Degradation kinetics of di-(2-ethylhexyl) phthalate (DEHP) and organic matter of sewage sludge during composting

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Abstract

The potential degradation of di-(2-ethylhexyl) phthalate (DEHP) and organic matter of sewage sludge by composting was investigated using laboratory reactor at different operating conditions (E-1, E-2 and E-3). In all conditions, single stage thermophilic phase was observed within 2 days and almost, 60% of DEHP was degraded under this phase. At the end of composting, total DEHP degradation of more than 85% was observed in all conditions and total carbon reduction was 11.8% in E-1, 7.6% in E-2 and 10.8% in E-3. Similar trend was observed in the degradation of total nitrogen. The reduction of volatile solids (VS) in the composting reactors was 5.4% in E-1 (18 days), 5.5% in E-2 (12 days) and 4.3% in E-3 (18 days). The degradation kinetics of DEHP in thermophilic phase (including initial mesophilic phase) and the phase there after were determined by first order and fractional power kinetics, respectively. The significance of experimental parameters in DEHP degradation was assessed by Pearson correlation approach. Elevated temperature produced during composting was effective for the rapid degradation of DEHP from sewage sludge compared to mesophilic treatment.
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Keywords: Di-(2-ethylhexyl) phthalate; Composting; Kinetics; Reactor study

1. Introduction

Di-(2-ethylhexyl) phthalate (DEHP), a non-reactive plasticizer used in the manufacture of plastics, which increases the flexibility and workability of rigid polymers such as polyvinyl chloride (PVC) matrix [1]. It is characterized by very low water solubility (0.6–400 μg/L at 20 °C) and high octanol–water partition coefficient (log K_{ow} of 7.54) [2]. DEHP is not chemically bound to polymeric matrices and can mobilize from plastics. Owing to its stability and lipophilic nature, DEHP accumulates in fish tissues and tends to adsorb into suspended solids (SS) in surface and subsurface waters [3]. DEHP is characterized under the category of endocrine disrupting chemicals (EDCs) and it is regulated under the list of environmental priority pollutants [4].

DEHP was frequently detected in municipal sewage sludge and its concentration in dewatered sludge cake was more than the recommended value (100 mg/kg) by European Union (EU)

for land application [5]. The sewage sludge conserves abundant nutrients and hydrocarbons and hence, the application of municipal sewage sludge in agriculture has been receiving considerable attention. However, the presence of DEHP in sewage sludge along with heavy metals, organic pollutants and pathogens reduces the feasibility of land application [6]. In order to avoid or minimize the possible risks caused by sewage sludge, USEPA and EU formed stringent regulations for land and agricultural applications of sewage sludge.

Changes in public perception and legislation reduced the likely disposal of sewage sludge by incineration and landfilling [7] and in many European nations, ocean disposal of sewage sludge was banned. On the other hand, aerobic composting is the biological decomposition and stabilization of organic substances in the presence of oxygen under the thermophilic conditions created by biologically produced heat [8]. Composting favors the disappearance of larger amounts of biodegradable contaminants by the activity of micro-flora throughout the duration of composting. Biodegradation is an attractive alternative for the degradation of phthalate esters, and the rate of DEHP biodegradation was accelerated under thermophilic

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Table 1
Biodegradation of DEHP in composting processes

Reference	Composting method	Feeding materials (bulking agent)	Organic pollutants	Degradation
Hartlieb et al. [29]	In-vessel composting (1.8 m^3)	Municipal biowaste (shredded shrubbery)	DEHP, pyrene and simazine	86% after 370 days
Moeller and Reeh [27]	In-vessel composting (10 L, insulated)	Municipal solid waste (garden park waste)	DEHP (38–160 mg/kg dry wt in MSW compost)	96–99% after 25 days
		Sewage sludge (straw and garden park green waste)	DEHP (3–6 mg/kg dry wt. in sewage sludge compost)	91% after 25 days
Marttinen et al. [7]	Compost bins (220 L)	Raw municipal sewage sludge	DEHP (60 µg/g dry wt.)	58% after 85 days
	Rotary drum (5 m^3)	Anaerobically digested municipal sewage sludge (tree bark and peat)		34% after 85 days
Amir et al. [3]	Platform (semi-industrial and industrial trials)	Lagooning sludge (straw)	Phthalic acid esters (DEHP concentration 6.26 mg/kg)	91% after 180 days
		Activated sludge (grass)	Phthalic acid esters (DEHP concentration 28.67 mg/kg)	94% after 135 days

sludge digestion process [9]. Some of the DEHP biodegradation studies by composting are shown in Table 1. It was reported that aerobic composting under elevated temperature is effective in the biodegradation of organic pollutants [10]. Besides, composting under well-controlled condition is effective in the stabilization of organic matter present in the sewage sludge [11].

The main objective of the present study was to investigate the stability, and degradations of DEHP and organic matter present in the sewage sludge during composting. In addition, the study was focused to investigate the degradation kinetics of DEHP and the significance of various parameters on DEHP degradation during pilot scale composting.

2. Materials and methods

2.1. Feeding materials

Dewatered sludge cake (combined primary and secondary sludges, pre-thickened and dewatered in a belt filter press without digestion) was collected from Nei-Hu municipal sewage treatment plant (MSTP) located in the Northern Taiwan, which has a treatment capacity of 240,000 m^3/day. Sawdust was used

as a bulking agent and also to adjust the moisture content of the feeding materials. The characteristics of the feeding materials and the experimental conditions are shown in Table 2. The initial C/N ratio of three experiments was in the range of 15–30 and the moisture content was controlled at $60 \pm 2\%$ (initially) in all experiments. The porosity, bulk density and pH were maintained uniformly in all experiments.

2.2. Bioreactor

A static reactor (BK 365 Komposter) with a capacity of 110 L was used for the composting experiments (Fig. 1). The feedstocks were mixed thoroughly as per the designed proportion and fed into the reactor in equal layers. Representative samples were collected at various depths of the reactor, homogenized in a static shaker and analyzed for various parameters. After feeding, the reactor was kept permanently on a digital balance (least count ± 100 g) and the weight of the reactor was recorded throughout the experiment. The reactor was maintained in aerobic condition using an air pump and the air was supplied at a rate of 10 L/min from the bottom of the reactor.
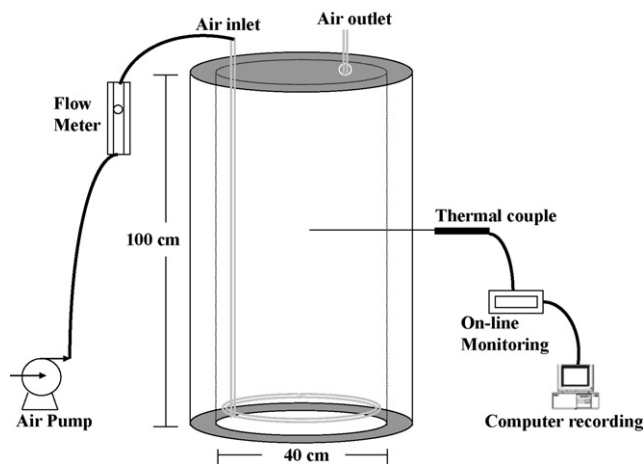
Table 2
Characteristics of the feedstocks and the experimental conditions

Parameters	E-1	E-2	E-3	Sludge cake[a]	Sawdust
Porosity (%)	35	32	31	31	67
Bulk density (g/cm^3)	0.49	0.38	0.41	0.68	0.15
pH	6.4	6.4	6.1	7.1	5.1
Moisture content (MC, %)	62	58	60	81	23
Mix ratio of sludge cake and sawdust[b]	1:0.25	1:0.5	1:1		
C (g/kg of TS)	383.67	428.84	407.32	370.64	398.14
N (g/kg of TS)	26.20	15.75	13.69	39.92	0.77
C/N	15	27	30	9	518

[a] Initial DEHP concentration in the sewage sludge is 333 mg/kg.
[b] Wet weight basis under the calculated moisture content of 60%.

Fig. 1. Schematic diagram of the composting reactor.

2.3. Acclimation and composting experiments

Prior to the composting reactor study, two acclimation experiments (A-1 and A-2) were carried out using the bioreactor as shown in Fig. 1. In A-1, the feedstocks were mixed in the similar ratio as that of reactor E-1 (Table 2) with a moisture content of around 60%, however, sewage sludge itself was used as a seed in A-1 to start up the microbial activity. The experiment was continued until the reactor temperature equalized with ambient temperature. After the completion of A-1, a part of compost (nearly, 1/10, w/w) was recycled to A-2 as a seed and the experiment was repeated as described A-1. The compost obtained after two-acclimation experiments, weighing exactly 1/10 (w/w) was used as seed in each compost reactor and the reactor studies (E-1, E-2 and E-3) were carried out under different operating conditions (Table 2). The samples were collected from each composting reactor at every 12 h interval and analyzed for various parameters.

2.4. Analytical techniques

At every 12 h intervals the samples were collected from various depths of the reactor and homogenized in a static shaker. The samples were analyzed for moisture content (MC), volatile solids (VS), pH, water-soluble organic carbon (TOCw), total Kjeldhal nitrogen (TKN) and ammonia nitrogen [12]. For the determination of pH and water-soluble fraction, the sample was mixed with deionized water (1:10, w/v) and extracted for 1 h using a shaker, centrifuged for 30 min, and filtered using a 0.45 μm membrane filter. A wet oxidation method (O-I-Analytical Model 1010) was used to analyze TOCw. The total carbon (TC) and total nitrogen (TN) in the samples were determined by elemental analysis (EA).

2.5. Extraction and measurement of DEHP

Prior to the analysis of DEHP containing real sludge and compost samples, DEHP standard solutions were spiked on quartz sand (less matrix effect)/clean sludge (without DEHP) and the DEHP spiked samples were extracted by Soxhlet extraction method [13]. The percentage recoveries of DEHP from the quartz sand and sludge (for 100 mg/kg) was 101.48% and 90.74%, respectively ($n = 3$).

For real sludge and compost samples analysis, three grams of oven-dried sample was mixed with 150 mL of n-hexane/dichloromethane in a ratio of 1:1 (v/v) and extracted by Soxhlet extraction method. The extracts were further concentrated to less than 5 mL and analyzed in a HP-6890 gas chromatograph fitted with 5973 MSD system (Agilent) and 30 m × 0.25 mm DB-5 capillary column (J&W Scientific, Folsom, CA, USA). Exactly, 1 μL of the sample was injected through an auto sampler in splitless mode. The temperatures of the injector and detector were maintained at 260 °C and 280 °C, respectively. The mass spectrometer was operated at 70 eV and scanned from 30 to 425 amu at 1 scan/s. At these operating conditions, the retention time of DEHP was obtained at 24.62 min.

3. Results and discussions

3.1. Evolution of composting temperature

Measurement of temperature in the composting matrix during active phase gave an adequate real time indication of the establishment of ideal conditions that support microbial degradation (Fig. 2). In the acclimation step (A-1), the temperature was increased to 70 °C within 2 days, and persisted for relatively
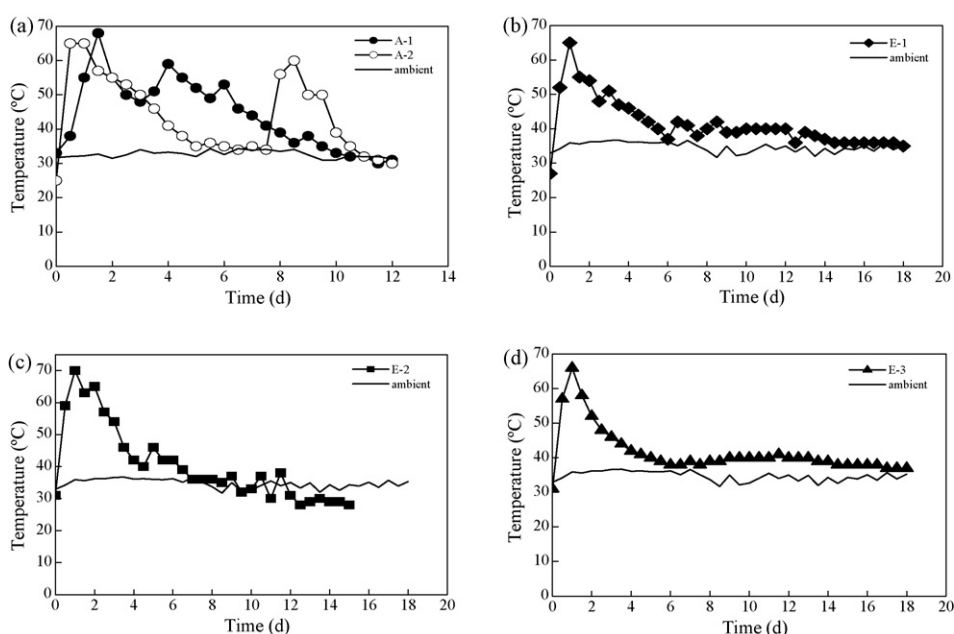

Fig. 2. Temperature profile during (a) acclimation process and composting in reactors (b) E-1 (c) E-2 and (d) E-3.

shorter period. Thereafter, the temperature was decreased drastically and reached the ambient temperature after 8 days. After completion of A-1, a part of compost from A-1 was seeded and the second stage acclimation (A-2) was conducted at similar operating conditions. In A-2, the temperature was increased rapidly compared to A-1 without much lag-phase (Fig. 2a). Moreover, the thermophilic phase in A-2 was prevailed more than 24 h compared to A-1. At the end of 8 days, a second stage thermophilic phase was observed in A-2. Following to the acclimation experiments, composting reactor studies (E-1, E-2 and E-3) were conducted at different operating conditions as shown in Table 2. The temperature profiles in E-1 to E-3 are shown in Fig. 2b–d, respectively. In all composting reactor studies, single stage thermophilic phase was observed within 2 days. The rapid rise in temperature is due to the self-heating of organic matter as a result of microbial respiration. Also, it indicates that the feedstocks composition is capable of supporting aerobic microbial respiration [14,15]. The thermophilic temperature prevailed in all composting experiments was significant to germinate the most pathogen in the compost.

3.2. Organic matter, carbon and nitrogen fractions

The contents of total carbon (TC) and nitrogen (TN) decreased with composting time appreciably in thermophilic phase ($t \leq 3$ days) owing to the higher microbial activity. The variation of TC and TN during composting is shown in Fig. 3. After thermophilic phase ($t > 3$ days), the degradation rate of organic matter was reduced considerably. Carbon loss during composting is attributable to the mineralization of organic matter resulting in the evolution of CO_2 and heat [6,16,17]. The reductions of TC in E-1, E-2 and E-3 were significantly higher in thermophilic phase compared to mesophilic phase. At the end of composting, the TC reduction was 11.8% in E-1, 7.6% in E-2 and 10.8% in E-3. It was reported elsewhere that operating the bioreactors at higher temperature (thermophilic phase) offers better degradation of organic compounds and improves the solubility of some organic compounds such as fats, oils and grease [9]. This reason can be attributed to the increase in reduction of TC in the thermophilic phase of composting. The change in TN of the composting mixture with time in all composting
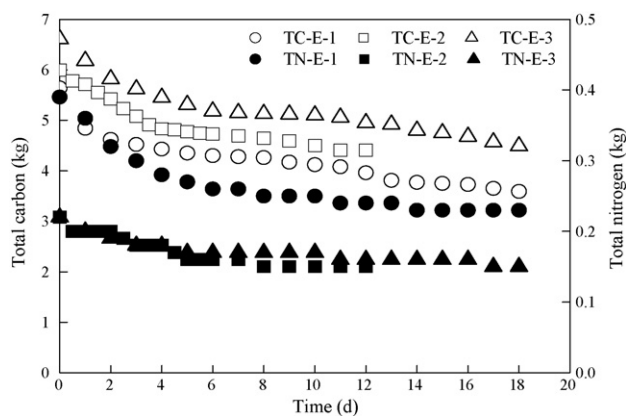

Fig. 3. Variation of total carbon and total nitrogen content during composting.

experiments followed the profile of TC reduction. At the end of composting, 18.8%, 14.4% and 9.4% of TN reductions were observed in E-1, E-2 and E-3, respectively. The rate of TN reduction in thermophilic phase of E-1 was more compared with other two runs (Fig. 3) that can be attributed to the low initial C/N ratio adopted in E-1. The nutrient value and nutrient balance of sludge compost can be determined either by the ratio of carbon and nitrogen content [18] or by monitoring the profile of volatile solids (VS) in the system. At the end of composting, the reductions of VS were 5.4% in E-1 (18 days), 5.5% in E-2 (12 days) and 4.3% in E-3 (18 days) (Table 3).

3.3. Water-extractable organic carbon and nitrogen

The changes of water-soluble carbon and nitrogen in the composting experiments are shown in Fig. 4a and b. Under thermophilic condition, a drastic increase of TOCw was observed due to the transfer of organic matter into biofilm. A noticeable concomitant effect was observed in the values of TOCw (Fig. 4a) and temperature (Fig. 2). The concentration of TOCw in E-1 (thermophilic phase) was comparatively more than E-2 and E-3. However, the concentration of TOCw was declined quickly after the thermophilic phase in all composting experiments. These results are in good correlation with VS and TC reductions (Fig. 3) in the thermophilic phase of composting.

Table 3
Physicochemical characteristics of the reactor contents before and after composting

Experiment	VS	TC	TN	C/N	TOC$_w$	TKN$_w$	NH$_4{}^+$–N$_w$	DEHP
E-1								
Initial	820.40	383.67	26.20	15	196	114	65	296
Final	775.82 (5.4%)	338.26 (11.8%)	21.27 (18.8%)	15	256	138	111	44 (85%)
E-2								
Initial	883.36	428.84	15.75	27	232	101	54	250
Final	834.48 (5.5%)	396.14 (7.6%)	13.47 (14.4%)	29	242	129	95	32 (87%)
E-3								
Initial	917.28	407.32	13.69	30	114	47	28	213
Final	880.30 (4.3%)	363.38 (10.8%)	12.40 (9.4%)	30	57	33	20	26 (88%)

All calculations reported in the table are based on dry weight of reactor contents and the data presented in parenthesis are removal percentages. The units of table contents: VS, TC and TN are g per kg of TS; TOC$_w$, TKN$_w$ and NH$_4{}^+$–N$_w$ are mg per L; and DEHP is mg per kg of TS.
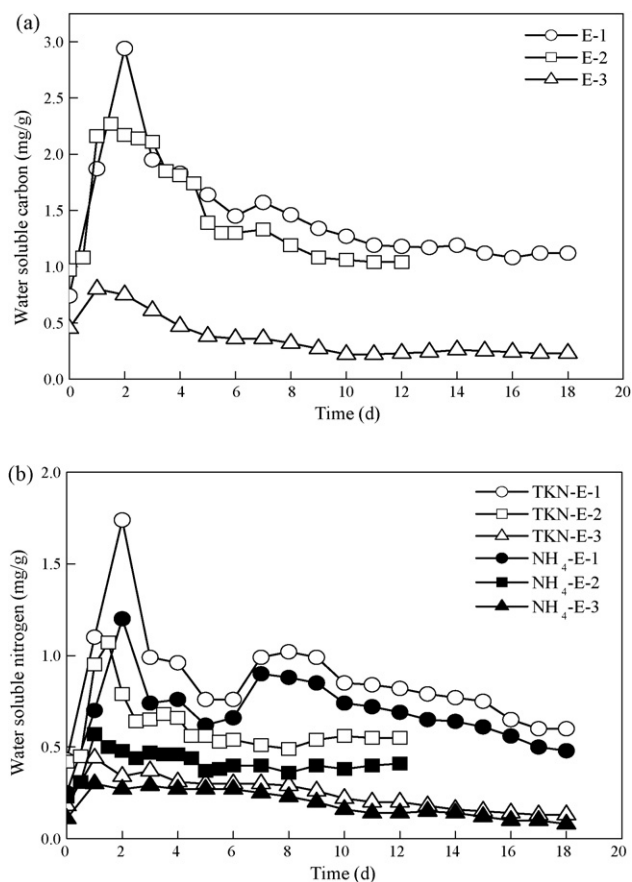
Fig. 4. Water-soluble fractions of (a) carbon and (b) nitrogen during composting.

Similar trend was observed in the concentration of water-soluble nitrogen in all experiments (Fig. 4b). The initial increase in NH₄–N and TKN concentrations can be due to the mineralization of organic nitrogen (proteins) in the aerobic thermophilic treatment that caused the release of nitrogen in the form of ammonium and/or ammonia. In addition, the degradation and/or transformation of nitrogenous matters increased the pH value during composting [11]. Due to the increased temperature and pH in the thermophilic phase of composting, the solubility of ammonia gas was decreased and stripped off along with the air stream [9]. Therefore, the reduction of ammonium nitrogen was envisaged after 2 days (Fig. 4b). This can also be attributable to nitrogen stripping in the form of ammonia and the utilization of this nutrient by the process culture. At the end of 18 days, the decrease to near-zero concentration of NH_4^+–N was observed in E-1 whereas only a considerable reduction of NH_4^+–N was observed in both E-2 and E-3.

3.4. Degradation of DEHP

The degradation profile of DEHP in all composting experiments is shown in Fig. 5. The total removals of DEHP at the end of composting were around 85% in E-1, 87% in E-2 and 88% in E-3. However, most of DEHP was degraded under thermophilic phase (66% for E-1, 78% for E-2 and 60% for E-3), which has persisted for 3 days. At the end of this period, DEHP concentra-
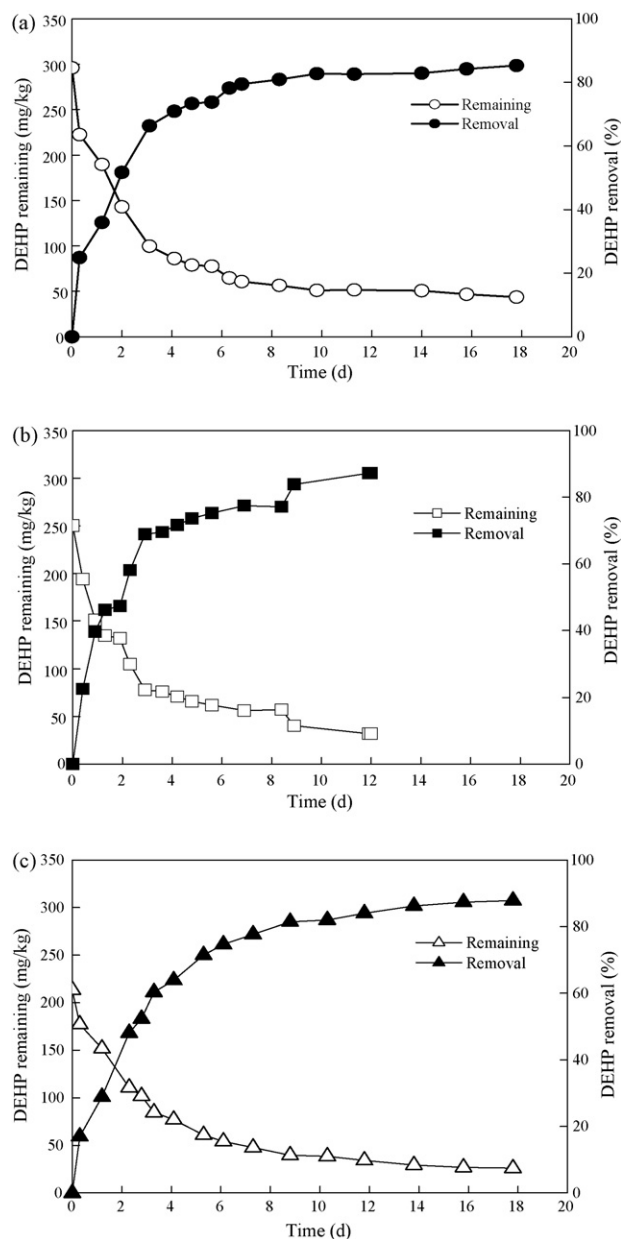

Fig. 5. Percentage removal of DEHP in various composting reactors (a) E-1 (b) E-2 and (c) E-3.

tion was reduced to less than 100 mg/kg, which is well below the standard set by EU for the land application of DEHP containing sewage sludge. The experimental results show that the highest reduction of DEHP was achieved at the highest temperature. Furthermore, the reduction of DEHP was in good correlation with the reductions of VS and TC in all three experiments. The overall rate of DEHP reduction in the present investigation was high compared to the DEHP degradation reported in sludge-amended soils [19]. These findings reflect that higher temperature generated during composting has shown to be effective in the rapid degradation of DEHP compared to mesophilic treatment.

Metabolic breakdown of phthalate esters (PAEs) by microorganisms is considered as one of the major routes of environmental degradation for these widespread pollutants [20].

Table 4
Kinetics of DEHP degradation

Experiment	Time (day)	Temperature prevailed in experiments (day)		Rate constant in phase-I[a]		Model parameter value in phase-II[a]	
		Phase-I	Phase-II	k (day^{-1})	r^2	K	r^2
E-1	0–18	0–3	4–18	0.37	0.96	0.52	0.93
E-2	0–12	0–3	4–12	0.40	0.93	0.82	0.89
E-3	0–18	0–3	4–18	0.27	0.97	0.97	0.99

[a] Rate constant and model parameter calculated using first order kinetics [19] and fractional power kinetics [25], respectively.

Owing to the low vapor pressure and thermal-stability of DEHP, volatilization may not occur in environmental systems or in other words, loss of DEHP caused by physicochemical pathway was insignificant compared to biodegradation [1,21]. From our earlier investigation, it was observed that loss of DEHP from the sterilized sludge cakes was negligible after the completion of 100 days of aeration experiments [5]. The PAEs with shorter side alkyl chains are relatively water soluble, i.e. DBP and DEP, and readily biodegradable under aerobic and anaerobic conditions. Amir et al. [3] reported that DEHP underwent hydrolysis or de-esterification step to form monoesters (MEHP), and Phthalic acid was produced after the biotransformation of MEHP. The rates of MEHP degradation were of the same order as those of DEHP, meaning that de-alkylation and de-esterification were the major biodegradation mechanisms [3]. However, the degradation intermediates of DEHP were not quantified at this stage of our study and the investigations are in progress to identify the intermediates of DEHP.

3.4.1. Kinetics of DEHP degradation

The first-order kinetic model was used in most case to describe the behavior of DEHP degradation in biological treatments under aerobic or anaerobic conditions [1,22–24]. However, a simple first-order model is insignificant to describe the degradation of PAEs especially in long-term treatment trials, such as sludge-amended soil and composting. Poor regression values ($r^2 < 0.6$) were often obtained when a simple first-order kinetic model was used to describe whole degradation behavior with two distinct degradation phases. Roslev et al. [19] and Madsen et al. [25] proposed an empirical biphasic model with two independent kinetic expressions to describe the two distinct phases of DEHP degradation in sludge-amended soil. The initial DEHP mineralization activity was well described by first-order kinetics and the mineralization of long-term trials (>40 days) was described by fractional power kinetics [19,25].

The first order and fractional power kinetics were used in this study to describe the behavior of DEHP biodegradation in thermophilic phase (including initial mesophilic phases) (Phase-I) and the phase thereafter (Phase-II), respectively. The fractional power kinetic model parameters, i.e. K and N were calculated by Eqs. (1–3), and derived from a plot of $\log (C/C_0)$ versus $\log (t)$. The half time ($t_{0.5}$) of DEHP degradation in phase-I and II was calculated using first-order and fractional power kinetic equations (Eq. (3)), respectively. The degradation rate constant of active-phase (Phase-I) and the model parameters of stabilization-phase (Phase-II) of the composting experiments are

shown in Table 4.

$$\frac{C}{C_0} = Kt^{-N} \tag{1}$$

$$\log \left(\frac{C}{C_0} \right) = \log K - N \log t \tag{2}$$

$$t_{0.5} = 10^{(1/N \log (K/0.5))} \tag{3}$$

The observed half times of DEHP were 1.89 day, 1.73 day and 2.54 day for E-1, E-2 and E-3, respectively, in phase-I. However, the half times of DEHP increased in phase-II, which could be attributed to the transformation of hydrophobic components of DEHP into hydrophilic moieties by polymerization and microbial activity [26]. The degradation of DEHP was divided into two distinct phases and represented by first order kinetics (Fig. 6a)
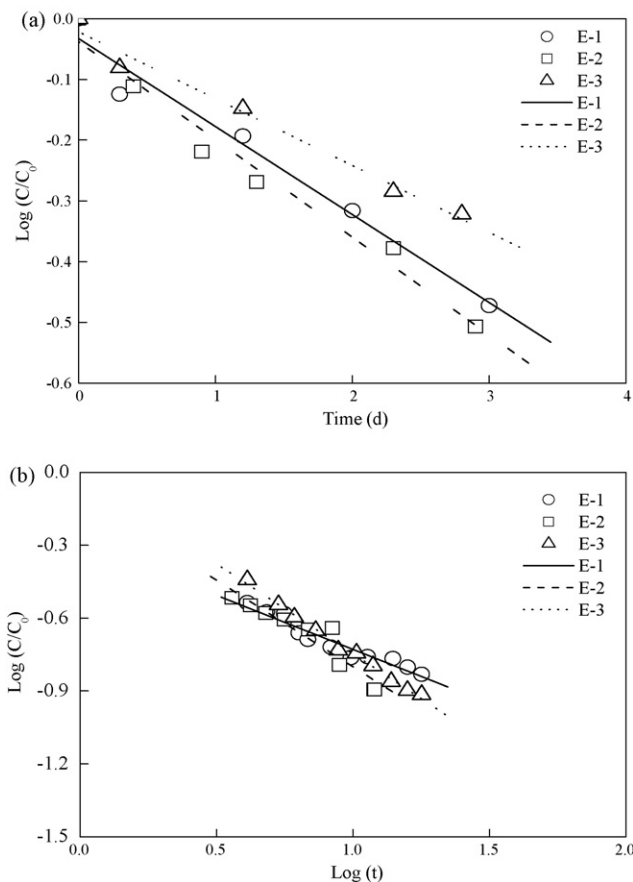

Fig. 6. Kinetics of DEHP degradation in (a) phase-I and (b) phase-II.

Table 5
Pearson correlation coefficient matrix for the degradation of DEHP and organic matter during composting

Parameters	Experiment		
	E-1	E-2	E-3
Temperature	0.33	**0.63**	0.45
Volatile solids (VS)	**0.90**	**0.89**	**0.99**
Total carbon (TC)	**0.93**	**0.97**	**0.97**
Total Nitrogen (TN)	**0.99**	**0.94**	**0.97**
TOCw	0.33	0.21	**0.84**
TKNw	0.23	0.19	**0.61**
DEHP	1	1	1

Data provided in bold were significant at $p < 0.05$ ($n = 19$).

and fractional power kinetics (Fig. 6b). The r^2 values for E-1 to E-3 were in the range of 0.93–0.97 in phase-I. The degradation activity in phase-II was well described by fractional power kinetics as indicated by a straight line in a double logarithmic plot (r^2 of 0.89–0.99). The application of this empirical model has several advantages including a more realistic calculation of long-term depletion times and more accurate prediction of theoretical pollutant concentrations [25]. In phase-I, the highest DEHP degradation rate (k) of 0.4 per day was observed in E-2 followed by E-1 and E-3. However, it was in the order of E-3 followed by E-2 and E-1 in phase-II. Although TOCw (Fig. 4a) was released moderately in all three experiments, phase-I was persisted relatively for a longer time in E-2 (Fig. 2c). The observed rate of DEHP degradation in this study was relatively high compared to the degradation of DEHP observed in sludge-amended soils [19,25]. Besides, the concentration of DEHP after phase-I was well below the stipulated values of land application requested by EU. Hence, composting can be successfully used to reduce the hazardous organic pollutants like DEHP in sewage sludge before the land application.

3.4.2. Influence of experimental parameters on DEHP degradation

The relationship between DEHP degradation and other experimental parameters were assessed using Pearson correlation coefficient matrix (Table 5). In all experimental runs, DEHP degradation was in good correlation with VS, TC and TN. In addition, temperature and water-soluble fractions (TOCw and TKNw) were in good correlation in E-2 and E-3, respectively. The degradation of DEHP was not directly related to the releasable TOCw, perhaps the presence of TOCw has improved the solubility and microbial degradability under thermophilic condition [27], which is evident from Pearson correlation coefficient matrix. Bauer and Herrmann [28] revealed that the elution of DEHP from municipal solid wastes (MSW) was facilitated by the presence of dissolved organic matter (DOM) even under anaerobic conditions, which is in good agreement with the present investigation.

4. Conclusions

The degradation of DEHP and other organic matter of sewage sludge were effective during in-vessel composting. The rate of DEHP degradation was rapid in thermophilic phase (including initial mesophilic phase) (phase-I) compared to phase thereafter (phase-II). DEHP degradation was in good correlation with reductions of VS, TC and TN. The final compost produced in the present investigation has met the requirements of EU and USEPA for land application.

Acknowledgement

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