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Investigating the reduction of BTEX in automotive paint sludge combined with biological sludge by vermicomposting process using *Eisenia fetida*

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Abstract

Background: Due to the fact that in the process of car painting in the automotive industry, sludge containing dangerous compounds of benzene, toluene, ethylbenzene, xylene which cannot be released into the environment without purification, is inevitably produced, this study was conducted to investigate the feasibility of removing BTEX (benzene, toluene, ethylbenzene, and xylene) from the paint sludge of Saipa Automotive Company using *Eisenia fetida* worms.

Methods: This is an experimental study. First, mixtures with different proportions of sludge were prepared and loaded in suitable boxes. After preparing the desired sludge, their quantitative and qualitative characteristics were determined in terms of type and amount of BTEX, volatile materials, moisture content, and C/N ratio. Then, to check the changes in BTEX, sampling was performed on different days during 90 days. BTEX measurements were performed using GC-MS method (NIOSH Method 1501).

Results: The results showed that in the best mixing ratio of sludge, the amount of benzene decreased from 3 mg to less than 0.01 mg in 30 days, toluene decreased from 1.5 mg to zero over a 45-day period, ethyl benzene was reduced from 7 mg to zero mg over 70 days, and xylene decreased from 18 mg to 0.9 mg over 90 days. In addition, in the same optimal mixing ratio, the amount of volatile organic matter, pH, and C/N ratio also had a decreasing trend in the vermicomposting process.

Conclusion: According to the results, *E. fetida* worms are able to work in mixed sludge and have the ability to break down BTEX.

Keywords: Benzene, Toluene, Ethylbenzene, Xylene, Sewage, Paint

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Introduction

One of the most important environmental aspects of the automotive industry is its paint sludge, the amount of which is very high. For example, in Saipa Automotive Company, which produces 1000 cars per day, about 3000 kg of paint sludge is produced. This sludge contains toxic mineral and organic substances such as benzene, toluene, ethylbenzene, and xylene known as BTEX, the direct discharge of which into the environment is dangerous for humans and the environment. Volatile organic compounds (VOCs) are low molecular weight hydrocarbons. An extremely important component of VOCs are BTEX compounds which contain benzene, toluene, ethylbenzene, o-xylene, and xylene isomers. BTEX hydrocarbons have a single loop aromatic structure that is caused by many natural processes or by human activities. BTEX has many applications in various industries such as oil, petrochemical, and automotive industries (1-3).

The US Environmental Protection Agency (EPA) and the US Food and Drug Administration (FDA) have identified benzene in the cancer group A1, which confirms its definitive carcinogenicity for humans (bone marrow and blood cancers). The EU has set the standard benzene concentration in open air at 5 μg/m^3, the American Conference of Governmental Industrial Hygienists (ACGIH) considers time-weighted average (TWA) of exposure to benzene to be 0.5 ppm, and the US Department of Health and Safety has set the exposure limit to about 1 ppm. The ACGIH also states the TWA exposure to toluene and xylene to be 50 and 100 ppm, respectively, while Occupational Safety and Health Administration (OSHA) considers exposure levels of about 200 ppm for toluene and 100 ppm for xylene. The ACGIH has classified toluene and xylene in the cancer group 3A, which confirms their potential for carcinogenicity and mutagenicity in humans or animals (4-7).

There are several methods, such as burning, encapsulating, stabilizing, and solidifying, etc. for removal of paint sludge, but removal of sludge contaminants by bioremediation is a flexible, inexpensive, and environmentally friendly method. In general, siliceous waste management involves physical, chemical, and biological methods. One of the biological processes (bioremediation) is fertilization and preparation of compost and vermicompost. Vermicompost is the result of a semi-aerobic process with about 80% moisture, which is carried out by a specific species of insects, fungi, bacteria, and actinomycetes. The types of earthworms that are commonly involved in the process of stabilizing organic wastes include: *Eisenia fetida* or red worm or compost worm, Dendrobaena veneta, Lumbricus rubellus, and Lumbricus terrestris (8-11). On the other hand, one of the problems of large refineries such as refineries in the automotive industry is the existence of a large amount of biological sludge, which is a good environment for growing *E. fetida* worms to produce vermicompost. Today, the use of biological sludge bed for the reproduction of worms has been studied and done in several cases. In the process of producing a composite of sewage sludge, earthworms are used as biological agents for the stabilization of organic matter. Since sewage sludge contains a large amount of organic and nutritious materials, it can be used as a suitable nutrient for feeding worms in the process of producing vermicompost (12,13).

Eisenia fetida is the most widely used worm in the vermicompost production. These worms are extraordinary worms, which are mostly used in the production of fertilizers from waste. Red worms endure different environmental conditions. There is no limit to the amount of the fertilizer and the number of worms. Their population is proportional to available food. Factors affecting the production of fertilizers by vermicompost with E. fetida include temperature, humidity, C/N ratio, pH, oxygen content, particle size, the content of volatile solids, and soil surface density (14,15).

Numerous studies have been conducted that show the ability of some types of earthworms to purify environmental pollutants such as heavy metals and a variety of organic matter. One of the most widely used species is *E. fetida*. Schaefer and Juliane reported that earthworms can activate the decomposition process of petroleum compounds in the vermicomposting process, therefore, they can be used in refining contaminated soils with medium concentrations (<4000 mg/kg) for total petroleum hydrocarbon (TPH). They also reported that the rate of decomposition of oil compounds depends on the species of earthworms. Schaefer and Juliane showed that increasing the catabolic activity caused by the presence of E. fetida resulted in the removal of 91% of hydrocarbon compounds with an infection concentration of 1074 mg/kg in 56 days of biological treatment by vermicompost along with biological wastes (16). The study of Takdastan et al on the biocomposting process of biological sewage sludge with the aim of regulating and neutralizing the risk of drilling in Khuzestan oilfield showed that the amount of TPH in excavation logs in Ahvaz oilfield is 42.004 g/kg, and in the course of 2 months of biocomposting process combined with biological sludge reached a concentration of 18.77 g/kg at a proportion of 1 to 1. According to the literature, sludge contaminated with toxic chemicals and other chemical compounds have been treated with worms, and these worms can reduce many toxic organic pollutants through the biological process of vermicomposting (17).

On the other hand, the growing number of cars in the country brings about the production of more industrial wastes such as sludge. Considering that BTEX compounds are an important part of the car paint sludge which have their own health hazards, careful measures should be taken for the management of these dangerous compounds. An analysis of the solvents used in the paint shops of the automotive industry shows that VOCs such as xylene, ethylbenzene, benzene, and toluene have the highest percentage in diluent thinner. A part of this VOC is directly introduced into the air and the rest enters the sludge. Based on this view, this research was done (18).

Materials and Methods

This is a pilot-scale experimental study (19). For this research, both samples of paint sludge and biological sludge were prepared from Saipa Automotive Company. Then, these samples were mixed with the ratios mentioned in a roofed warehouse with suitable temperature and humidity conditions. As this study was conducted in autumn and winter 2018 in the northern region of Iran, the temperature was set in the range of 18 to 25°C by a gas heater and water was sprayed to regulate humidity. Table 1 shows the mixing percentage of sludge (amount of paint sludge and biological sludge) in 27 samples. Based on similar articles in this study, 500 *E. fetida* worms were dispersed inside each box. A very small net was used to prevent the worms from escaping inside the boxes.

The number of worms was counted on days 1, 15, 30, 50, 70, and 90. In the optimal mixing ratio, the number of *E. fetida* worms increased from 500 in the first day to 980 on day 90. This suggests that *E. fetida* worms, with the help of microorganisms in biological sludge, are able to grow and reproduce increasingly in a mixed sludge environment contaminated with BTEX. Pilots F and G had the highest proliferation of worms and growth and activity of microorganisms. And, in pilots A, B, and C, the worms often disappeared. This is due to the threshold of tolerance of worms to sludge contaminants such as BTEX.

The sludge mixture was rotated for aeration three days per week in each pilot after starting the vermicomposting process. The time of vermicompost of paint sludge with biological sludge in all mixes was 90 days. The C/N ratio was low at the beginning of the vermicomposting process. Horticultural waste and straw were used to increase and adjust the C/N ratio within the range of 25 to 30.

Then, sampling was done at specified times. Random sampling from each box (three 25-g samples from each box with a diameter of less than 2 mm) was done using quadruple method on days 1, 15, 30, 50, 70, and 90. During the decomposition process, in addition to BTEX, the amount of heavy metals, pH, moisture content, the content of solids in sludge, C/N ratio, and temperature were also measured. BTEX was measured by GC-MS (NIOSH Method 1501). Different parameters such as humidity, organic nitrogen, organic carbon, dry solids, volatile and fixed solids, and pH were measured using standard methods (20-23). BTEX was measured in a trusted laboratory of the Environmental Protection Agency called Arman Mohit Pak Iranian. At specified times, 25 g of sample from each box was transferred to a trusted laboratory of the Environmental Protection Agency (Arman Mohit Pak Iranian) for BTEX measurement. The samples were analyzed after preparation of a gas chromatograph equipped with a flame ionization detector (FID) system. To determine the amount of BTEX, Agilent GC model 6890 made in USA with HP-5MS-5% phenyl silox column was used (mass spectrometer).

Results

The characteristics of the sludges used and their mixtures are given in Tables 2 and 3. Table 4 also shows the amount of BTEX in the Saipa paint sludge. Table 5 shows the reduction trend of BTEX in different pilots during 90 days.

The rate of change of the C/N ratio in vermicompost

Adjusting the C/N ratio is a critical factor in performing the composting and vermicomposting processes. Therefore, if its ratio is high in the mixed sample, its ratio should be reduced with substances such as sewage sludge and urine; and if its ratio is low, it should be increased with substances such as straw and other cellulosic materials. Due to the low C/N ratio of the prototypes tested in this study, the amount of C/N ratio was increased to about 27 with sawdust (24,25). The changes in the C/N ratio is shown in Figure 1. As shown in this figure, in all sludge mix treatments, the C/N ratio decreased during the study period (90 days). While in the three pilots A, B, and C with low biological sludge, the C/N ratio on day 90 was 23.2, 22.5, and 18.8, respectively, in the three pilots E, F, and G with the highest level of biological sludge, the C/N ratio decreased to 14.8, 14.3, and 13.5, respectively.

The decomposition of organic matter is generally done by microorganisms, especially bacteria, in the presence of worms. As shown in Figure 1, the initial C/N ratio in the seven pilots A, B, C, F, E, D, and G were 25.6, 26.7, 24.8, 27.2, 26.3, 27, and 27.9, respectively. At the end of the process, the C/N ratio in pilot A, B, and C with the lowest biological sludge, it reached 23.2, 22.5, and 18.8, respectively. And in the three pilots E, F, and G with the highest amount of biological sludge, the C/N ratio

Table 1. Characteristics of the pilots of vermicompost mixed sludge

Pilot	The amount of biological sludge (kg)	The amount of paint sludge (kg)	The number of pilot boxes
A	6 ± 0.1	6 ± 0.1	3
B	6 ± 0.1	3 ± 0.1	3
C	6 ± 0.1	1.5 ± 0.1	3
D	6 ± 0.1	1 ± 0.1	3
E	6 ± 0.1	0.750 ± 0.01	3
F	6 ± 0.1	0.600 ± 0.01	3
G	6 ± 0.1	0.375 ± 0.01	3
M	-	6 ± 0.1	3
N	-	6 ± 0.1	3

Table 2. Characteristics of the sludge used

Parameter	Paint sludge + SD	Biological sludge + SD
pH	8.3 ± 0.1	6.7 ± 0.1
Humidity (%)	21 ± 1.7	64 ± 3
Dry solids (%)	79 ± 2.6	36 ± 1.5
Volatile solids (%)	73 ± 3.65	87 ± 3.9
Fixed solids (%)	27 ± 1.2	23 ± 1.1
Nitrogen (mg/kg)	464 ± 11.3	3.7 ± 0.1
Carbon (mg/kg)	35000 ± 36	16.2 ± 0.3
C/N ratio	74 ± 1.3	16.6 ± 0.2

Table 3. The C/N ratio of sludge samples used

Treatment	Organic Carbon	Nitrogen	C/N
6 kg/6 kg	30.92	3.962	7.81 ± 0.1
6 kg/3 kg	29.71	3.95	7.52 ± 0.1
6 kg/1.5 kg	28.19	3.8	7.22 ± 0.1
6 kg/1 kg	26.21	3.81	6.92 ± 0.1
6 kg/750 g	23.62	3.76	6.33 ± 0.1
6 kg/600 g	22.93	3.7	6.25 ± 0.1
6 kg/375 g	21.61	3.61	6.01 ± 0.1
Biological sludge	19.81	3.51	5.65 ± 0.1
Pain sludge	35.11	0.464	75.55 ± 0.1

Table 4. The amount of BTEX in the paint sludge

BTEX Type	Xylene	Ethylbenzene	Toluene	Benzene
The Amount of BTEX+ SD (mg/kg)	151 ± 6.54	65 ± 2.21	12.6 ± 0.7	23.5 ± 1.01

Table 5. BTEX removal rate in different samples (mg/kg)

Pilot	Time					
	Day 1	Day 15	Day 30	Day 50	Day 70	Day 90
A	252.1± 5.4	244± 5.2	235± 4.6	226± 4.4	216± 4.2	199± 4.1
B	137± 1.7	130± 1.6	120± 1.4	181± 1.6	101± 0.9	86± 0.9
C	91± 0.9	82.5± 0.6	78.5± 0.4	71± 0.4	61± 0.2	50.8± 0.1
D	64± 0.9	59.8± 0.7	53.4± 0.7	45.8± 0.5	38.4± 0.4	32± 0.3
E	46± 0.6	40.8± 0.5	36.4± 0.4	31.6± 0.3	21.8± 0.2	17.9± 0.2
F	29.5± 0.4	22.6± 0.3	17.91± 0.2	11.5± 0.1	6± 0.05	8± 0.01.0
G	17± 0.2	12.9± 0.9	6.91± .05	4.9± 0.05	1± 0.01	5± 0.01.0

decreased to 14.8, 14.3, and 13.5, respectively. In other words, due to the high degradation of organic matter in pilots F and G, their carbon consumption was higher than samples in pilots A and B, therefore, the C/N ratio in these active samples decreased more.

pH changes in the vermicompost samples of mixed sludge

As can be seen in Figure 2, the pH of the mixed samples was initially alkaline, which reached a neutral level after the activity of the biological mass. Similar studies have suggested an optimal pH for the vermicompost activity to be 6 to 9. The main reason for the decrease in pH is the decomposition of organic matter by worms and bacteria and the production of CO_2 and the formation of intermediate acids (26,27).

Changes in the percentage of volatile solids in the vermicompost

Table 6 also shows the trend of significant reduction of VOCs, especially in samples in which the activity of worms and bacteria was higher. The optimum sludge mixing ratio of VOCs decreased from 80.4% to 37%.

The decomposition process of volatile organic solids in different pilots is shown in Table 6. The observation of the decomposition process in 90 days showed that the changes in the percentage of organic solids for the pilots, especially two pilots F and G, had a decreasing trend. In three pilots with low mixing of biological sludge including pilots A, B, and C, the amount of organic matter decreased from 60.6, 65, and 72.8 to 53, 49, and 42.4, respectively (a slight decrease). The main reason for which is very low biodegradation. It has been caused by microorganisms, possibly due to the escape of VOCs or BTEX in the paint sludge. The highest reduction of volatile organic matter occurred in pilots F and G; therefore, the highest reduction of BTEX occurred in the same pilots. This is due to the increased activity of bacteria, microorganisms, and worms in the biological sludge of wastewater, which increased the rate of decomposition and removal of organic matter. The increased growth of microorganisms and greater mobility of worms eventually led to further decomposition of organic solids and BTEX in the vermicompost. Bacteria, microorganisms, and worms, by feeding on organic matter in biologic sludge reduced them in the masses. As a result, it can be concluded that measuring the changes in the organic solids index is very important for this study because it shows the decomposition and removal of BTEX well (28).

Changes in BTEX in vermicompost samples

Figures 3 to 6 show the amount of BTEX in the bulk of vermicompost of mixed sludge. In the tested dye sludge, the amount of benzene, toluene, ethylbenzene, and xylene was reported as 23.5, 12.6, 65, and 151 mg/kg, respectively. As shown in Figures 3 to 6, in all sludge treatments, the amount of BTEX decreased during vermicomposting process. Due to the presence of a set of microorganisms, especially heterotrophic bacteria, biological sewage

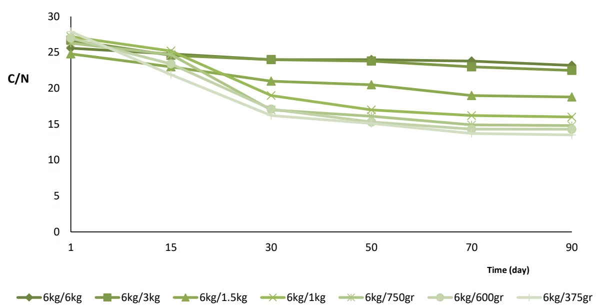

Figure 1. Changes in the C/N ratio in the vermicompost samples during destruction period.

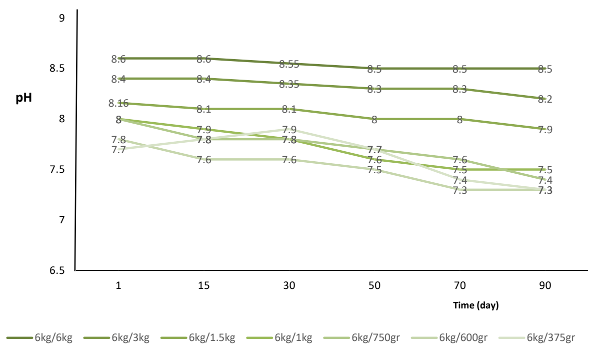

Figure 2. pH changes in the vermicompost samples during destruction period.

Table 6. Variation in the percentage of volatile solids in pilots during degradation period (mg/kg)

Pilot	Date (day)					
	Day 1	Day 15	Day 30	Day 50	Day 70	Day 90
6 kg/6 kg-A	60.5	58	56	54	54	54
6 kg/3 kg-B	66	62	59	52	50	49.9
6 kg/1.5 kg-C	73.8	65	61.5	53	44	42.9
6 kg/1 kg-D	76	65.8	59.5	52.6	44	41
6 kg/750 g-E	79.3	66	57	52	42	40.2
6 kg/600 g-F	80.5	66.5	54.8	50	38.9	36
6 kg/375 g-G	86.5	68.5	52	44.6	37	35.5

sludge has a very high potential for the decomposition of organic matters such as BTEX, which is considerably increased by the presence of *E. fetida* worms (29). According to the results of this study, E. fetida worms, along with microorganisms in the biological sludge, are able to function well and refine the pollutants when the proportion of the paint sludge to the biological sludge is 1 to 6. However, the best results were obtained when this ratio was 1 to 10, as shown by the figures. At this ratio, BTEX in the paint sludge was significantly degraded and reduced during the period from 30 to 90 days.

The total BTEX concentration in the paint sludge of Saipa was about 252.1 mg/kg, which was reduced due to mixing with biological sludge in the samples. For example, in sample F on the first day, BTEX was 29.5 mg/kg, which was completely decomposed on the 90th day. In pilots A, B, and C, the BTEX reduction was not significant due to the presence of a high proportion of paint sludge to the biological sludge of the treatment plant. However, in pilots E, F, and G, due to the mixing of more biological sludge with paint sludge and the lower initial BTEX, despite the combination of vermicompost worms and microorganisms, especially heterotrophic bacteria, the BTEX volume significantly degraded and reduced on day 90. As shown in Figures 3 to 6, it can be stated that the BTEX ratio of the samples in each of the pilots A, B, C, F, E, D, and G decreased from day one to day 90, but the highest reduction of BTEX occurred in pilot G (at the mixing ratio of 6 kg of biological sludge with 600 gr of paint sludge, i.e, the ratio of 10 to 1). At this ratio, benzene decreased from 3 mg to 0.01 mg. Toluene reached 1.5 mg over a period of 45 days. Ethylbenzene ranged from 7 mg to about zero over a period of 60 days, and xylene, over 90 days, ranged from 18 to about 0.9 mg (30-32).

Discussion

This study showed that BTEX was significantly reduced during the mixed sludge vermicomposting process. Therefore, in general, the results of this study are consistent with the results of other similar studies (33-40). However, the results of this study have some differences with those of some previous studies according to the method of work, bed characteristics, sludge type, treatment method, test conditions, study time, number and characteristics of earthworms and bed microorganisms.

Hosseini Panah and Takdastan concluded that the ratio of 2:1 mixing of oil-based drilling mounds with sludge biological vermicomposting has the highest TPHs decomposition efficiency compared to other percentages of waste mixing over a period of 60 days in this study (34). Amouei et al also reported that hazardous compounds such as heavy metals were significantly reduced by the composting process on soil in the North of Iran (35). Opuada Ameh et al also investigated the effect of earthworms (*Eudrilus eugeniae*) on the removal of organic

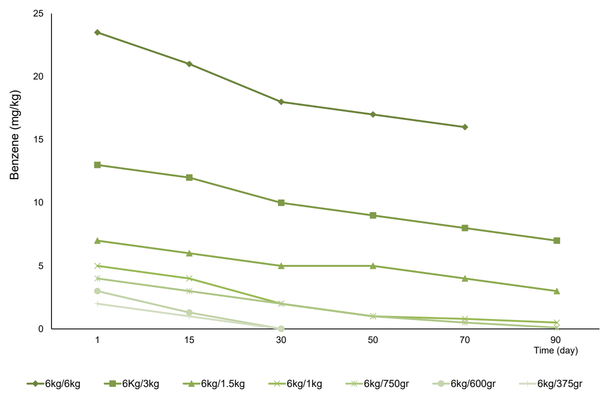

Figure 3. Variations of benzene concentration throughout the degradation time (mg/kg).

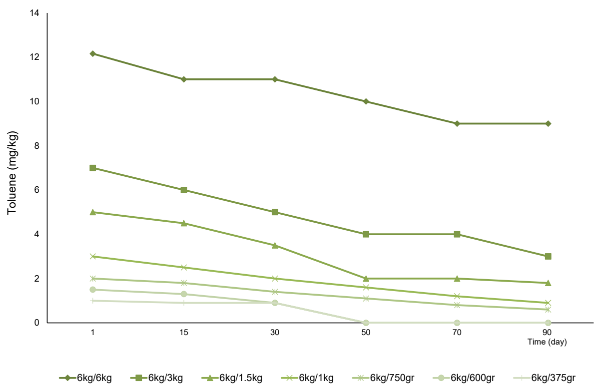

Figure 4. Variations of toluene concentration throughout the degradation time (mg/kg).

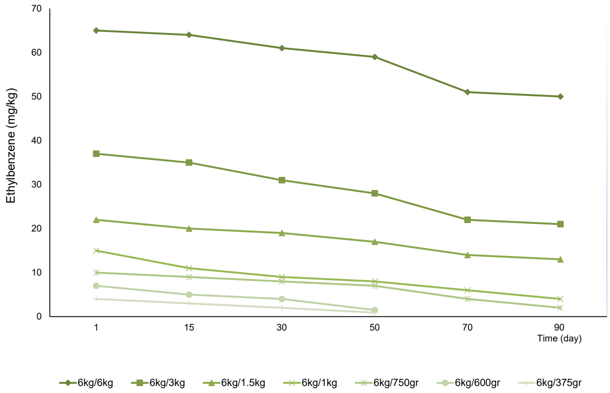

Figure 5. Variations of ethylbenzene concentration throughout the degradation time (mg/kg).

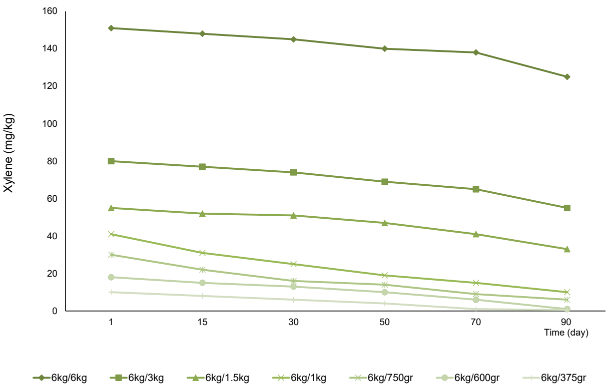

Figure 6. Variations of xylene concentration throughout the degradation time (mg/kg).

compounds in engine oil. They found that the rate of biodegradation of the pollutant increased with increasing the initial motor oil concentration. The concentration of engine oil used per kilogram of soil was 5, 10, 15, and 20 g, respectively, and the rate of reduction of hydrocarbon engine oil due to biodegradation with the earthworms was 16.91%, 20.82%, 34.68%, and 36.28%, respectively (36). Using 1000 earthworms (*E. fetida*) in hydrocarbon-contaminated soil, a study showed that not only the worms tolerated the toxic environment and survive, but they also significantly reduced toxic hydrocarbons. The hydrocarbons C10–C14 were reduced by 99.9%, the C15–C28 by 99.8%, and the C29–C36 by 99.7% by earthworms (37).

As the aim of this study was to investigate the removal of BTEX from the paint sludge of the automotive industry. This type of sludge in addition to BTEX also has other toxic substances, so compared to the mentioned studies, BTEX decreased in pilots when the larger volumes of biological sludge were mixed. In other words, this study showed that biological wastewater sludge with a ratio of 10 volumes along with paint sludge with a ratio of one volume, the best BTEX removal efficiency occurs compared to other mixtures over a period of 90 days. In fact, worms are unable to survive in large quantities in paint sludge environments and can only survive and reduce organic pollutants such as BTEX if they contain ten times the volume of biological sludge. Compared to similar studies on reducing BTEX paint sludge in the automotive industry, the time of the present research was longer and the amount of biological sludge required was higher. And it was revealed that earthworms in paint sludge are able to grow and reproduce as long as the level of contamination is not too high.

Conclusion

In this study, the rate of BTEX reduction of paint sludge in the automotive industry was investigated by the vermicomposting process mixed in different proportions with biological sludge from a wastewater treatment plant. Analysis of the results showed that the highest amount of BTEX removal occurs by mixing 6 kg of biological sludge with 600 g of paint sludge. That is, at the ratio of 10 to 1 BTEX, at which the sludge of Saipa Automotive Industries decreased significantly in 30 to 90 days. At this ratio, benzene mixture was reduced from 3 mg to less than 0.01 mg in 30 days while its volatility was 36%. Toluene decreased from 1.5 mg to zero over a 45-day period while its volatility rate was 30%. Ethyl benzene decreased from 7 mg to about zero in 60 days while its volatility rate was 23%. And xylene decreased from 18 mg to about 0.9 in 90 days while its volatility was 17%. At the same mixing ratio, the C/N ratio decreased from 27 on the first day to 13.5 on the 90th day. The amount of volatile solids or organic matter in the mass also decreased significantly. Therefore, it was found that *E. fetida* worm, along with microorganisms in biological sludge, is able to work in mixed sludge and has a very high ability to remove BTEX.

Acknowledgments

Professor Ghasem Ali Omrani is certainly known as the father of Iranian waste management science due to his efforts in the field of waste for many years. He is one of the first Iranian graduated student in the field of environmental engineering in Germany, who returned to Iran after gaining a lot of experience in the field of waste management and implemented many aspects of waste management for the first time in Iran. For about half a century, he has been a professor at the Faculty of Health of Tehran University of Medical Sciences and the Faculty of Natural Resources and Environment at Tehran University of Science and Research. Many professors in Iranian universities have been his students. All those who have benefited from the presence of this great master will certainly acknowledge the sincerity of his behavior and compassionate efforts for all students. He has authored numerous articles and books in the field of waste. This article has been done with the efforts and guidance of this great master during his lifetime, so the authors appreciate him and wish him rest in peace.

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Ethical issues

The authors certify that all data collected during the study are as presented in this manuscript, and no data from the study has been or will be published elsewhere separately.

Conflict of interests

The authors declare that they have no conflict of interests.

Authors' contributions

All authors have participated in the study design, literature search, writing of the manuscript, and data collection, analysis, and interpretation. All authors carefully reviewed, refined, and approved the manuscript.

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