paper, all the industrial sewage sludge samples were found to be rich in nitrogen, phosphorous and potassium in sewage sludge and can be used for basic soil according to Iran legislation standard (ISIRI 1071). As well as TOM in the Oshtorjan sample was relatively lower than that in the others. In addition, the pH and EC of all the sludge samples were suitable for plant growth. The study of Yang *et al.* showed the main nutrients that is, potassium, phosphorus, and total nitrogen are 7.1%, 22.7%, and 24.5% in urban sewage sludge, respectively and these concentrations are higher than those in this research.^[17] The study of Izewska *et al.* was done on the assessment of physical, chemical, and energetic properties of sludge in industrial and municipal sewage treatment plant has shown among many macronutrients, sludge contains much of the following: Nitrogen, phosphorus, calcium, and magnesium, but little potassium and result according to this research.^[23]

Heavy metals include Hg, Cu, Cr, Pb, Cd, Se, and Ni, which are common elements in sludge and have severe negative effect on the environment at a high concentration. For the HMs with a higher toxic potential, their high levels in exchangeable and absorbed, carbonate-precipitated, oxidizable bound fractions predict a possible negative effect on the reuse of sludge, particularly when they are used for land agriculture application and the possibly polluted agricultural crops for human consumption.^[24] In this study, on the basis of HM concentrations from Table 4 and according

to Iran legislation standard, the Morchehort, Mobarake, and Oshtorjan sewage sludge are not recommended for use in agriculture. Particularly, the highest HMs concentrations were measurement in Morchehort sewage sludge.

The concentrations of HMs, including Cd, Cr, Cu, Ni, Pb, and Zn in sludge samples from urban WWTPs in Turkey did not exceed the limits described in standard limitations.^[15] The survey of HM concentrations of sewage sludge samples from 107 municipal sewage treatment plants located in 48 cities in China was showed, the average concentrations of As, Cd, Cr, Cu, Hg, Ni, Pb, and Zn in sewage sludge were 20.2, 1.97, 93.1, 218.8, 2.13, 48.7, 72.3, and 1058 mg/kg, respectively.^[17]

As a result of the decreased discharge of HMs into industrial wastewater in China and the additionally stringent regulations governing the concentration of industrial wastes entering sewers, the average concentrations of HMs such as Cd, Cr, Cu, Hg, Ni, Pb, and Zn have decreased by 32.3%, 49.7%, 54.9%, 25.0%, 37.2%, 44.8%, and 27.0%, respectively.^[17] The higher concentration of Cd, Cr, Pb, As, and Hg in certain sludge samples might result from industrial sources.^[25,26] Therefore, different factories that are exiting in industrial parks and presented in Table 2 have a significant role in increasing the concentration of HMs. Although the high HMs concentration in sewage sludge may not only result from industrial source but also from leaching of pipes.^[17]

Due to the potential risks of PAHs could not be recommended for direct use in agriculture, before a suitable treatment.^[27] The EU-suggested limit 6 mg/kg d.w. for PAHs.^[28] In this study, on the basis of PAHs concentrations from Table 5 and according to European Union (EU) legislation standard, the Morchekhort, Mobarake, and Oshtorjan sewage sludge were suitable for use in agriculture after suitable treatment. Particularly the highest PAHs with 257.9 µg/kg d.w. were measurement in Morchekhort sewage sludge. The ΣPAHs concentration is ranging from 960 to 7680 µg/kg d.w. shows that sewage sludges of WWTPs in Turkey from plant #1 could be used for agricultural objective. But, sewage sludges from plant #2 (ΣPAHs: 1077-17,509 µg/kg d.w.) were not appropriate for using in agriculture because of industrial wastewater entering sewers.^[15] The study was done by Ning *et al.* on the levels, composition profiles and risk assessment of PAHs in sludge from 10 textile dyeing plants was showed that the total concentrations of 16 PAHs (Σ₁₆ PAHs) varied from 1463 ± 177 ng/g to 16,714 ± 1507 ng/g with a mean value of 6386 ng/g.^[29] Total PAH concentrations (Σ₁₂ PAHs) determined by Salihoglu *et al.* in Turkey in all of the sludge samples ranged from 1781 to 19,866 µg/kg DM.^[30] Karaca and Tasdemir have found that 10 PAH (Σ₁₀ PAH) compounds were targeted, and their average value in the industrial sludge was found to be 4480 ± 1450 ng/g DM.^[31] Although, the concentration of PAHs are investigating to the numbers of isomers. However, the type of industries, transport vehicles, and fuel-driven machineries could increase the content of PAHs in the sludge of WWTPs located in an industrial zone. Berset and Holzer have found that the level of PAHs in industrial sewage sludge is higher than domestic sewage sludge.^[20]

Finally, the quantity and type of industrial wastewater that are produce in industrial parks effects on the concentration of PAHs sewage sludge. The studies were showed that the quantity and type of industrial wastewater flowing into the treatment plants have a significant effect on the concentration of PAHs in sewage sludge.^[27] Zhai *et al.* have found that significantly differences were observed in overall PAH values between WWTPs receiving domestic effluents and those receiving industrial effluents. The total amounts of PAHs in sludge from the WWTPs, which mainly received industrial effluents, were significantly higher than those of them, which received only domestic effluents.^[16]

In this study, phenanthrene, fluorene, and pyrene with concentration of 91.67, 80.67, and 31.33 µg/kg appeared to be the primary components in most of the tested sludge samples, respectively. Phenanthrene, fluorene, and pyrene have a 3, 3, and 4 rings. The study was showed that PAHs with 4-6 rings have a greater carcinogenic potential than those with 2, 3, or 7 rings.^[7] Therefore, pyrene is a carcinogenic in our sewage sample. The composition profiles of PAHs were characterized by 3- and 4-ring PAHs, among which phenanthrene, anthracene and fluoranthene were the most dominant components. The mean benzo[a]pyrene equivalent concentration of Σ₁₆ PAHs

in textile dyeing sludge was 423 ng/g, which was 2-3 times higher than concentrations reported for urban soil.^[29] The distributions of PAHs compounds in digested sludges were analyzed. 2-, 3-, 4-benzene rings were predominant, of the sum concentration of PAHs.^[16]

We are measurements of PAHs in sludge dry bed if the concentration is higher in wet sludge possibility. Karaca and Tasdemir have found that total PAH content of the sludge was decreased by 25% in the ambient air environment. Meteorological conditions, atmospheric deposition, evaporation and sunlight irradiation played an effective role in the changes in PAH levels during the tests carried out in ambient air environment.^[31]

CONCLUSION

All the results showed that sewage sludges from three WWTPs were found to be rich in macronutrient and had the potential for being used in agriculture. As well as the pH and EC of all the sludge samples was suitable for plant growth according to Iran legislation standard (ISIRI 1071).

The concentrations of toxic HMs such as Cd, Cr, Pb, As, and Hg in sludge samples exceed the limits described in standard limitations. Particularly in Morchekhort sewage sludge. The higher concentration of HMs in sludge samples might result from industrial sources particularly metal, electronic and plating industries.

According to EU legislation standard, PAHs concentrations of the Morchekhort, Mobarake, and Oshtorjan sewage sludge were suitable for land application after suitable treatment and the most concentration was measurement in Morchekhort sewage sludge. The quantity and type of industrial wastewater that are produce in industrial parks effects on the concentration of PAHs sewage sludge. Particular industries are chemical, cellulose, etc. Phenanthrene, fluorene, and pyrene were appeared to be the primary components in most of the tested sludge samples. Pyrene is a carcinogenic in sewage samples. The PAH content of the sludge was decreased in the ambient air environment.

Finally, we are not recommended the using of industrial sewage sludge in agriculture because of the high value of HMs was found in samples.

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