Effect of compost age and composition on the atrazine removal from solution

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Abstract

Compost samples from two composting facilities, the Urbana (Illinois) Landscape Recycling Center (ULRC) and Illinois State University (ISU), were selected to examine the effect of compost age on atrazine removal from solution. The ULRC samples were made from yard waste without an additional nitrogen source. The ISU samples were made from yard waste or sawdust with the addition of manure. The 6-month-old ULRC compost had the greater capacity to remove atrazine from solution, which we attributed to its greater organic carbon content. The addition of nitrate into ULRC compost could influence the extent of atrazine removal, but did not have a significant impact on atrazine removal when applied to ISU compost, probably because manure was added to the yard waste to produce the compost. For both ULRC and ISU samples, the presence of sodium azide inhibited atrazine removal, suggesting that microbial activity contributed to the atrazine removal. Metabolic analysis demonstrated that hydroxyatrazine was the major identified metabolite that accumulated in solution before significant ring mineralization could occur. When compared with the ISU compost, the ULRC compost sample had a greater capacity to remove atrazine from solution during the 120 days of study because of the larger humic acid content. The experimental results suggested that less-mature compost may be better suited for environmental applications such as removing atrazine from tile-drainage waters.
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1. Introduction

Agricultural practices is one of the major sources of water contamination in the Midwestern United States, and the application of nitrogen fertilizer and herbicides has been implicated as the major component of this problem [1–4]. When the agricultural chemicals are applied on a field, they can enter surface water directly through overland drainage, or they can move in subsurface drainage water. In many areas, the nutrients and pesticides transported through subsurface drainage are discharged into steams or lakes directly, and pose a threat to surface water quality [1–6]. Much research have focused to minimize the environment impacts from subsurface drainage, and substantial researchers have focused on designing inline bioreactors to remove agricultural chemicals from tile-drainage effluent [2,4].

Compost contains a large amount of organic carbon, and can sustain a large population of microorganisms, and therefore could act as a potential treatment medium to remove atrazine from tile-drainage water.

Among all the agricultural chemicals, atrazine (2-chloro-4-(ethylamino)-6-(isopropylamino)-1,3,5-triazine) is the herbicide most often detected in tile-drainage and surface water [1,6,7]. When atrazine was released to the soils, several mechanisms determine the environment fates of atrazine, including adsorption, hydrolysis, and biodegradation. It has been observed that there is a positive correlation between the sorption of atrazine and soil organic matter content [8,9], although several researchers have pointed out that the types of organic matter could have significant impacts on the adsorption of atrazine [10–14]. Organic matter does not only contribute to the sorption of atrazine, but it also plays an important role in the catalytic transformation of atrazine to hydroxyatrazine [15,16]. Compared with the abiotic hydrolysis of atrazine, microbial hydrolysis of atrazine has been considered as a minor fate mechanism

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[17,18]. However, complete mineralization of atrazine has been reported with mixed bacterial cultures and a few pure cultures [19–21]. The addition of easily biodegradable carbon substances would generally stimulate atrazine mineralization [22], but the addition of nitrogen sources slows the atrazine mineralization [21,23,24].

When compost is used in typical agricultural applications, fully matured material is usually required to prevent nitrogen immobilization in the soil. However, if used as a sorbent to remove pesticides from tile-drainage, less-mature compost might be more desirable because it contains more organic carbon than mature compost which would promote sorption of hydrophobic contaminants. In addition, less-mature compost could also provide greater carbon sources for microorganisms to co-metabolize pesticides, and hence increase the capacity for pesticide removal from tile water. The goal of this laboratory-scale study was to test the hypothesis that less-mature compost would be more effective for removing atrazine from solution. Compost samples from two composting facilities in Illinois were chosen to study the relationship between compost age and the sorption and degradation of atrazine. Both sites used yard wastes as the initial feedstock material, but different composting strategies were applied to make mature compost. The effects of the adding nutrients to the compost to potentially enhance its ability to remove atrazine from solution were also examined in this study.

2. Materials and methods

2.1. Compost samples

Compost samples were collected from windrows of different ages at the Urbana Landscape Recycling Center (ULRC) in Urbana, Illinois, and the Compost Facility at ISU near Normal, Illinois. To assess how the chemical properties of compost vary with the age of the compost, one could collect samples from the same pile as the windrow aged. This approach was, however, deemed to be impractical for the current study. At the Urbana Landscape Recycling Center, each new windrow is established in November. Because the starting green wastes tended to be the same type of material each year, it was assumed that the relationship between compost properties and age could be approximated by sampling the different-age piles at the same time even though the starting materials were not literally identical. The same assumption was applied during ISU compost sampling.

The ULRC samples were collected in 2001, and consisted of compost of three different ages: 6 months (U-6), 18 months (U-18), 30 months (U-30), as well as yard waste parent material (U-0). All the ULRC compost was made from yard waste without adding an additional nitrogen source. Two compost samples were collected from the ISU site in 2002, with materials composted for 3 months (I-3) or 15 months (I-15). The ISU samples were made from yard waste with the addition of swine manure (Table 1). Both sites used windrow processes to produce compost.

Table 1
The compost samples used in this study

Compost site	Age (months)	Feedstock materials
Urbana, IL (ULRC)	6	Yard wastes
	18	Yard wastes
	30	Yard wastes
Normal, IL (ISU)	3	Yard wastes and swine manure
	15	Yard waste and swine manure

Around 10 kg of compost sample was collected from each windrow. The compost samples were collected from different depths inside the compost windrow, but special care was used to prevent collecting compost particles on the surface of the compost piles. The collected composite compost samples were first screened through a 4-mm sieve, and smaller subsamples were selected for subsequent chemical characterization. Before chemical characterization, the compost were examined to prevent undecomposed plant materials exited in the subsamples. The reaction pH of the compost samples was determined in a 1:2 soil:water suspension. The total water content was determined gravimetrically at 80 °C for 24 h. Humic acid was extracted with 0.1N NaOH under N_2 and then precipitated with 6 M HCl to pH 1 [25]. The fulvic fraction was estimated by difference between alkaline-extractable humic substance and humic acid content. The C/N/H ratio was determined using a CE 440 CHN Analyzer (Exeter Analytical Inc.).

2.2. Atrazine and its metabolites

Unlabeled atrazine (99% purity) and degradation products deethylatrazine (2-amino-4-chloro-6-isopropylamino-*s*-triazine) (98%), deisopropylatrazine (2-amino-4-chloro-6-ethylamino-*s*-triazine) (99%), and hydroxyatrazine (2-hydroxy-4-ethylamino-6-isopropylamino-*s*-triazine) (99%) were obtained from Chem Service (West Chester, PA). Uniformly ring-labeled ^{14}C-atrazine (specific activity 15.1 mCi mmol^{-1}) (99% purity) was purchased from Sigma Chemical Co. (St. Louis, MO). The atrazine stock solution was prepared by dissolving ^{14}C-labeled and non-labeled atrazine in methanol, and then deionized water was added to prepare a concentration of 20 mg L^{-1}. The stock solution concentration was verified by high performance liquid chromatography (HPLC).

2.3. Adsorption isotherms

Adsorption isotherms were measured using the batch methods developed by Roy et al. [26]. A mass of 0.5 g dry compost sample (without undecomposed plant materials) or yard waste parent material was added to 50-mL polyethylene tubes containing a 25-mL solution containing a known amount of ^{14}C-labeled atrazine, with a concentration of 0.1 mM sodium azide (NaN_3) to inhibit microbial activity. Sample blanks (no compost) showed that losses of atrazine resulting from adsorption by the centrifuge tubes were negligible. The suspensions were mixed in a rotating tumbler at 23 ± 2 °C, and the ^{14}C-labeled atrazine was analyzed using a Packard 2000CA TriCarb liquid scintillation

counter. Kinetic experiments showed that the atrazine concentrations continued to decrease with time for periods up to 120 h, but the rate of change of the atrazine concentration was less than 5% within the first 24 h interval. Hence, a contact interval of 24 h was selected as equilibrium time for all adsorption experiments [26]. The difference between the initial concentration of atrazine and the equilibrium concentration was assumed to be the amount of atrazine adsorbed, and adsorption isotherms were constructed using the adsorption data. The adsorption procedures were run in triplicate for each sample, and linear regression analysis was used to determine K_d and K_{oc} values for atrazine adsorption.

2.4. Incubation study

Incubation experiments were initiated within 1 week after the compost samples were collected, and were carried out for 4 months. The compost samples were not air-dried in order to preserve microbial activity. A 40-g mass of each compost sample was placed into 800-mL Mason jars containing 200 mL of a solution containing an initial atrazine concentration of 20 mg L^{-1} of ^{14}C-labeled compound. The Mason jars were kept in a dark room without any mixing except during sampling.

Four different treatments were created in triplicate for each compost sample: (1) atrazine solution with no additional nitrogen (no-N); (2) atrazine solution with an initial concentration of 20 mg L^{-1} nitrate (low-N); (3) atrazine solution with an initial concentration of 200 mg L^{-1} nitrate (high-N); (4) atrazine solution with 0.1 mM sodium azide (inhibition). A vial containing 10 mL 0.5 M NaOH solution was suspended from the top of each jar to trap CO_2. The trapping solutions were periodically replaced and analyzed for ^{14}C-CO_2 content using a liquid scintillation counter.

At pre-selected times, 5 mL of the solution was collected from each Mason jar bioreactor. The aliquots were first filtered through a 0.45-μm filter membrane (HV, Millpore), and a 1 mL of the filtered solution was analyzed for its total ^{14}C-radioactivity. The atrazine metabolic products were quantified by HPLC using a Hewlett-Packard 1090 liquid chromatograph equipped with a Prodigy 5 μm ODS(3) 100A, 250 mm × 4.6 mm column (Phenomenex, Torrance) and a 10-μm guard column, and a diode array detector which was set at 215 and 230 nm [27]. The solvents used were acetonitrile and a 0.01 M KH$_2$PO$_4$ buffer adjusted to pH 2.0 with a mobile phase flow rate of 1 mL min^{-1}. The operation courses and solvent ratios were as follows (time, %acetonitrile:%buffer): 0–2 min, 5:95; 2–5 min, 5–25:95–75; 5–8 min, 25:75; 8–12 min, 25–50:75–50; 12–20 min, 50:50.

Table 3
Summary of the linear sorption constants (K_d) and organic carbon–water partition coefficients (K_{oc}) for atrazine in equilibrium with the compost samples

Samples	K_d (L kg^{-1})	r^2	K_{oc} (L kg^{-1})
U-0	5.75	0.96	–
U-6	39.8	0.96	119
U-18	21.8	0.98	102
U-30	22.4	0.97	115
I-3	15.1	0.97	45.4
I-15	11.8	0.99	65.2

Standard retention times were determined using external standards of atrazine, hydroxyatrazine, deethylatrazine, and deisopropylatrazine.

3. Results and discussion

3.1. Characterization of the compost samples

As indicated earlier, compost samples were collected from windrows of different ages, but derived from the same type of green wastes to assess the changes in chemical properties of the compost with time. The carbon content and C/N ratio of the ULRC compost samples appeared to decrease with increasing age (Table 2), which is the typical trend when composting organic materials [28,29]. In addition, the humic acid content appeared to increase with composting age for the ULRC compost samples. On the other hand, although the ISU compost samples also showed that the carbon content and C/N ratio decreased with increasing composting time, the humic acid concentration decreased, probably resulting from the addition of pig manure. When compared ULRC samples with ISU samples, it showed that ULRC compost had a greater humic acid content and a smaller fulvic fraction than the ISU samples (Table 2).

3.2. Sorption isotherms

The linear isotherms fit the experimental data well for both ULRC and IRC compost (Table 3). Among the ULRC samples, the composted samples had a significantly greater affinity for atrazine than the uncomposted parent material. Sample U-6, which had the largest carbon content, yielded the largest sorption constant (K_d). However, although U-18 sample had a greater carbon content when compared with U-30 sample, U-18 sample had smaller K_d value for atrazine. Wang et al. [11,12] had pointed out that soil humic acid had much greater sorption capac-

Table 2
Characteristics of the compost samples used in this study

Compost site	Age (months)	Moisture content	pH	Humic acid content (g kg^{-1})	Fulvic fraction content (g kg^{-1})	C	N (g kg^{-1})	H	C/N	C/H
	6	44.1	6.8	98	19	335	17	43	19.7	7.8
Urbana, IL (ULRC)	18	48.3	7.5	108	18	222	15	26	14.8	8.5
	30	48.3	7.8	129	14	214	15	26	14.3	8.2
Normal, IL (ISU)	3	40.2	7.9	51	29	330	22	30	15.0	11.0
	15	20.6	7.9	44	27	182	15	18	12.1	10.1

ity for atrazine when compared with fulvic acid. The higher K_d value in U-30 sample than in U-18 sample could result from its higher humic acid. Similar principle could be used to explain that ULRC compost had a greater affinity for atrazine than the ISU samples, despite that U-6 sample had similar carbon content when compared with I-3 sample, because the ULRC compost had almost double the amount of humic acid as the ISU compost (Table 2). The above observations suggested that not only the amounts of total organic carbon, but the types of organic materials, contributed to the sorption of atrazine. The mean organic carbon–water partition coefficient (K_{oc}) for the ULRC samples ($112 \, L \, kg^{-1}$) was close to the generally accepted value of $100 \, L \, kg^{-1}$ for atrazine in soil [30], but the two ISU samples yielded smaller K_{oc} values.

3.3. Batch incubation studies

Incubating the compost samples with atrazine solutions resulted in reductions in atrazine concentrations during the 120-day contact interval (Fig. 1). More than 65% atrazine was removed within 1 day of incubation, implying that sorption was the major mechanism for atrazine dissipation from solution. The concentration of radiocarbon atrazine continued to decrease after the 1st day of incubation, which was also observed in the sorption isotherm experiments. Li et al. [31] pointed out that the sorption behavior of atrazine could be described as two-stage sorption mechanism: a fast adsorption of pesticide onto soil surface and a slow intra-particle diffusion of pesticide into the soil matrix. Other mechanisms, however, could also be used to explain the continuous decrease of atrazine from solution.

Although ULRC compost could adsorb more atrazine from solution than ISU compost, more atrazine was removed when the solution was incubated with ISU compost from day 1 to day 120 (Fig. 1). As suggested by Li et al. [31], one reason for this observation might result from that ISU compost had greater amounts of pore diffusion sorption sites than ULRC compost, and hence could sorb more atrazine molecules at late stage of incubation. The other possible explanation that ISU compost could remove more atrazine than ULRC compost at late incubation stage could result from that ISU compost contained larger fulvic fraction (Table 2), providing more protonated car-

Fig. 2. The effects of the different treatments on atrazine dissipation from solution for compost U-6. The error bar represents one S.D.

boxylate sites for hydrolysis of atrazine [16]. Since the rate of atrazine hydrolysis follows the first-order reaction [15,16,18], more atrazine could be hydrolyzed and adsorbed by compost when the solution was incubated with ISU sample, due to more atrazine remained in solution.

When the atrazine solution was incubated with compost U-6, the presence of $20 \, mg \, L^{-1}$ nitrate (low-N treatment) resulted in greater atrazine removal when compared with atrazine solution incubated with compost alone (no-N treatment), and the addition of and $200 \, mg \, L^{-1}$ nitrate (high-N treatment) also seemed to favor the atrazine removal (Fig. 2). However, the addition of nitrate to the ISU compost had no significant effect on atrazine removal (Fig. 3). As noted earlier, manure had been applied to the ISU samples during composting, and this could mask the effects of adding nitrate for atrazine removal. On the other hand, no addition nitrogen was added into ULRC samples during composting processes. Therefore, adding nitrate into ULRC compost during incubation could enhance microbial activity, and hence improve the atrazine removal efficiency. For both ULRC and ISU samples, the addition of sodium azide (inhibition treatment) reduced microbial activity (Fig. 2), and resulted in less atrazine removal.
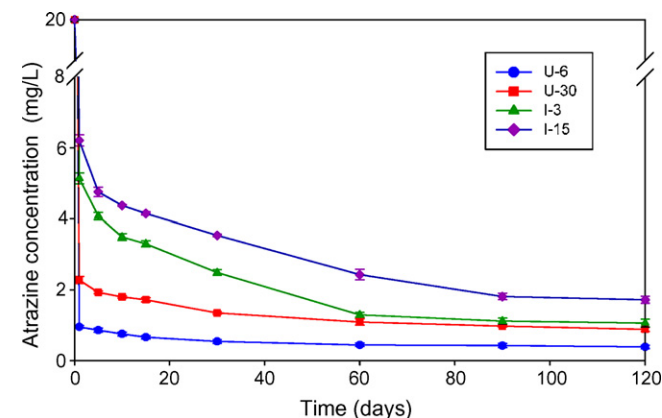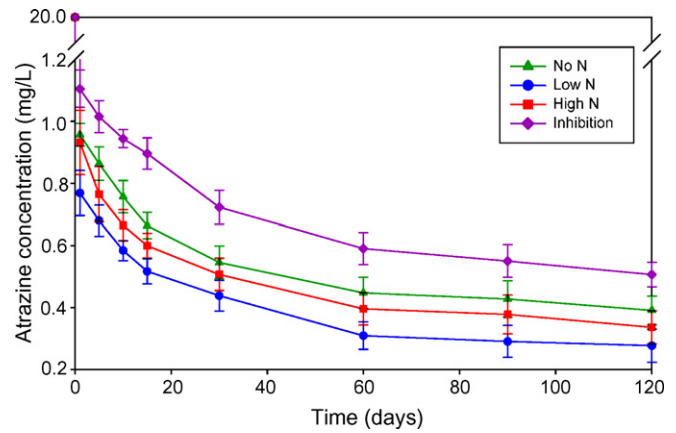

Fig. 1. Atrazine dissipation from solution as a function of time for four compost samples with no-N treatment. The error bar represents one S.D.
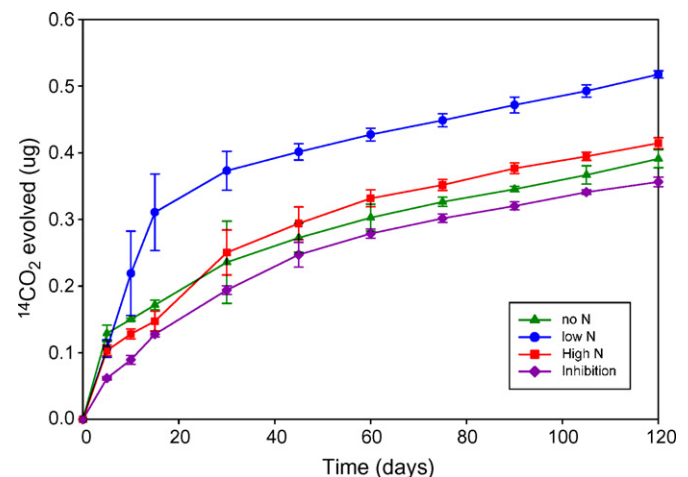

Fig. 3. The effects of the different treatments on atrazine dissipation from solution for compost 1–3. The error bar represents one S.D.
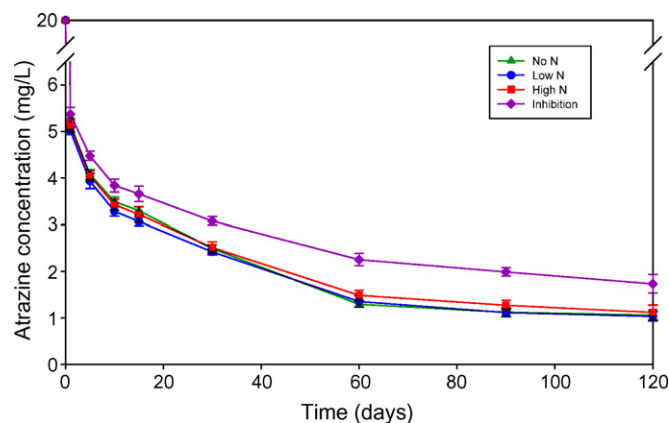
Fig. 4. The effects of the different treatments on atrazine mineralization for compost U-6. The error bar represents one S.D.

Fig. 6. The distribution of atrazine and metabolites in solution for the no-N treatment during incubation with 1–3 compost. The solution data indicated the total radio-labeled compounds in solution obtained from Fig. 3. The error bar represents one S.D.

The amount of $^{14}CO_2$ evolved from the cleavage of the atrazine ring structure for U-6 is shown in Fig. 4. The maximum amount of atrazine mineralized was less than 1.2 µg (0.03%) for all the treatments during 120 days of incubation, probably because most atrazine had been stabilized by compost through sorption [32]. Except for U-6 sample, the amounts of $^{14}CO_2$ evolution decreased as the additional nitrate concentration increased in U-18 and U-30 samples (data not shown). Similar results were also observed in ISU samples (Fig. 5). Several studies have demonstrated that the addition of N sources can reduce the extent of atrazine mineralization because the additional N can inhibit microbial utilization of the atrazine ring-N [21,23,24], and thus less $^{14}CO_2$ evolved with increasing N addition in most conditions. However, for the U-6 sample, it had highest C/N content among all the selected samples. Although adding nitrate would inhibit microbial efficiency to utilize atrazine ring-N, the additional nitrate could increase total microbial activity in U-6 samples, and thus greater amount of $^{14}CO_2$ was observed.

3.4. Atrazine metabolites analysis

When I-3 was incubated, the concentration of hydroxyatrazine increased steadily after day 20 (Fig. 6). Deethylatrazine
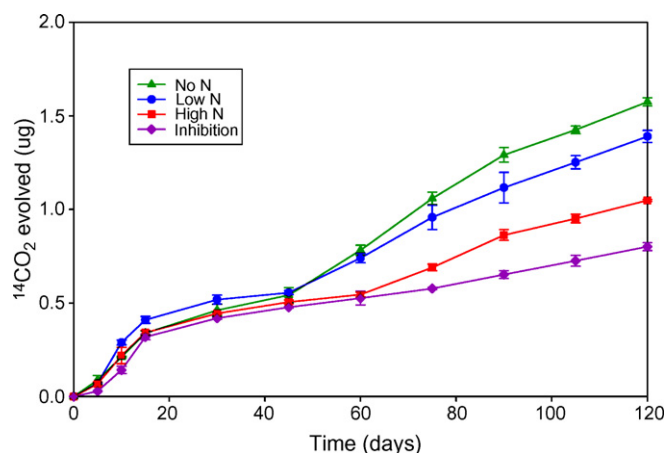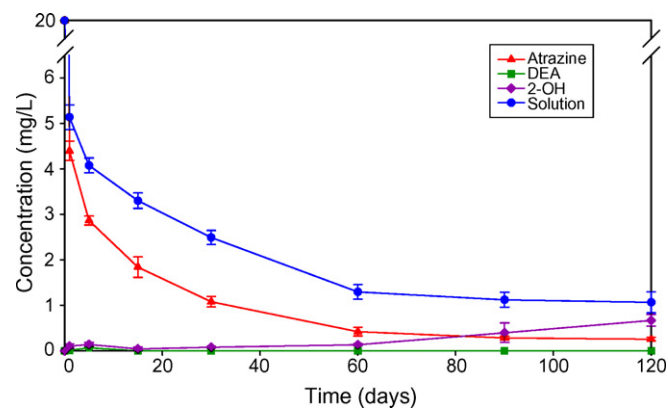

Fig. 5. The effects of different treatments on atrazine mineralization for compost 1–3. The error bar represents one S.D.

was detected at concentrations less than $0.1 \, mg \, L^{-1}$ only during the first 10 days of incubation, and deisopropylatrazine was not detected during the entire incubation period. Several unidentified peaks accounted for 16–60% of the total radiocarbon atrazine in the starting solution based on the mass balance, and these unidentified peaks probably resulted from the dissolved organic materials associated with atrazine molecules. The concentrations of these unidentified products increased from day 1 to day 30, and then decreased.

The results showed that hydroxyatrazine was the major identified metabolic product in solution (Fig. 6). However, little hydroxyatrazine was observed in solution from day 1 to day 30, probably because most of the hydroxyatrazine was adsorbed by the compost during this period. Organic substances have been shown to adsorb larger amounts of hydroxyatrazine than atrazine [33–35]. The hydroxyatrazine began to accumulate in solution since day 60 (Fig. 6), corresponding to the observation that greater amount of atrazine ring mineralization during that period (Fig. 5). These observations suggested that hydroxyatrazine was the major precursors for atrazine ring mineralization.

3.5. Summary and other considerations

Of all the compost samples, U-6 sample removed the most atrazine. It was found that about 98% of the original atrazine was removed from solution after 120 days of incubation (Fig. 1). Although adsorption was the major mechanism by which U-6 removed atrazine from solution, other mechanisms, such as hydrolysis and biodegradation, also contributed to the dissipation of atrazine from solution. It has been demonstrated that when ^{14}C-ring-labeled atrazine was incubated with organic amendments in soil, there was a lag phase, up to 1.5 months, before the evolution of $^{14}CO_2$ was significantly detectable [36,37]. When ^{14}C-ring-labeled atrazine in solution was incubated with ULRC compost, no lag period was observed before the start of atrazine mineralization (Fig. 4), indicating that the microorganisms in the compost could readily use the decomposable carbon in the compost to co-metabolite atrazine. The less-mature compost could apparently provide more labile car-

bon for the microorganisms to use relatively older compost. The labile carbon sources in less-mature compost could be readily available to microorganisms to degrade the pollutants, in contrast to the use of other materials that require a lag phase before significant biodegradation occurs.

This study only represents the batch data showing the possibility of using compost to remove atrazine from tile-drainage water. However, compost itself can contribute some negative qualities to the solution [38]. For example, the compost imparts an amber color via dissolved organic matter. A significant amount of dissolved organic carbon could have been associated with atrazine molecules, which may have accounted for the unidentified peaks in the HPLC chromatographs. In addition, compost could also add trace elements or nutrients to the solution. Therefore, before using compost in the field for environmental application, more research is needed to evaluate the efficiency and environmental impact of using compost as in in-line filter system for drainage tile.

4. Conclusions

Although fully matured compost is more desirable in the traditional agricultural application, the experimental results demonstrate that less-matured compost is more potential in the environmental application to remove pollutant from solution due to its larger organic carbon content. This study suggests that several mechanisms contribute to the atrazine dissipation from solution when incubated with compost, including adsorption, hydrolysis, and biodegradation. The sorption of atrazine by compost would not only depend on the total organic carbon content in compost, but also on the types of compost organic composition. The amounts of atrazine hydrolysis could vary, depending on the atrazine concentration in solution and the fulvic acid content in compost. The addition of nitrate into solution would generally decrease the atrazine mineralization rate, except for the sample with high C/N ratio. Overall, the compost could be a potential filter material for removing atrazine from solution, but more studies are still needed in order to apply compost biofilters in field applications.

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