

Removal of dibutyl phthalate (DBP) from landfill leachate using an ultrasonic field

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ABSTRACT

The work detailed here investigated the efficiency of ultrasonic technologies in the treatment of landfill dibutyl phthalate (DBP) leachate, where this substance is a known as endocrine disrupting compound (EDC). Investigated effects took in such operating parameters as ultrasonic amplitude, solution pH, time, and type of leachate. Results confirmed effective DBP decomposition under the influence of ultrasonic waves, which have oxidative properties over a wide range of pH values, in relation to physicochemical substance properties. In this process, natural pH proved to offer optimal conditions. Reaction time has an impact on the results, but the oxidation is slow. Ultrasonic degradation of DBP of 33.4%, 37.6%, and 54.6% is achieved within 15, 30, and 60 min. respectively. The results also demonstrated that chemical oxygen demand, total organic carbon, total nitrogen, ammonium nitrogen (N-NH₄⁺), and total phosphorus removal is not effective by ultrasonic methods. Additionally, ultrasonic degradation was found to depend on the amplitude of the waves used. A change of amplitude from 20% to 50% resulted in a yield increase of about 10%. More effective oxidation was also achieved during treatment of stabilised leachate. An ultrasonic field is, therefore, a suitable means of removing organic micropollutants from leachate, to a degree that conventional purification methods cannot achieve.

Keywords: Micropollutants; Dibutyl phthalate; Landfill leachate; Ultrasonic waves

1. Introduction

In municipal engineering, one of the main problems is the treatment of leachate arising on landfills as well during the dehydration of fermented sewage sludge at sewage treatment plants. Choice of an appropriate method to purification of this leachate proves difficult, mainly given varying compositions and amounts that arise. In addition, leachate is highly polluted by anthropogenic organic compounds mostly resistant to decomposition. Substances such as polycyclic aromatic hydrocarbons (PAHs), pesticides, phthalates, etc., are very dangerous for the environment and human health on account of their genotoxic, mutagenic, and carcinogenic properties [1–10].

Phthalates are commonly used plasticizers added to plastics to increase their flexibility, transparency, durability, and longevity. They can be found as common additives in cosmetics, paints, lubricants, and packaging. The global production of phthalate plasticizers is an estimated 3 million tonnes per year, though enhanced awareness of their harmfulness has been reflected in declining production in recent years. The EU's Commission Directive 2007/19/EC imposes restrictions on the presence in the environment of dibutyl phthalate (DBP), butyl benzyl phthalate, Di(2-ethylhexyl) phthalate, diisononyl phthalate, and diisodecyl phthalate [1,11].

An important source of phthalates in landfill leachate is the emission of phthalate esters from plasticizers and cosmetics. In such leachate, concentrations of the esters

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as high as 18 mg/L can be detected. As a reflection of its higher solubility, DBP is one of the phthalates found most commonly in an aqueous environment, especially at land-fills [7]. While disposal of leachate is important for the protection of the environment, in particular groundwater and surface waters, it proves very difficult on account of the composition. However, the introduction of landfill leachate into the sewer system or the environment needs to follow treatment or at least pre-treatment, and purification makes use of physical, chemical, physico-chemical, and biological methods [4,12,13].

Among the promising physicochemical methods are advanced oxidation processes (AOP). It is a combined method based on chemical solutions that are mainly applied, including AOPs. In AOPs, a reaction with a hydroxyl radical generated in the reaction medium is encouraged, through the application of hydrogen peroxide, ozone, an ultrasonic field, UV radiation, and catalysts such as Fe^{2+} or TiO_2 . These methods are sensitive and selective, and implementation is shown to result in the formation of simpler molecules susceptible to biodegradation [14,15]. However, the complexity of chemical composition of leachates requires multistage, physicochemical, and biological systems [14,16].

Sonochemical process is clean, and does not generate additional wastes, and is hence regarded as an environmentally beneficial technology particularly recommended in the pre-treatment of leachate. Over the past several years, ultrasound has been applied effectively as an emerging AOP for a wide variety of pollutants in wastewater treatment. A growing number of studies have demonstrated that ultrasound irradiation results in a rapid and effective decomposition of micropollutants in aqueous solution. The main advantage is that the ultrasound process does not require added chemicals, oxidants or catalysts, and does not generate additional waste streams as compared with other processes (such as ozonation and adsorption). A downside is the rather slow rate of ultrasonic degradation achievable. However, several factors, including ultrasonic frequency, solution pH, and addition of hydrogen peroxide, may influence the sonochemical degradation of organic contaminants [17,18].

The work described here thus sought to investigate the efficiency of ultrasonic waves in removing DBP from landfill leachate. The variables in the study in question were pH, reaction time, amplitude of ultrasonic waves, and type of leachate.

2. Materials and methods

2.1. Characteristics of the landfill

An active landfill located at Kozodrza, SE Poland, (50°06'38N, 21°37'10E) was selected for the investigation of leachate properties. This site (of about 200.000 m²) has been in operation since 1990, mainly accepting solid municipal wastes from areas of Podkarpackie Voivodeship (with its ca. 2,000,000 inhabitants). Landfill leachate samples were collected and analysed in September 2017 from two tanks. First collects leachate (LT1) from eight cells that were being filled up to 2008. Second is in turn fed by six landfill cells in which wastes continue to be dumped (LT2).

2.2. Reagents and materials

Standard (1 mg/mL) solutions of DBP (catalog number: 36736) and benzyl benzoate (catalog number: 68183) were obtained from Sigma-Aldrich (St. Louis, USA). Ultra-pure water was obtained from Purix CNX-100, while solid phase extraction (SPE) cartridges and C18 sorbents were purchased from SiliCycle Inc. (Quebec City, Canada), and HCl (catalog number: 575327160) and NaOH (catalog number: 810935163) solutions from POCH (Gliwice, Poland). Analytical grade dichloromethane (catalog number: 628414194), methanol (catalog number: 621993194), and acetone (catalog number: 102485197) were also purchased from POCH. All glasswares were submerged in NaOH solution for 24 h, rinsed with ultrapure water, and dried for 5 h. A further rinse with acetone was applied prior to heating at 280°C for 5 h.

2.3. Methods

2.3.1. Sample collection and analysis

The samples were collected into labelled clean bottles rinsed thrice before being filled. The pH and electrical conductivity (EC) were recorded on site at the time of sampling, using a digital pH meter and digital EC meter, respectively. The samples were then transported immediately to the laboratory in cooler boxes at a temperature below 5°C. Collected leachate was stored in a tinted glass bottle with zero headspace in a refrigerator at 4°C. TOC and TN were determined using a TOC-V_{CPN} analyser (Shimadzu, Japan). total phosphorus (TP) and N-NH4+ were determined colorimetrically (Aquamate, Thermo Spectronic, UK): TP in non-filtered medium after mineralisation with H₂SO₄ and peroxodisulfate, as phosphate using the molybdate method, and N-NH₄⁺ using Berthelot's reaction. Concentrations of heavy metals were determined by ICP-EOS (Quantima, GBS, Australia) whereas PAHs were determined using a GC-MS system coupling Trace GC ULTRA with TriPlus to an ITQ 1100 (Thermo Scientific, Waltham, USA) based on PN-ISO 18287 (initial temperature 60°C held for 2 min, increasing temperature ramp by 30°C/min to 120°C, increasing temperature ramp by 5°C/min to 300°C held for 15 min, inlet temperature 260°C, injection volume 1 µL, transfer line temperature 295°C). COD, BOD₅₇ and suspension were determined using wellknown standard methods.

2.3.2. Preparation of model solutions

Model solutions of DBP were produced at concentrations in landfill leachate of 1 mg/L, by adding specific amounts of a standard solution and mixing with an electromagnetic stirrer. Concentrations of DBP resembling those found in landfill leachate were thus achieved. In this study, model solutions were prepared using two type of leachate – stabilised (LT1) and young (LT2).

2.3.3. Use of ultrasonic waves

Ultrasonic irradiation experiments were conducted at bench-scale in a reactor comprising an ultrasonic processer, reactor cell and water ice bath. The ultrasound source was a SONOPULS HD 3200 Bandelin ultrasonic processor (Berlin, Germany), equipped with a 1.3 cm-diameter titanium probe tip. The homogenizer operated at 20 kHz, with 200 W nominal power. Irradiation with ultrasonic waves at an amplitude of 20% and 50% was applied, with the ultrasonic process conducted for 15, 30, and 60 min, in respect of 200 mL volumes of prepared samples poured into the glass reactor. The effects of time, pH, amplitude and type of leachate on efficiency were investigated. Sonication was terminated by neutralization of the sample, and the TOC was measured. The efficiency of the oxidation processes was estimated by measuring TOC values. The efficiency of DBP removal was determined using gas chromatography. All batch experiments were performed in triplicate.

2.3.4. Solid phase extraction

SPE cartridges were activated using 6 mL methanol, followed by 6 mL of ultra-pure water, before loading samples. After activation, 20 mL aqueous samples were passed through the SPE cartridges. They were then dried in vacuum for 20 min to remove retained water. The analytes were eluted with 6 mL solutions of methanol and dichloromethane (5:1, v/v), before samples were made subject to concentration. Sample concentration entailed evaporation of the solvent with nitrogen using multiple-sample XcelVap evaporators (Salem, New Hampshire).

2.3.5. Chromatographic analysis of DBP

Analysis of DBP was performed using a GC flame ionization detector system (Shimadzu, Kyoto, Japan). A ZB-5MSplus Zebron Capillary GC Column (analytical column) of 30 m × 0.25 mm × 0.25 μ m was used. Hydrogen and synthetic air served as the carrier gases. The temperature program was initial temperature 60°C held for 1 min, increasing temperature ramp by 20°C/min to 270°C and then held for 5 min, the total time being 16.5 min, inlet temperature 280°C, injection volume 1 μ L, detector temperature 250°C. Quantification was performed using the internal calibration method based on a seven-point calibration curve. Benzyl benzoate was used as the internal standard for the quantification of DBP, which was achieved using calibration curves for which the correlation coefficients all exceeded 0.99. Recoveries for DBP resulted in values in the range 88%–110%.

3. Results and discussion

Physical and chemical analysis showed high concentrations of pollutants in the raw leachate samples (Table 1) indicating the importance of this effluent being treated prior to discharge into bodies of water. Studied landfill leachates are dark liquids with a strong odour carrying high organic and inorganic loads. Characteristics of landfill leachate nevertheless depend on type and moisture content of the waste deposited, site hydrogeology, age of the landfill, seasonal weather variations, composition of waste, dilution with rainfall, and degree of decomposition within the landfill [19].

Values obtained for the leachate from tank LT2 were significantly higher than from LT1. The chemical oxygen demand (COD) of LT2 was $3,728 \text{ mg O}_2/\text{L}$, total organic carbon (TOC) was 2,665 mg C/L, and biochemical oxygen demand

Table 1

Characteristics of leachate from studied landfill.

Parameter	LT1	LT2
pН	8.35	8.45
Conductivity, mS/cm	11.06	22.8
TOC, mg C/L	941.4	2,665.5
BOD ₅ , mg O ₂ /L	262	1,371
COD, mg O ₂ /L	1,944	3,728
Suspension, mg/L	106	180
Total PAHs, mg/L	< 0.005	0.039
Cu, mg/L	0.012	0.029
Zn, mg/L	0.120	0.16
Pb, mg/L	< 0.010	< 0.010
Cd, mg/L	0.0018	< 0.0005
Cr⁴⁺, mg/L	< 0.010	< 0.010
Hg, μg/L	< 0.0010	< 0.0010
N-NH ₄ ⁺ , mg/L	97.05	94.04
TN, mg/L	564.1	1,413
TP, mg/L	6.12	92.43
BOD/COD	0.13	0.37

LT1, stabilised leachate; LT2, young leachate; TOC, total organic carbon; BOD₅, biochemical oxygen demand; COD, chemical oxygen demand; N-NH₄⁺, ammonium nitrogen; TN, total nitrogen; TP, total phosphorus.

 (BOD_5) amounted to 1,371 mg O₂/L. In turn, in LT1, COD was of 1,944 mg O₂/L, TOC 941 mg C/L and BOD₅ 262 mg O₂/L. LT2 was richer in total nitrogen than LT1: 1,413 vs. 564 mg N/L, whereas ammonium nitrogen was noted at a similar level of about 95 mg N/L in both cases. Total phosphorus was higher in LT2 (92.43 mg P/L) than LT1 (6.12 mg P/L). Neither tank produced leachate with higher metal concentrations. However, very high conductivity values were observed, reflecting high concentrations of inorganic compounds dissolved in the effluent.

Landfill leachate is commonly characterised by its high loading with micropollutants. In the raw LT1 leachate, the total PAH concentration remained just under 0.005 mg/L, while in LT2 it was at 0.039 mg/L. Encouraging results were also reported by Klauck et al. [8] who found 28 organic compounds in raw leachate. Among these, eight substances are listed by the US Environmental Protection Agency (US-EPA) [20] as potential endocrine disruptors. According to Kjeldsen et al. [21], around 200 organic compounds have been detected in landfill leachate. Due to its complexity, the leachate has a high potential for contaminating both the soil surface and groundwater, so it should be tested appropriately prior to its disposal.

A typically high ratio of BOD/COD beyond 0.1 combines with a slightly alkaline pH and high ammonium nitrogen values to characterise this as a leachate from a stabilised landfill. In the analysed leachate from LT1, the value of BOD₅/COD ratio was 0.13, encouraging the idea that the landfill is stabilised. In such a case, biological treatment itself would not be effective, because leachate contains compounds not biodegraded readily. In turn, in line with its composition, LT2 must be seen as leachate from an unstabilised site, given that this is commonly characterised by higher BOD and COD concentrations, and by a high BOD/COD ratio exceeding 0.37. The changes in parameters, for example, COD, TOC, pH, conductivity, total nitrogen, ammonium nitrogen and TP were determined following sonification (Table 2; Figs. 1(A)–(D)). It was mainly the effects of the ultrasonic process in removing DBP in the treatments that were investigated. The results demonstrated that COD removal using ultrasonic waves is not effective. The efficiencies of removal of COD ranged between 0.6 (natural pH, time 30 min, amplitude 20%) and 4.3% (pH 10, time 30 min, amplitude 20%),

with the initial value 1,944 mg/L for LT1, and 0.54% for LT2 (natural pH, time 30 min, amplitude 20%). According to Ahmed and Lan [9], the reduction of COD in leachate from stabilised landfill is a huge challenge for conventional treatment plants, due to the presence of recalcitrant substances and toxic contaminants, requiring several operational steps and large systems for even moderate results to be obtained.

TOC removal efficiency reached about 20% with an initial TOC value of 941.4 mg/L in the case of LT1, but only 3.7%

Table 2 Parameters of the solutions after sonication

Parameters	pH after process		Electrical conductivity (mS/cm)		Suspension (mg/L)			
	LT1	LT2	LT1	LT2	LT1	LT2		
рН	Time 30 min, amplitude 20%							
3	3.12		14.76		161			
8	8.35	8.41	10.95	23.6	89	151		
10	9.82		11.73		160			
Time (min)	Natural pH, amplitude 20%							
15	8.68		10.70		92			
30	8.37	8.47	10.92	23.7	89	158		
60	8.55		10.94		76			
Amplitude (%)	Natural pH, time 30 min							
20	8.31	8.51	10.96	22.3	95	162		
50	8.55		10.83		84			

LT1, stabilised leachate, LT2, young leachate.



Fig. 1. Effect on the removal of TOC and COD of (A) pH (LT1, time 30 min, amplitude 20%), (B) time (LT1, natural pH, amplitude 20%), (C) amplitude (LT1, natural pH, time 30 min), and (D) type of leachate (natural pH, time 30 min, amplitude 20%).

for LT2. Ultrasound waves were not found to be efficient at TN, N-NH₄⁺, and TP removal. Solution pH and conductivity also proved stable during the sonication processes. After processes, suspension values for natural pH were lower.

Following leachate treatment using an ultrasonic field a decrease in DBP values was observable (Figs. 2(A)-(D)). In ultrasonic treatment, the degradation efficiency of DBP is affected by pH. Data in Fig. 2(A) show clearly how the efficiency of degradation of DBP is pH dependent, with a best efficiency of about 38% obtained at natural pH. The mechanism of sonochemical degradation of organic pollutants is usually based on the formation of short-lived radicals generated in violent cavitation events. By itself, pH has little influence on the phenomenon of cavitation, though it could affect the reactions of transient species escaping from the bubble. A compound's pK value plays an essential role in ultrasonic processes. Compounds with modifications of the ionic form will be affected by pH changes because their molecular or ionic form will not present the same hydrophobicity. Depending on pH, the ionic form will degrade less rapidly than the molecular form, as it is more soluble than the uncharged structure and therefore has lower accumulation in the interface area [22-24]. Other researchers also reported a marked influence of solution pH on the sonochemical degradation of organic compounds. In a study by Villaroel et al. [25], results for the removal of acetaminophen from water showed that ultrasonic degradation in an acidic (pH 3.0-5.6) medium is greater than can be obtained in basic aqueous solutions (pH 9.5-12.0). In turn, Jiang et al. [26] investigated the idea that the rate of sonochemical degradation of 4-nitrophenol is reported to decrease with increasing pH, while the destruction of aniline is favoured by an alkaline solution.

In further experiments, a natural pH value (8.37) and lower value for amplitude (20%) were mainly chosen for economic reasons. Continuing the process, a change of pH and increased ultrasonic amplitude does not increase productivity significantly, and thus proves uneconomical.

In the second step, DBP degradation was examined with various durations ranging from 15 to 60 min in optimising conditions. Ultrasonic degradations of 1 mg/L concentrations of DBP equal to 33.4%, 37.6%, and 54.6%, respectively, were obtained within 15, 30, and 60 min. It was noted that the usual rate of destruction of DBP by ultrasound waves is slow. Ultrasonic treatment has been known as an attractive advanced technology for the removal of various contaminants. However, the use of these technologies in degrading complex organic contaminants does not always result in complete mineralisation [27]. Ultrasonic technologies degradation of DBP was assessed under amplitudes of 20% and 50%, at a frequency of 20 kHz. The results suggest increased degradation with amplitude increasing in the 37.6%-46.2% range. An increase in ultrasonic amplitude may increase the number of active cavitation bubbles and the production of hydroxyl radicals. Similar results have been reported in the literature [22,28–30].

Optimum efficiency of removal of DBP from stabilised and unstabilised leachates is as shown in Fig. 2(D). As the figure indicates, removal efficiency was greater for stabilised leachate LT1 than for LT2. The lower efficiency in the latter case can be explained in terms of the presence of greater amounts of organic and inorganic contaminants. These compounds are well known as hydroxyl-radical scavengers.



Fig. 2. Effect on the removal of DBP of (A) pH (LT1, time 30 min, amplitude 20%), (B) time (LT1, natural pH, amplitude 20%), (C) amplitude (LT1, natural pH, time 30 min), and (D) type of leachate (natural pH, time 30 min, amplitude 20%).

4. Conclusions

The treatment of landfill leachate poses a major complex wastewater treatment challenge in today's society. The research conducted shows that ultrasonic oxidation is efficient at removing DBP from both stabilised and unstabilised landfill leachates. However, the removal of COD, TOC, TN, N-NH₄⁺, and TP was only achieved at a low level. This indicates a possibility of ultrasound being used as a stage in effluent treatment that mainly removes organic micropollutants. Achievable efficiency here relates to several variables, namely pH, amplitude as well as type of leachate and treatment time. This all connects with the formation of OH•, which is, for example, dependent upon pH, as well as concentrations of organic and inorganic compounds. In all of this, it needs to be recalled that landfill leachates going without proper treatment can represent a potential hazard to the environment.

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