2013. Proceedings of the Indiana Academy of Science 122(2):69–77

DECOMPOSITION OF NITROGLYCERIN IN SMOKELESS POWDERS BY AEROBIC THERMOPHILIC COMPOSTING

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ABSTRACT. The reported study evaluated the disappearance of nitroglycerin as applied in commercial smokeless powder (SP), in compost prepared from vegetative matter. Double base smokeless powder was applied to mesocosms at rates of 0, 1 and 5% (w/w) and mesocosms were incubated for 70 d. Compost was prepared at two moisture levels, *viz.*, −0.5 bar and −0.1 bar (*wet* and *moist*, respectively). Amendments to microcosms included agricultural limestone and alkaline powerplant fly ash. Determination of free NG was performed using gas chromatography with electron capture detection. In wet compost, NG concentrations decreased by 72% from Day 7 to Day 35. In contrast, NG in moist compost exhibited significantly ($p < 0.05$) less NG solubilization and decomposition. Compost nitrate levels increased markedly after 28 d incubation; however, no correlation was determined between NG concentrations and NO_3 levels or compost pH. Incorporation of limestone or fly ash had no marked impact on NG disappearance. Destruction of NG appears to be substantially more rapid via thermophilic composting compared with incubation in soil, and should serve as a low-cost, viable technology for NG-contaminated media.

Keywords: Composting, nitroglycerin, smokeless powder

INTRODUCTION

Nitroglycerin (NG; glycerol trinitrate; $C_3H_5N_3O_9$) is commonly employed by military forces as an ingredient in artillery and rocket propellants (Accashian et al. 1998). It is also used extensively by private firearms enthusiasts, as NG is a component of smokeless powders (Ahlner et al. 1991; Halasz 2010). Nitroglycerin has been documented as a significant contaminant of soil, surface water, and groundwater resulting from both military conflict and munitions manufacturing and testing (Jenkins et al. 2002; 2001; Pennington et al. 2003, 2002; 2001). It has been detected in soils at concentrations as high as 14,000 mg/kg (Jenkins et al. 2007; Hewitt et al. 2004). Thiboutot et al. (2004a, 2004b) measured soil NG concentrations at 6,560 mg/kg. Nitroglycerin occurs in soil at Indiana military bases and private firing ranges.

In recent decades a range of chemical, physical and biological methods have been applied for remediation of NG and other energetic materials in soil and groundwater, including sorption to activated carbon, reduction with inorganic chemicals, Fenton reaction, alkaline hydrolysis and bioremediation (Kalderis et al. 2011; Accashian et al. 1998). Many of the above chemical treatment processes, however, employ hazardous chemicals, require specialized training by operators, and tend to adversely affect soil biological and physical quality.

In the United States, environmentally-friendly remediation technologies such as bioremediation are encouraged by federal and state agencies (USEPA 1993a; 1993b). Degradation of NG has been documented under both aerobic and anaerobic conditions using mixed or pure strains of bacterial species (Marshall and White 2001; Meng et al. 1995; White et al. 1996; Wendt et al. 1978; Pesari & Grasso 1993). Recent research has revealed the existence of NG-degrading bacteria in activated sewage sludge, river water, and soils (White et al. 1995; Wendt et al. 1978; Zhang et al. 1997).

Various studies have investigated composting as a means to treat energetics-contaminated soils; however, virtually all have addressed treatment of TNT (2,4,6-trinitrotoluene), RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine), and HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) (Pennington & Brannon 2002; Williams et al. 1992; Griest et al. 1990; Lowe et al. 1989; Isbister et al. 1984). Few have studied decomposition of propellants. During 45-day incubations using bench-scale compost reactors, no biodegradation of triple (M3 IAIEl) and

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double base (NOSIH-AA2) propellants was observed by Adrian (1996).

Both limestone and powerplant fly ash have been used as additives in composting food waste and yard waste (Wong et al. 2009; Koivula et al. 2004). According to the American Coal Ash Association (2011), 59.9 million tons of fly ash were generated in the US in 2011. Beneficial uses of fly ash include as a bulking material and buffering agent in composting. Wong et al. (2009) found that fly ash buffered pH during composting of food waste and enhanced decomposition efficiency. When ash was mixed with agricultural lime, the composting period was shortened to ~28 days compared with ~42 days in 3% lime. The addition of ash to compost increased the porosity of the feedstock, and also increased concentrations of macro- and micronutrients of the final product (Koivula et al. 2004).

Limited work has been documented regarding the decomposition of NG, and even less is known with respect to the reactions of smokeless powder and consequent release of NG. In the reported study, the feasibility of aerobic thermophilic composting was assessed for decomposition of NG in smokeless powder. Specifically, the objectives were to compare the efficiency of smokeless powder-derived NG decomposition in vegetable composts: (1) of two moisture regimes; and (2) treated with liming agents (agricultural limestone and powerplant fly ash).

MATERIALS AND METHODS

Characterization of compost feedstock and fly ash.—Compost feedstock consisting of fruit and vegetable scraps and yard waste (leaves, grass clippings) was prepared, with paper scraps added as a bulking agent. The feedstock was incubated for approx. four weeks and aerated via physical mixing every two days. Bulk samples of fly ash were collected from air pollution control devices at the Breed power station (Fairbanks, IN).

Solids (compost and fly ash) pH was measured using a standardized AB15 Accumet pH meter on a 1:5 solids:deionized H_2O slurry. Total organic carbon (TOC) and total nitrogen (N) were analyzed on a Perkin Elmer Series II CHNS/O Analyzer 2400 (Shelton, CT). Acetanilide was the standard used. Water soluble nitrate was measured using Szechrome reagents (Polysciences, no date) in a BioteK PowerWave XS2 microassay system® (Winooski, VT).

Potassium concentrations were determined after extraction by 1.0 N ammonium acetate, pH 7.0, followed by analysis using a Perkin Elmer AAnalyst 2000 flame atomic absorption spectrometer (FAAS) set in emission mode (Knudsen 1982). Phosphorous was measured using Bray-1 extractant combined with a microplate method (PowerWave XS2 Microplate Spectrophotometer) (Olsen 1982). Metal (Cd, Cr, Cu, Fe, Ni, Pb, and Zn) concentrations were determined using DTPA (diethylene triamine pentaacetic acid) extraction followed by FAAS. Samples were extracted with DTPA solution (0.05 M) for 2 h on an oscillating shaker. The mixtures were filtered through Whatman no. 2 filter paper and analyzed using FAAS (Sposito 1982). All glassware was washed with Alconox^{TM} detergent and rinsed with deionized water prior to use.

Nitroglycerin incubation.—Smokeless powder was added at a rate of 1% (w/w) and 5% each to four nylon mesh bags (500g) of compost and mixed manually with a stainless steel spatula. The mesh bags were subsequently placed into 20-l compost-filled mesocosms, which were transferred to a Model 815 Freas Incubator set at 55°C. There were four replications for each treatment.

Compost treatments included two moisture regimes, i.e., wet versus moist. The wet treatment was set to approx. −0.5 bar moisture, and the moist treatment to −0.1 bar as measured using an Irrometer® tensiometer.

A second set of mesocosms (−0.1 bar moisture only) was treated with either agricultural limestone or fly ash at 1% or 5% (w/w). One treatment received 2.5% fly ash plus 2.5% limestone. The compost feedstock was aerated by mixing with a steel spatula every 7 d. No supplemental nutrients were added to the mesocosms. Four replicates were prepared for each treatment.

Compost solids were collected weekly during 70 days of incubation. Samples were collected using a stainless steel rod and transferred immediately to plastic bags. Solids (5 g) were extracted by shaking with 25 ml 92% ethanol for 30 min on a reciprocating shaker (Asbaghi & Pichtel 2012). The suspensions were filtered using Whatman no. 2 filter paper and stored at 4°C until analysis. A sterile, autoclaved sample was also collected at each date as a control.

Gas chromatographic (GC) analysis of extracts was conducted using a Perkin Elmer Clarus 500 gas chromatograph with an electron capture detector (ECD) and a Programmed on-Column (POC) Inlet System. The system included a 6 m Perkin Elmer fused silica capillary column measuring 0.53 mm ID with a 1.5 µm film thickness. Samples measuring 1 µl were injected into the column. The GC oven was temperature programmed as follows: 130°C for 1 min, 10°C/min ramp to 160°C, 30°C/min ramp to 285°C hold for 1 min. The carrier gas was He at a 7.0 ml/min flow rate. The ECD temperature was set to 300°C and the makeup gas was N_2 at a 30 ml/min flow rate. A 1000 mg/L nitroglycerin (NG) standard in ethanol was obtained from AccuStandard, Inc., New Haven, CT. The TotalChrom® Navigator Application (v. 6.3) (Perkin Elmer, Shelton, CT) was used to process, record and report the chromatographic results.

At each sampling date, the pH and soluble nitrate content of the compost mass was determined as described above.

Statistical analysis.—Data obtained for NG decomposition as a function of treatment were tested for statistical significance using analysis of variance (ANOVA). Tests showing significance at $\alpha = 0.05$ were analyzed using a Pairwise Comparisons Test. SPSS™ and MS Excel were used on a Windows-based PC for all statistical analyses.

RESULTS AND DISCUSSION

Characterization of compost, fly ash and smokeless powder.—The compost feedstock was alkaline (pH = 7.9). Total C and N measured 31.3 and 2.5%, respectively, resulting in a C:N ratio of 12.6:1. The feedstock contained low concentrations of P and extractable metals (Table 1). The fly ash was highly alkaline (pH = 11.8) and contained low concentrations of total C and N (1% and < 0.1%, respectively). Concentrations of P and extractable metals were low. Mean NG concentration in the SP was 255,150 mg/kg (data not shown). This value is comparable to those for other commercial smokeless powders (Western 2010, 2007).

Nitroglycerin incubation.—In the wet compost containing 1% SP, soluble NG was detected at 1382 mg/kg on Day 7 and attained the highest level, 2023 mg/kg, at Day 14 (Fig. 1). By Day 63 soluble NG concentrations had declined to 36 mg/kg. In the 5% SP treatment, soluble NG was measured at 13,192 mg/kg on Day 7, following which it declined to 5438 on Day 14 and 1523 on Day 35 (Fig. 1), a 72% decrease over 28 d. Nitroglycerin concentrations declined gradually from Day 42 through Day 70 (2950 to 2303 mg/kg, respectively).

Soluble NG was significantly ($p < 0.05$) higher in the wet compost compared with moist compost (Fig. 2), implying the enhanced participation of anaerobic microorganisms. At Day 14, NG concentrations in the moist compost measured 66 mg/kg, 97% less than that measured in wet compost.

For both moisture regimes, composting SP resulted in significantly ($p < 0.05$) more rapid release and decomposition of NG as compared with decomposition in soil. Asbaghi (2012) found that NG occurring in smokeless powder persisted in soil in significant quantities (152 mg/kg at a 1% SP rate) after 60 d. The current findings also contrast those of Adrian (1996), where negligible biodegradation of triple and double base propellants was observed during 45 d of composting. Double base smokeless powders typically contain nitrocellulose, dibutyl phthalate, diphenylamine, ethyl centralite, waxes and other hydrocarbon-based additives (Western 2007). All should be amenable to beta-oxidation as carried out by heterotrophic microorganisms present in compost. As these compounds are acted upon by microorganisms, the NG is released.

For both the limestone- and fly ash-amended compost, NG was released at 56 d (Figs. 2–3), following which the concentrations returned to baseline.

The nascent NG should be readily amenable to microbial decomposition. Mixed microbial cultures from aeration tank sludge were capable of metabolizing NG (Accashian et al. 1998; Wendt et al. 1978; Zhang et al. 1997). Aerobic microbial cultures have been shown to have the capacity to remove NG rapidly even in the absence of a supplemental carbon source. Most studies, however, have shown the benefits of additional carbon sources (Christodoulatos et al. 1997; Wendt et al. 1978).

Substantial N losses were detected in the compost mixtures (Fig. 4). Most of the losses occurred during Days 28–70 of composting and maturation. Soluble nitrate concentrations increased throughout the study; from Day 35 to

Table 1.—Selected chemical and physical properties of the compost and fly ash amendment.

Parameter	Compost	Fly ash
pH	7.9	11.8
EC, dS/m	0.23	2.4
C, %	31.3	1.0
N, %	2.5	< 0.1
C:N ratio	12.6:1	n/a
Bray-1 P, %	2.8	1.0
Total metals, mg/kg		
Cd	< 0.1	16
Cu	0.2	90
Fe	4290	122500
Zn	160	890

day 70, a 63% increase in nitrate was noted for the 1% SP rate (2.6 to 7.0 mg/kg, respectively). It is not known, however, whether the nitrate originated from the NG or the compost feedstock. Eklind & Kirchmann (2000) found that net N losses were 43–62% in various mixtures of vegetable compost. All recent work is in agreement as regards a single NG denitration pathway under both aerobic and anaerobic conditions in which NG is used as a nitrogen source. Biodegradation occurs via successive denitrations to glycerol dinitrates (GDNs) and glycerol mononitrates (GMNs). Nitroglycerin metabolism produces glycerol 1,2- and 1,3-dinitrate (1,2-GDN and 1,3-GDN), and glycerol 1- and 2-mononitrate (1-GMN and 2-GMN) (Wendt et al. 1978), while nitrate is lost from the parent molecule.

Compost pH trends were similar for both the limestone and fly ash treatments (Figs. 5–6).

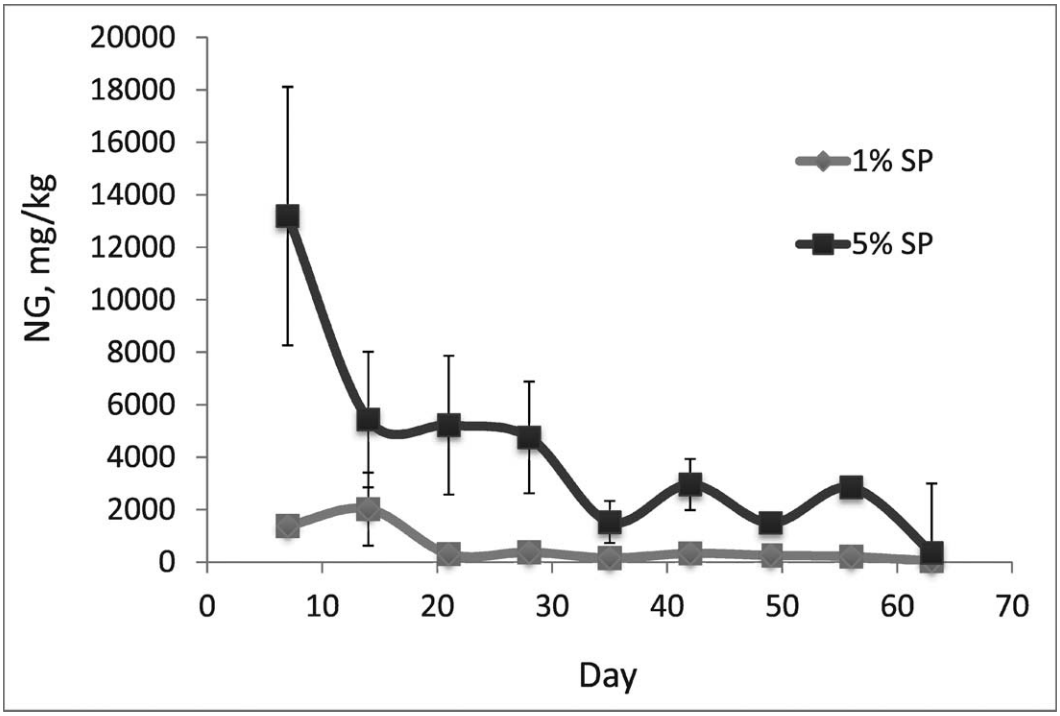

Figure 1.—Nitroglycerin occurring in the wet compost at two rates of smokeless powder, 63 d.

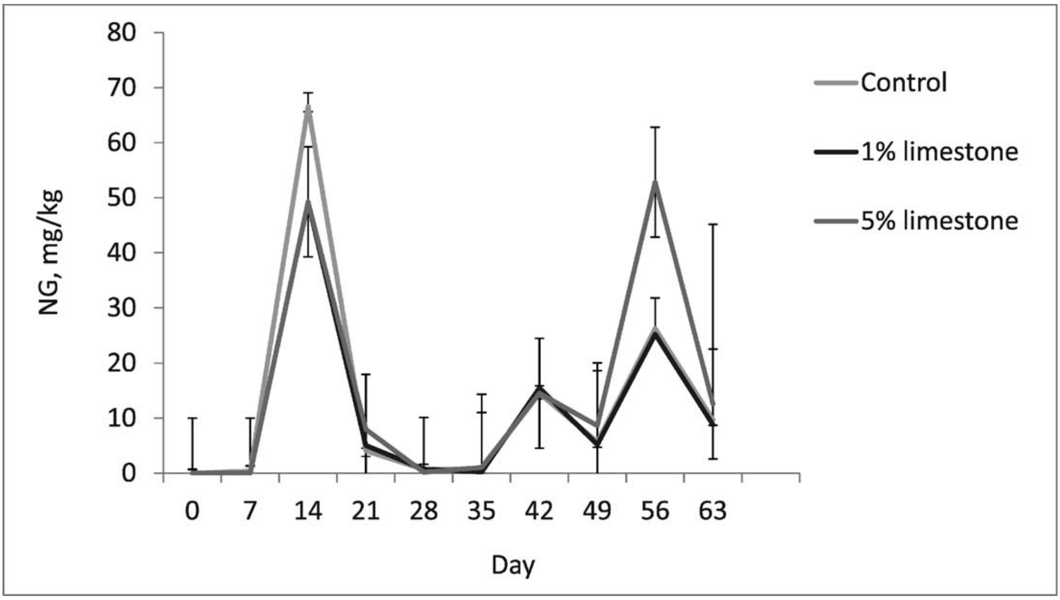

Figure 2.—Nitroglycerin occurring in compost amended with two rates of limestone, 63 d.

Initial pH values ranged between 7.5–7.8. By Day 14, pH climbed to 9.1 and subsequently declined in all treatments. By Day 70, pH values ranged from 7.9 for the 5% limestone treatment, to 8.6 for the 5% fly ash treatment. Such pH values are fairly typical for compost originating from vegetable wastes. Eklind & Kirchmann (2000) measured pH values of 8.2 in household waste compost after 600 d. Incorporation of limestone or fly ash had no marked impact on NG disappearance (Figs. 5–6).

Compost pH measurements did not correlate with nitroglycerin solubilization or decomposition. Nitroglycerin decomposition does not result in the release of basic compounds;

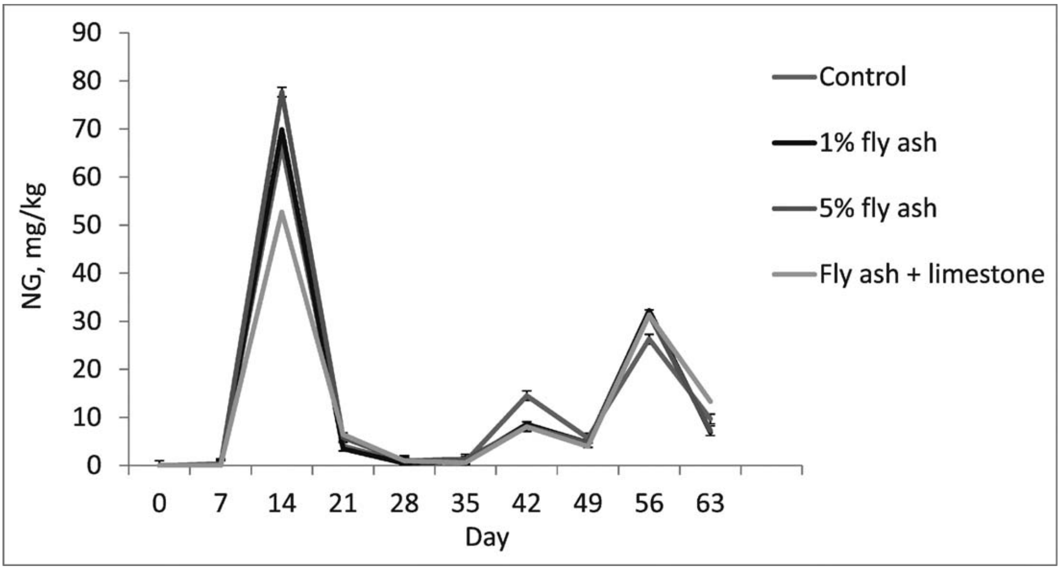

Figure 3.—Nitroglycerin occurring in compost amended with two rates of fly ash, and fly ash + limestone, 63 d.

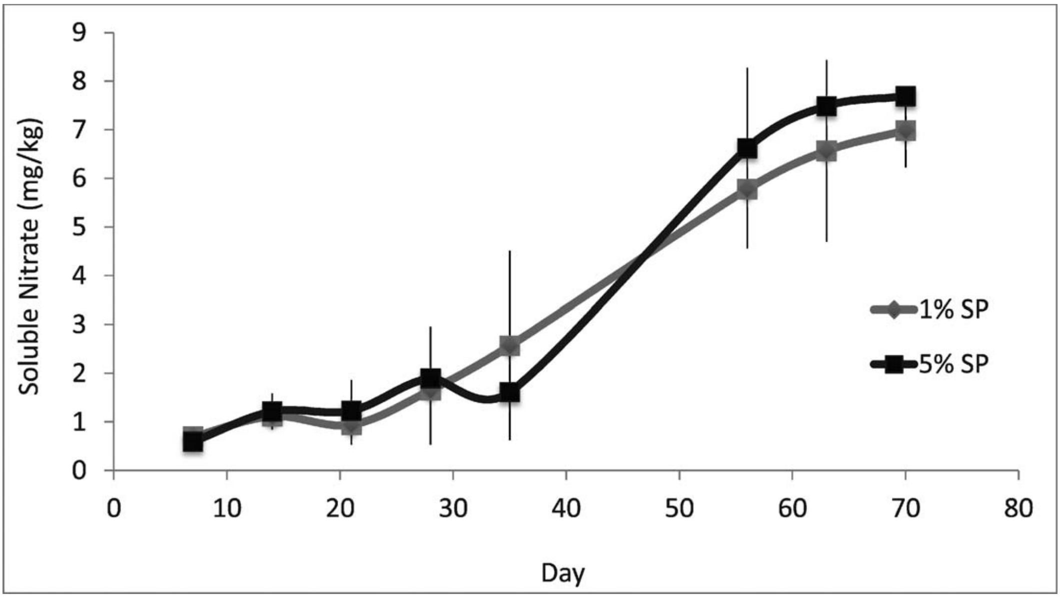

Figure 4.—Soluble nitrate levels in compost treated with two rates of smokeless powder, 63 d.

however, when NO_3^- is released from the NG molecule there is the potential for biological reduction to ammonium (NH_4^+) (Brady & Weil, 2010). In a study of smokeless powder decomposition in soil, Trensey (2013) found that ammonium concentrations ranged from 6.4 to 16.3 mg/kg.

Destruction of NG appears to be substantially more rapid via thermophilic composting compared with incubation in soil, and should serve as a low-cost, viable technology for NG-contaminated media. Studies are currently underway in our laboratory attempting to isolate and enumerate the various microbial

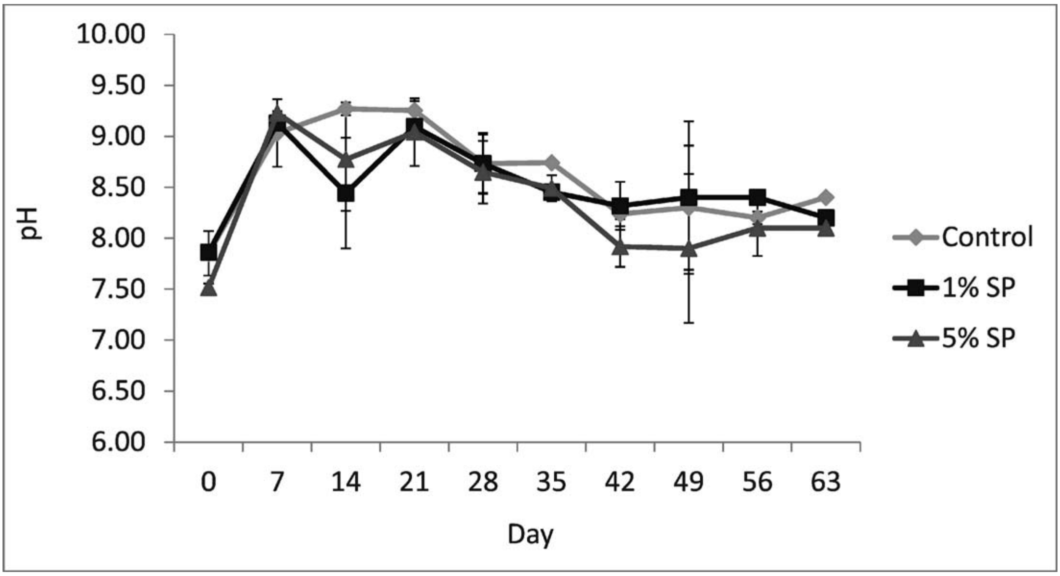

Figure 5.—pH trends in compost amended with limestone, 63 d.

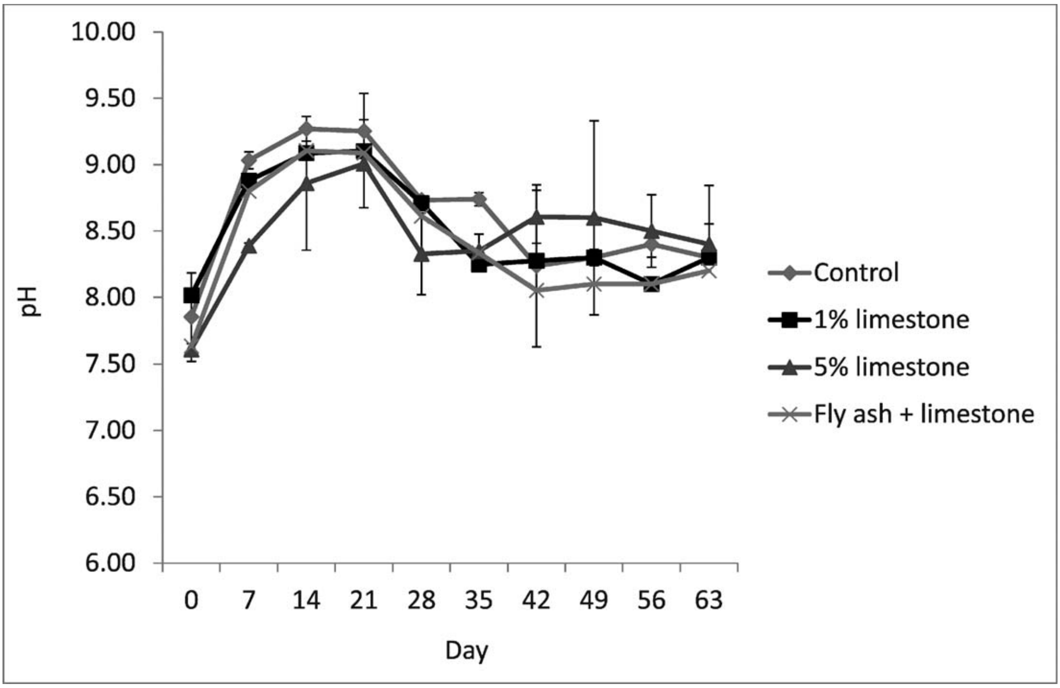

Figure 6.—pH trends in compost amended with fly ash, and fly ash + limestone, 63 d.

populations that are active during microbial decomposition of nitroglycerin.

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Manuscript received 18 September 2013, revised 18 March 2014.