[RETRACTED ARTICLE] USE OF A SONOCATALYTIC PROCESS TO IMPROVE THE BIODEGRADABILITY OF LANDFILL LEACHATE

[RETRACTED ARTICLE] A. Roodbari, R. Nabizadeh Nodehi, A. H. Mahvi, S. Nasseri, M. H. Dehghani, and M. Alimohammadi, Use of a sonocatalytic process to improve the biodegradability of landfill leachate," Braz. J. Chem. Eng. vol. 29 no. 2 p. 221 - 230, São Paulo Apr./June 2012, http://dx.doi.org/10.1590/S0104-66322012000200003.

This article has been retracted because it contains substantial overlap in both text and technical content with two other publications of the same authors:

(1) Mahvi AH, Roodbari AA, Nabizadeh Nodehi R, Nasseri S, and Dehghani MH, Alimohammadi M. (2012), Improvement of landfill leachate biodegradability with ultrasonic process. PLoS ONE 7(7): e27571.doi:10.1371/journal.pone.0027571,

(2) Mahvi Amirhossein, Roodbari Aliakbar, Nabizadeh Nodehi Ramin, Naseri Simin, Dehghani Mohammadhadii, and Alimohammadi Mahmood (2012) Improvement of Landfill Leachate Biodegradability with Ultrasonic Process. E-Journal of Chemistry, Volume 9 (2012), Issue 2, Pages 766-771. http://dx.doi.org/10.1155/2012/820971

Prof. Reinaldo Giudici
Editor-in-Chief, Brazilian Journal of Chemical Engineering

Brazilian Journal
of Chemical
Engineering

ISSN 0104-6632
Printed in Brazil
www.abeq.org.br/bjche

Vol. 29, No. 02, pp. 221 - 230, April - June, 2012

USE OF A SONOCATALYTIC PROCESS TO IMPROVE THE BIODEGRADABILITY OF LANDFILL LEACHATE

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(Submitted: July 24, 2011 ; Revised: October 22, 2011 ; Accepted: November 2, 2011)

Abstract - Landfill leachate is one of the most important sources of toxic organic compounds for ground and surface waters. Advanced oxidation processes (AOPs) offer an effective and environmentally friendly method for pretreatment of landfill leachates. In this study, an ultrasonic process was used for the pre-treatment of landfill leachate with the objective of improving the overall biodegradability, evaluated in terms of the BOD_5/COD ratio, up to a value compatible with biological treatment. Under optimized experimental conditions (pH of 10, power of 110 watts, frequency of 360 kHz, TiO_2 concentration of 5 mg/L and exposure time of 120 min), this method showed suitability for partial removal of chemical oxygen demand (COD). The biodegradability was significantly improved (BOD_5/COD increased from 0.210 to 0.786) which allowed an almost total removal of COD by a sequential activated sludge process.
Keywords: Landfill leachate; Biodegradability; ultrasound.

INTRODUCTION

Leachate consists of many different organic and inorganic compounds that may be either dissolved or suspended (Bila *et al*., 2005). These compounds pose a potential pollution problem for local ground and surface waters (Esplugas *et al*., 2004). Leachate from mature landfills is typically characterized by a high ammonium (NH_4^+) content, low biodegradability (low BOD_5/COD ratio) and a high fraction of refractory and large organic molecules such as humic and fulvic acids (Acgdacg and Sponza, 2005), but leachate from young landfills contains low organic compound concentrations (Li *et al*., 2010). Usually young landfill leachates are treated more easily as compared to the old one (Koh *et al*., 2004). Leachate is classified as stabilized (mature), intermediate, or fresh (young) given BOD_5/COD values of <0.2, 0.2–0.5, and >0.5, respectively (Robinson *et al*., 1983; Sun *et al*., 2010; Pilli *et al*., 2011). Some methods have been reported for the removal organics from landfill, such as biological treatment (aerobic, anaerobic and anoxic processes), physical methods (sedimentation, air stripping, adsorption, and membrane filtration) (Çeçen *et al*., 2003; Dehghani *et al*., 2007) and chemical methods (chemical precipitation (Lim *et al*., 2009; Kettunen *et al*., 1996), chemical oxidation (Zhao *et al*., 2010; Kurniawan *et al*., 2006), reverse osmosis, granular activated carbon (Peixoto *et al*., 2010), ion exchange

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resins and electrochemical oxidation (Bashir *et al*., 2010; Schuk *et al*., 1986)). In general, biological processes are preferred for the treatment of leachates with a high ratio of biochemical oxygen demand to chemical oxygen demand (BOD_U/COD) (Peixoto *et al*., 2009) and of fresh leachates containing mainly volatile fatty acids (Abdul Aziz *et al*., 2011). Compact physical and chemical methods have been suggested for the treatment of old, stabilized and dilute leachates with low biodegradability (Kurniawan *et al*., 2004; Kargi *et al*., 2004). Because of the variation in leachate composition and the wide range of pollutants contained in leachate, it is difficult to predict a treatment technique that will be effective for leachate (Atmaca, 2009; Mahvi, 2009). Usually a combination of physical, chemical and biological methods is used for effective treatment of landfill leachate, since it is difficult to obtain satisfactory results by using any one of those methods alone (Kurniawan *et al*., 2004). Traditionally, the degradation of organic compounds can be achieved by advanced oxidation processes (AOPs) (Anglada *et al*., 2011; Kargi *et al*., 2003). AOPs have been used to enhance the biotreatability of liquids containing various organic compounds that are non-biodegradable and/or toxic to common microorganisms (Chiang *et al*., 1995; Cortez *et al*., 2011). The ultrasonic process is one of AOPs and involves pyrolysis phenomena (thermal degradation) and the generation of the hydroxyl radicals (•OH), which has a very high oxidation potential and is able to oxidize almost all organic pollutants and volatile matter such as NH_3 (Mahvi *et al*., 2009). Although this process is very effective for complete mineralization of pollutants, it is applied as the only treatment process it will be expensive. However, if the ultrasonic process is used as a pre-treatment with other methods such as biological processes, its use can be optimized and be more useful for leachate treatment (Ben Yahmed *et al*., 2009; Lim *et al*., 2010). The objective of this study was to investigate the effect of the ultrasonic process as a pre-treatment for leachate biodegradability improvement.

MATERIALS AND METHODS

Materials

Samples of landfill leachate were obtained from a municipal landfill site (over 10 years old) located in Shahrood (Semnan, Iran). All leachate samples were collected from leachate lift stations or storage tanks, stored at 3°C, and tested within 2 days of collecting the samples. The main chemical characteristics of the raw leachate are summarized in Table 1. The ammonium concentrations were analyzed with a C_{203} 8 parameter test meter (Hanna Electronics Co., Ltd.). The pH was measured by a Benchtop pH Meter (Cole-Parmer Co., Ltd). The pH meter was calibrated before each use with pH 3, 7 and 10 buffer solutions. BOD and COD measurements were determined following Standard Methods 5210 and 5220, respectively. Reagents and standard chemicals were purchased from Hach Co., except for the BOD buffer solution, which was prepared according to Standard Method 5210. Ferrous sulphate ($FeSO_4{\cdot}7H_2O$), sulphuric acid and hydrogen peroxide (Merck, 30 wt. %) were of analytical grade. BOD check standards were performed with each batch of BOD measurements. The results were considered good when the value of the BOD check standard fell within the range of 198 ± 30.5 mg /L. The average ± standard deviation of the BOD check standards for the entire duration of the project was 169 ± 29.0 mg /L, which demonstrates good results given the inherent variability in BOD measurements. COD check standards were also performed with each batch of COD measurements. A COD standard solution of 1000 mg /L was diluted to 200 and 500 mg /L to ensure the accuracy of COD measurements. The relative difference for calibration check standards (RD_{cal}) is defined as the absolute difference of the check standard concentration and the known concentration all divided by the known concentration. The RD_{cal} for COD was <10% for the entire duration of the project.

Experimental Set-Up

As shown in Figure 1, for the laboratory experiments a cylindrical shaped Plexiglas reactor with total volume of 1 L was prepared. The solution in the reactor was mixed with a magnetic stirrer, while sufficient aeration was provided by a compressor connected to a porous stone located in the bottom of the reactor. The compressor was used to ensure a completely mixed condition in the reactor. The ultrasonic source was a Model UGMA-5000 ultrasound generator with three (30, 45 and 60 kHz) transducers having a titanium probe with 20 mm diameter. The power input could be adjusted continuously from 60 to 120 W. A leachate sample of 1000 ml was sonicated in a covered cylindrical glass vessel. Aeration was supplied by a Model SALWAT air compressor. The sludge was continuously aerated using aeration pumps.

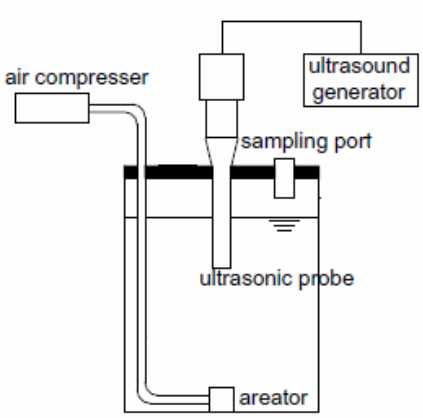

Figure 1: Schematic diagram of the reactor

Procedure

After the optimization by factorial design, the ultrasonic irradiation was applied in the treatment of raw leachates using a batch mode. At first, the raw leachate sample was filtered through filter paper ($0.45\,\mu$) to remove any suspended solid impurity. Then the sample was adjusted to the required pH with H_2SO_4 or NaOH. Then different scenarios were tested with regard to power intensities of 70 and 110 W, frequencies of 30, 45 and 60 kHz, reaction times of 30, 60, 90 and 120 minutes and pH of 3, 7 and 10. For determining the effect of catalyst on sonication efficiency, 5 mg/L of TiO_2 and ZnO have been used separately. To evaluate the effect of ultrasonic irradiation on the biodegradability of raw and sonochemically pre-treated leachates, BOD_5 and COD of both samples were measured.

Biological Procedure

The activated sludge system was applied in cylindrical aeration glass-vessels (30 cm internal diameter and 4 cm in height). The system was aerated by using air pumps placed at the bottom of the reactors. The initial volume of the culture was 150 mL which was completed to 300mL with substrates (leachate, pre-treated leachate or glucose) at the beginning of each cycle. The pH was controlled by a probe and adjusted to 7.0 by using H_2SO_4 or NaOH. The oxygen concentration was monitored by using an O_2 probe, located at the top of the reactor. All the experiments were carried out in duplicate and at room temperature (20–25°C) for periods of 72 h. For COD determinations, samples (10 ml each) were taken every 12 h, after they had been centrifuged and filtered through a 0.45 mm Millipore filter (De Morais and Zamora, 2005).

RESULTS AND DISCUSSION

Characterization of the Raw Landfill Leachate

The main chemical characteristics of raw leachate are summarized in Table 1. When the biodegradability ratio (BOD_5/COD) is lower than 0.25 and the pH near to 8, the samples can be considered to be moderately stabilized leachates (Wang *et al.*, 2009). In most cases, intensive and sophisticated physico-chemical processes are necessary for the treatment of mature leachates.

Table 1: Chemical characteristics of the studied landfill leachates

Parameters	Values	Parameters	Values
COD	5691±83	pH	7.9-8.1
Calcium	1061±0	Magnesium	8.65
BOD_u	738	NH_3-N	726±25
BOD_5	16.6	Alkalinity as $CaCO_3$	3650±123
TOC	36±20	TS	1420±29
BOD_5/CO	0.21		

Values (except pH) in mg /l

Effect of the Sonocatalytic Process on the Biodegradability of Leachate

The results indicated that the sonocatalytic process can improve leachate biodegradability (BOD_5/COD ratio). The BOD_5/COD ratio for raw leachate was 0.210, but it reached 0.786 (with TiO_2) and 0.783 (with ZnO) after sonication. Independent Samples T-test showed that there is significant difference between the BOD_5/COD ratios of raw leachate and pretreated leachate with the sonocatalytic process (p_{value} =0.000 for both TiO_2 and ZnO). The results indicated that the system operates with great efficiency (BOD_5/COD ratio=0.786) at pH 10, power of 110 watts, frequency of 60 kHz and TiO_2 concentration of 5 mg/L.

Effect of the Ultrasound Power Input on Biodegradability Improvement

Figure 2 shows the effect of power input on leachate biodegradability for TiO_2 and ZnO. As shown in this figure, the power input clearly improves biodegradability. Independent Samples T-test (Table 2) showed that there is a significant difference between the BOD_5/COD ratios of raw leachate and pretreated leachate at different powers. (p_{value} =0.000 for both TiO_2 and ZnO).

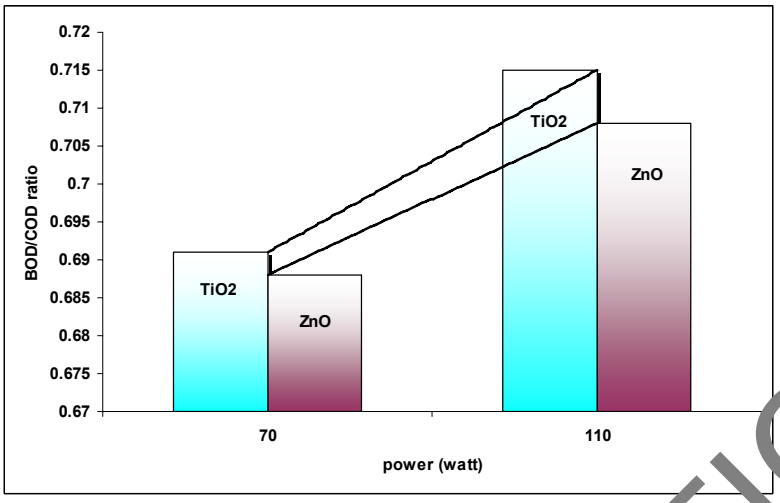

Figure 2: BOD_5/COD ratios for TiO_2 and ZnO at different power inputs (Frequency=30 kHz, concentration=5mg/L, pH=3)

Table 2: Results of Independent Samples Test for power inputs

		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
PERCENT of reduction with TiO_2	Equal variances assumed	41.3	0.000	-10.686	70	0.000	-0.043361	0.004058	-0.051454	-0.035268
	Equal variances not assumed			-10.686	47.26	0.000	-0.043361	0.004058	-0.051523	-0.35199
PERCENT of reduction with ZnO	Equal variances assumed	28.5	0.000	-9.651	70	0.000	-0.038556	0.004020	-0.046573	-0.030538
	Equal variances not assumed			-9.591	4.77	0.000	-0.038556	0.004020	-0.046643	-0.30538

According to sonochemistry theory, when the ultrasound intensity reaches or exceeds the cavitation threshold, bubbles will be formed easily and the cavities collapse violently. Increasing the ultrasonic power will increase the energy of cavitation, lowering the threshold limit of cavitation, and enhancing the quantity of cavitation bubbles (Kurniawan and Lo, 2009). In other words, at higher intensities, the concentration of hydroxyl radicals and mass transfer are higher, which leads to more degradation of organic material (Chianese *et al*., 1999) and also more biodegradable intermediate compounds. The efficiency of ratio improvement then increases with the increase of ultrasonic intensity.

Effect of Exposure Time on Biodegradability Improvement

Results indicated that the exposure time somehow improved biodegradability. Figure 3 shows the effect of exposure time on leachate biodegradability for TiO_2 and ZnO. A One-Way ANOVA test (Table 3) showed that there is no significant difference between the BOD_5/COD ratios of raw leachate and pretreated leachate with sonocatalytic process at different exposure times (p_{value}=0.467 for TiO_2 and 0.398 for ZnO).

Effect of Frequency on Biodegradability Improvement

Results indicated that the frequency clearly improve biodegradability. Figure 4 shows the effect of frequency on leachate biodegradability for TiO_2 and ZnO. A One-Way Anova test (Table 4) showed that there is significant difference between the BOD_5/COD ratios of raw leachate and pretreated leachate with the sonocatalytic process at different frequencies (p_{value} =0.000 for both TiO_2 and ZnO). Results of the Tukey statistical test (Table 5) also showed that there are significant differences between frequencies of 30 and 60 kHz (p_{value} =0.000) and between 45 and 60 kHz (p_{value} =0.000).

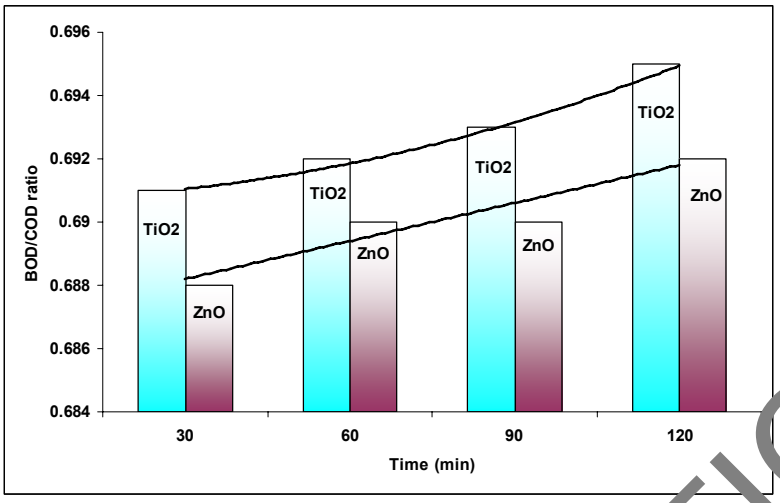

Figure 3: BOD_5/COD ratios for TiO_2 and ZnO at different exposure times (Frequency=30 kHz, concentration=5mg/L, pH=3)

Table 3: Results of One-Way ANOVA test for different exposure times

	Sum of Squares		df		Mean Square		F		Sgi	
	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO
Between Groups	0.002	0.002	3	3	0.001	0.001	1.00	0.859	0.398	0.467
Within Groups	0.052	0.045	68	68	0.001	0.001				
Total	0.055	0.047	71	71						

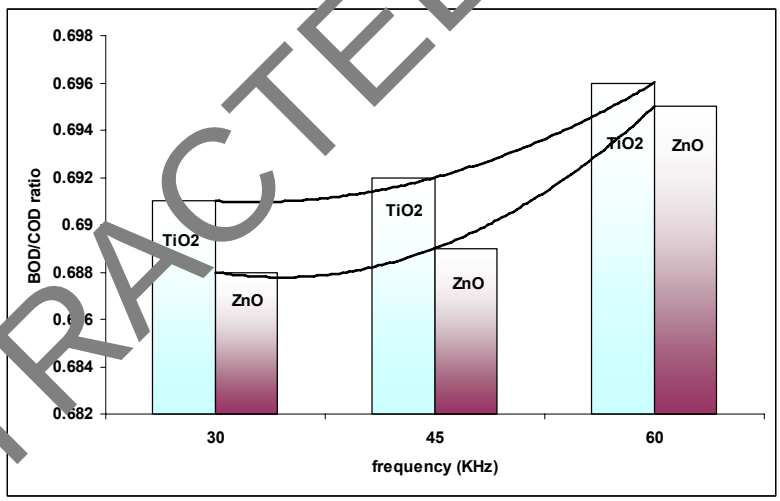

Figure 4: BOD_5/COD ratios for TiO_2 and ZnO at different frequencies (power=70watt, concentration=5mg/L, pH=3)

Table 4: Results of One-Way ANOVA test for different frequencies

	Sum of Squares		df		Mean Square		F		Sgi	
	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO
Between Groups	0.013	0.012	2	2	0.006	0.006	10.718	11.818	0.000	0.000
Within Groups	0.042	0.035	69	69	0.001	0.001				
Total	0.055	0.047	71	71						

Table 5: Results of Tukey test for different frequencies

(I) frequency	(J) frequency	Mean Difference (I-J)		Std. Error		Sig.		95% Confidence Interval			
								Lower Bound		Upper Bound	
		With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO
30	45	-0.0112	-0.0095	0.007	.0060	0.261	0.311	-0.028	-0.025	0.005	0.006
	60	-0.0323	-0.0309	0.007	0.006	0.000	0.000	-0.049	-0.046	-0.015	-0.015
45	30	0.01120	0.00958	0.007	0.006	0.261	0.311	-0.005	-0.006	0.028	0.025
	60	-0.0211	-0.0213	0.007	0.006	0.011	0.005	-0.038	-0.036	-0.004	-0.005
60	30	0.0323	0.03091	0.007	0.006	0.000	0.000	0.015	0.015	0.049	0.046
		0.0211	0.02133	0.007	0.006	0.011	0.005	0.004	0.005	0.038	0.036

Effect of pH on Biodegradability Improvement

Results indicated that the pH somehow improves biodegradability. Figure 5 shows the effect of pH on leachate biodegradability for TiO_2 and ZnO. A One-Way ANOVA test (Table 6) showed that there is no significant difference between the BOD_5/COD ratios of raw leachate and pretreated leachate with the sonocatalytic process at different pHs (p_{value} =0.503 for TiO_2 and 0.170 for ZnO).

Effect of Type of Catalyst on Biodegradability Improvement by Ultrasound

Figure 6 shows the effect of the type of catalyst on the leachate biodegradability (BOD_5/COD ratio). Results showed that the effects of the two catalysts on leachate biodegradability were similar but an Independent Samples T-test (Table 7) indicated that there is no significant difference between the BOD_5/COD ratios of raw leachate and pretreated leachate with TiO_2 and ZnO (p_{value} = 0.287).

Concurrent Effect of Power Input and Frequency on Biodegradability Improvement by Ultrasound

As mentioned above, power and frequency were effective parameters (statistically significant) for biodegradability improvement by the sonocatalytic process. The Univariate statistic test (Table 8) showed that estimates of the size of the effect were 88.6 % for power and 74.9 % for frequency but the concurrent effect was smaller (44.4 %).

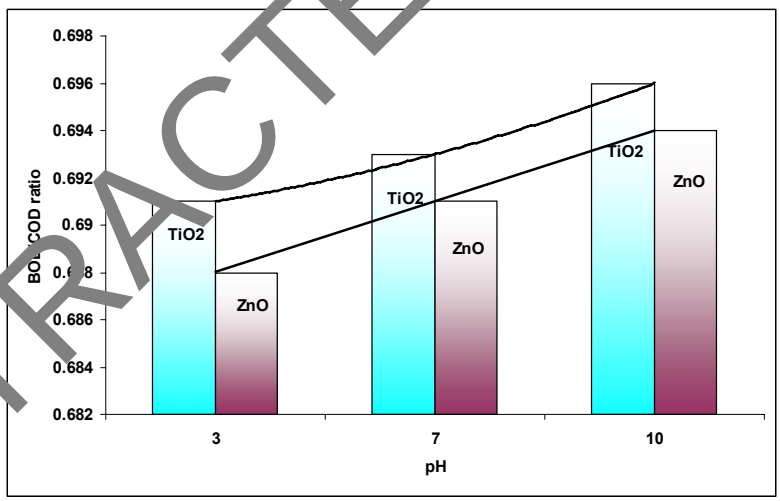

Figure 5: BOD_5/COD ratios for TiO_2 and ZnO at different pH_S (power=70watt, concentration=5mg/L, frequency=30 kHz)

Table 6: Results of One-Way ANOVA test for different PHs

	Sum of Squares		df		Mean Square		F		Sgi	
	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO	With TiO_2	With ZnO
Between Groups	0.001	0.020	2	2	0.001	0.001	0.693	1.821	0.503	0.170
Within Groups	0.054	0.045	69	69	0.001	0.001				
Total	0.055	0.047	71	71						

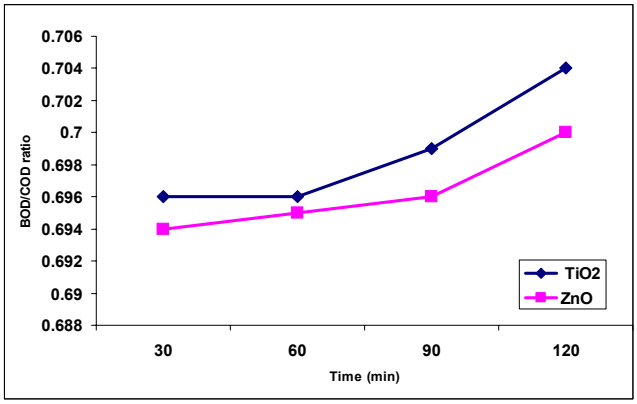

Figure 6: BOD_5/COD ratios for TiO_2 and ZnO (power=70watt, concentration=5mg/L, frequency=30 kHz)

Table 7: Results of Independent Samples Test for two catalysts

		Levene's Test for Equalityof Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
PERCENT of reduction	Equal variances assumed	0.571	0.451	1.068	142	0.287	0.00476	0.00446	-0.00405	-0.01358
	Equal variances not assumed			1.068	141.22	0.287	0.00476	0.00446	-0.00405	-0.01358

Table 8: Results of Univariate statistical test for concurrent effect of power and frequency

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	0.050	5	0.010	152.896	0.000	0.921
Intercept	37.673	1	37.673	5.731E5	0.000	1.000
POWER	0.03	1	0.034	514.876	0.000	0.886
FREQUENCY	0.0	2	0.006	98.421	0.000	0.749
POWER * FREQUENCY	0.0	2	0.002	26.380	0.000	0.444
Error	0.004	66	6.573			
Total	37.	72				
Corrected Total	0.055	71				

Biodegradability Change During Ultrasonic Decomposition

Initially, the biodegradability of the leachates was evaluated through the evolution of the BOD_5/COD ratio. For untreated samples, this parameter has values of about 0.2, while ultrasonic treatment for 120 min permits its enhancement up to values near 0.786, which represents substantial biodegradability according to the current literature (Bae *et al*., 1999; Pi *et al*., 2009). This result indicates that the ultrasonic process can break down or rearrange molecular structures of organic matter and convert the non-biodegradable organics to more biodegradable forms. This is a fact of remarkable importance in the case of the application of an integrated chemical–biological system to leachate treatment (De Morais and Zamora, 2005). In general, it is admitted that an ultrasonic process can transform recalcitrant organic compounds into easily biodegradable products, improving the efficiency and reducing the cost of further biological steps. In a second phase, raw and pre-treated leachate was submitted to a biological degradation process using a sequential batch reactor. The evolution of the COD during the biological treatment (Figure 7) confirms the low biodegradability of raw leachates, which achieve a maximal COD removal of about 30% at 72 h of treatment. On the other hand, the COD of pre-treated leachates decreases progressively, attaining COD removal higher than 90% at the end of the 72-h cycle. Additionally, the use of ultrasonically pre-treated samples favored the preservation of the physical characteristics of the biological sludge, which could be corroborated by the measurement of traditional physical parameters and microscopic observation.

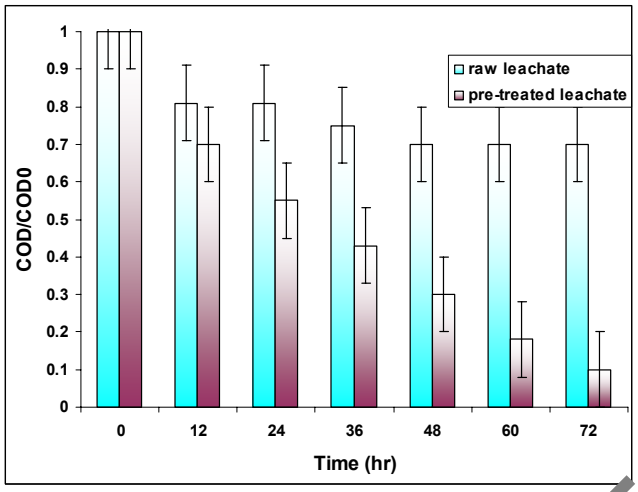

Figure 7: Evolution of the COD during biological treatment of the leachates (sample pretreated with a power of 110 watts, frequency of 60 kHz, pH of 7 and 5 mg/L of TiO_2 for 120 minutes).

CONCLUSION

Landfill leachates contain some macromolecular organic substances that are resistant to biological degradation. With very low biodegradability ratios (BOD_5/COD), usually lower than 0.21, these complex matrixes show a recognized resistance to conventional activated sludge systems. When applied as a relatively brief pre-treatment, the sonocatalytic process induces several modifications of the matrix, which results in a significant enhancement of its biodegradability. For this reason, the integrated chemical–biological systems proposed here represent a suitable solution for the treatment of landfill leachate samples with an efficient remediation of the relevant parameter (COD).

ACKNOWLEDGEMENTS

Authors would like to thank the research office of Tehran University of Medical Sciences.

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