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Composting of explosives and propellant contaminated soils under thermophilic and mesophilic conditions

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SUMMARY

Composting was investigated as a bioremediation technology for clean-up of sediments contaminated with explosives and propellants. Two field demonstrations were conducted, the first using 2,4,6-trinitrotoluene (TNT), octahydro-1,3,5,7-tetranitro-1,3,5,7-tetraazocine (HMX), hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), and N-methyl-N,2,4,6-tetranitroaniline (tetryl) contaminated sediment, and the second using nitrocellulose (NC) contaminated soil. Tests were conducted in thermophilic and mesophilic aerated static piles. Extractable TNT was reduced from 11 840 mg/kg to 3 mg/kg, and NC from 13 090 mg/kg to 16 mg/kg under thermophilic conditions. Under mesophilic conditions, TNT was reduced from 11 190 mg/kg to 50 mg/kg. The thermophilic and mesophilic half-lives were 11.9 and 21.9 days for TNT, 17.3 and 30.1 days for RDX, and 22.8 and 42.0 days for HMX, respectively. Known nitroaromatic transformation products increased in concentration over the first several weeks of the test period, but decreased to low concentrations thereafter.

INTRODUCTION

The manufacture and handling of explosives and propellants has resulted in soil and sediment contamination at U.S. Army munitions facilities. The United States Army is investigating bioremediation using composting as a candidate technology for decontaminating these contaminated soils. In addition, composting is being investigated as a disposal technology for the significant quantities of non-specification grade nitrocellulose (NC) that have accumulated at production facilities.

Composting is a biological process in which organic materials are biodegraded by microorganisms, resulting in the production of organic and inorganic by-products and heat. This heat energy is trapped within the composting matrix, leading to the self-heating that is a characteristic of composting. For bioremediation purposes, elevated temperature composting requires mixing readily biodegradable organic matter (such as agricultural wastes) with the contaminated matrix. Bulking agents such as wood chips may also be required to enhance the porosity and aeration of the mixture to be composted.

The mixture to be composted is typically shaped into a pile or placed in a vessel to facilitate heat retention and process control. Composting is implemented at one of

three general levels of technology; windrow, aerated static pile, and mechanical in-vessel. Piles (or windows) of the mixture to be composted may be constructed. Unmanipulated piles, however, are subject to poor process control. Turning (mixing) of the piles increases the ability to control temperature, oxygen, and moisture. Aerated static piles have a network of pipe underneath the mixture to be composted to facilitate temperature and oxygen control by ventilative means. Mechanical in-vessel systems provide the greatest process control, but require costly facilities. Although composting is typically an aerobic process, anaerobic microenvironments likely form within the central regions of particles in a composting mixture.

Composting is widely used to treat wastewater sludges, processing wastes, and municipal refuse. The advantages of composting as a bioremediation method have been discussed previously [1]. They include: 1) elevated temperatures that facilitate a higher contaminant degradation rate; 2) enhanced opportunities for cometabolism; 3) changing physical/chemical microenvironments within a composting mass that result in a diversity of microbial communities and metabolic activity; and 4) greater contaminant availability due to higher contaminant solubility and mass transfer at elevated temperatures.

Composting of explosives and propellants, especially 2,4,6-trinitrotoluene (TNT), has been investigated by several researchers. Osmon and Andrews [2] and

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Klausmeier et al. [3] both observed biotransformation of TNT in laboratory-scale compost systems. Kaplan and Kaplan [4] studied the degradation of ^{14}C -TNT in a laboratory-scale system, but did not detect the release of $^{14}\text{CO}_2$. They did, however, observe the formation of reduction products and the binding of ^{14}C to the organic matter fraction. The percentage of ^{14}C that was bound to the organic matter in compost increased with increasing compost age and stability.

Isbister et al. [5] evaluated the degradation of ^{14}C -TNT and ^{14}C -hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in laboratory-scale compost systems, as well as the degradation of unlabelled TNT and RDX in greenhouse-scale systems. Both studies demonstrated that RDX and TNT concentrations rapidly declined during six weeks of composting. ^{14}C -RDX was converted to $^{14}\text{CO}_2$, but no evidence of $^{14}\text{CO}_2$ production from ^{14}C -TNT was observed. Contrary to Kaplan and Kaplan [4], Isbister et al. [5] found only minor amounts of TNT reduction products. After additional work, Isbister et al. [5] concluded that the reason for this discrepancy was either that Kaplan and Kaplan did not achieve composting in their laboratory-scale system and/or that they formed the reduction products during their drying or extraction procedures.

Doyle et al. [6] conducted laboratory- (^{14}C) and pilot-scale (unlabelled) studies with TNT, RDX, octahydro-1,3,5,7-tetranitro-1,3,5,7-tetraazocine (HMX), and N-methyl-N,2,4,6-tetranitroaniline (tetryl), and NC. In the laboratory study, ^{14}C from ^{14}C -TNT was recovered primarily in the unextracted residue. Extractable TNT also was reduced to low levels in the pilot-scale studies. RDX, HMX, tetryl, and NC were mineralized to varying extents. Recent laboratory studies [7, 8] have demonstrated that TNT can be extensively mineralized and serve as the sole source of carbon and energy.

The primary objective of the present studies was to evaluate the technical feasibility of aerated static pile composting as a technology for remediating soils and sediments contaminated with the explosives TNT, HMX, RDX, and tetryl, and the propellant NC. The explosives were present as contaminants in lagoon sediments as a result of the disposal of 'pink water', which was generated during munitions packing and loading operations at the Louisiana Army Ammunition Plant (LAAP). The propellant NC was present as a soil contaminant as a result of disposal of NC production wastewater at the Badger Army Ammunition Plant (BAAP), Baraboo, WI. NC waste ('fines') had also accumulated in lagoons at BAAP. Secondary objectives included evaluating transformation under thermophilic vs. mesophilic temperatures, the change in microbial numbers, and the production of transformation products when Standard Analytical Reference Materials (SARMs) were available.

Values for relevant physical and chemical properties of TNT [9], HMX [10], and RDX [11], respectively are: water solubility at 20 °C, 0.013 g/100 g, 6.63 mg/l, and 42.3 mg/l; melting point, 80.1 to 81.6 °C, 276 to 280 °C, and 204.1 °C; vapor pressure, 0.053 mmHg at 85 °C, 3×10^{-9} mmHg at 100 °C (data not available for RDX). Information on sorption coefficients and Henry's law determinations could not be located. All three explosives and NC are generally considered to be nonvolatile.

MATERIALS AND METHODS

Concrete test pads were constructed at both BAAP and LAAP with drainage channels that connected to a sump located below grade. Water from the sump was

TABLE 1
Materials balance of mixture to be composted

Material	Volume (m ³)	Mass (kg)	Percent	
			Volume	Mass
LAAP piles 1 and 2				
Sediment	0.8	1040	3	24
Alfalfa	10.0	430	38	10
Straw/Manure	12.4	1120	47	25
Horse feed	3.4	1810	12	41
Total	26.6	4400	100	100
BAAP piles 1 and 2				
Soil	1.9	1836	9	19
Alfalfa	10.5	1072	49	11
Manure	4.6	4318	21	45
Horse feed	2.3	1550	11	17
Wood chips	2.1	727	10	8
Total	21.4	9503	100	100
BAAP pile 3				
Soil	1.2	1155	12	22
Alfalfa	4.0	410	41	8
Manure	2.8	2630	29	51
Horse feed	1.2	805	13	16
Wood chips	0.5	171	5	3
Total	9.7	5171	100	100
BAAP pile 4				
Soil	2.0	1925	19	33
Alfalfa	3.1	317	30	5
Manure	2.9	2724	28	44
Horse feed	1.2	805	12	13
Wood chips	1.1	376	11	5
Total	10.3	6147	100	100

re-applied to the compost piles when moisture was required. The pads were individually covered by an open-sided structure.

The mixture to be composted at LAAP (Table 1) was prepared using horse manure and soiled bedding (straw), alfalfa, horse feed (Purina Balanced Blend 14), and contaminated sediment. Slurried cow manure was used in place of horse manure at BAAP (Table 1). The LAAP sediment contained TNT (56 800 mg/kg), RDX (17 900 mg/kg), HMX (2390 mg/kg), and tetryl (650 mg/kg); BAAP soil contained only NC (18 800 mg/kg). Sawdust, wood chips, and baled straw were used to construct the compost pile base and insulating cover. A mechanical feed system, developed initially to meter explosives-contaminated soil into an incinerator, was used to homogenize sediment and to mix the material to be composted at LAAP. A Reel Auggie (Knight Manufacturing Corp.) was used to mix the material to be composted at BAAP.

Two landfill thermocouple probes (Atkins Technical) per pile were used to continuously monitor internal pile temperatures at LAAP. One probe was placed 60 cm inside the pile, 90 cm above pad level, and 60 cm from the blower end of the pile. The other probe was inserted into the center of the pile adjacent to the thermistor controlling the operation of the blower. A landfill probe equipped with a hand-held digital thermometer was used to manually monitor temperatures on a daily basis. At BAAP, six thermocouples per pile were used.

The mixture to be composted was placed on a base that contained a system of perforated ADS polyethylene drainage tubing (10-cm diameter) placed within a wood chip base and connected (nonperforated tubing) to an explosion-proof radial-blade blower. The blower was used to pull air through the compost pile. Blower cycling and pile temperature were controlled by both timer (start-up period) and temperature feedback systems. The temperature feedback system consisted of soil thermistors that measured compost temperature and panel-mounted Fenwal series 551 thermistor-sensing temperature controllers. The mesophilic set point (LAAP and BAAP pile 1) was 35 °C and the thermophilic set point (LAAP pile 2, BAAP piles 2, 3, and 4) was 55 °C.

The mixtures to be composted were prepared as follows. Sediment or soil was homogenized and analyzed for explosives or propellant concentrations. Soil, amendments, and 8 kg of fertilizer (nitrogen, potassium, phosphorus, 13 : 13 : 13) for each pile were then blended. The material balance of the mixtures is presented in Table 1. Mixtures were developed based on C : N ratio, with a goal of 30 : 1, and the presence of abundant degradable biomass. Soil was added at a level sufficient to result in significant detectable contamination. Each load of compost at LAAP was moistened with water as it was delivered to the pile base. Approximately 1500 liters of

water were applied per pile. No additional moisture was required at the start of the BAAP tests.

NC production waste from a storage basin at BAAP was blended with the pile 4 mixture to be composted to form nominal concentrations of 5, 15, 30, 60, and 80% by weight. Four sets of triplicate samples for each concentration were prepared. Three sets were individually placed in nylon bags that were then placed in polyethylene mesh sleeves. The three bagged sets were placed in the middle of pile 4 and removed at designated times for analysis. The fourth set was analyzed as a time zero sample.

Pile construction at LAAP was completed and the temperature control system and recorder were started on 25 February 1988. Samples were taken with a 3-inch diameter soil auger from the central region of the pile from at least three locations along the length of the pile for each sampling time. The insulating cover was removed prior to taking samples. Samples were mixed by hand prior to subsampling for extraction and analysis. Samples (at least three per sampling time) were analyzed for contaminant concentration nine times over a 153-day test period.

Moisture was added when temperatures started to fall. This generally occurred when percent moisture fell below approximately 40%. The LAAP compost piles were individually dismantled and the composting mixture remixed and remoistened on days 33, 60, and 111. Piles 1 and 2 at BAAP were monitored for 151 days starting on 29 April 1988. Piles 3 and 4 were monitored for 112 days starting on 28 September 1988. The BAAP piles were individually dismantled and the composting mixture remixed and remoistened at week 6 (piles 1 and 2) or week 7 (piles 3 and 4).

Analyses for metals were conducted by procedures described in Standard Methods for Chemical Analysis of Water and Wastes (U.S. EPA 600/4-79-020, 1979). Compost samples were analyzed for TNT, RDX, HMX, tetryl, and transformation products by U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) Method LW02, modified for the extraction and analysis of compost. The high absorptive capacity of compost required a higher volume of solvent (40 ml per 10 g) than specified in Method LW02. Explosives and transformation products were extracted using acetonitrile and analyzed by HPLC. NC was analyzed spectrophotometrically using USATHAMA Method LY02. NC was extracted with acetone using ultrasonic agitation. The extract was dried, washed in methanol/water, dissolved in acetone, and hydrolyzed with potassium hydroxide. Nitrate was then analyzed. A method blank and standard spikes for each analyte were analyzed with each batch of samples. Spike recoveries consistently exceeded 90% in standard soils. USATHAMA standard soil was used for recoveries since a standard compost could not be defined.

Microorganisms were enumerated on Difco nutrient

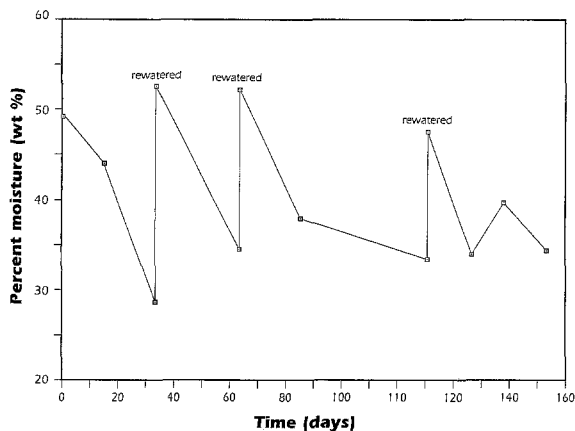


Fig. 1. Moisture content in LAAP pile 1.

agar by serially diluting a 10% (w/v) compost/phosphate buffer mixture, which had been vigorously shaken for 2 min. Large particles were allowed to settle prior to diluting the mixture. Plates were incubated in the dark at either 35 or 55 °C, and colonies were enumerated at day 2 and day 5. Samples were dried at 95 °C for 24 h for moisture determinations.

RESULTS

LAAP data

The percentage of moisture of LAAP compost samples during the test period varied from 25 to 56 weight percent. At test initiation and following remoistening, moisture content was approximately 50 weight percent. Moisture content dropped to the 25–35% range over a period of 30 to 40 days. Moisture data for LAAP pile 1 are shown in Fig. 1. Average temperatures within LAAP piles 1 and 2 are presented in Fig. 2. Temperatures dropped significantly when the percent moisture fell below 35 weight percent.

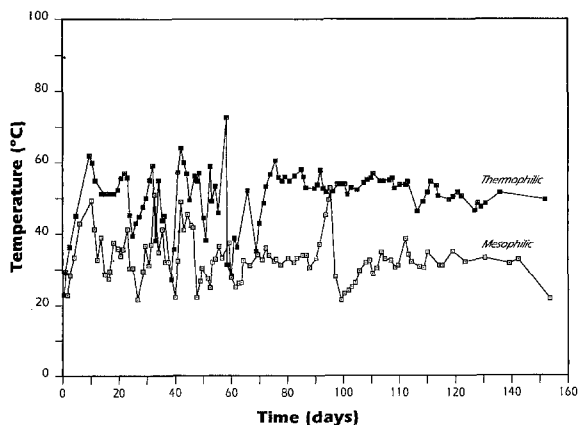


Fig. 2. Mean compost temperature in LAAP piles 1 and 2.

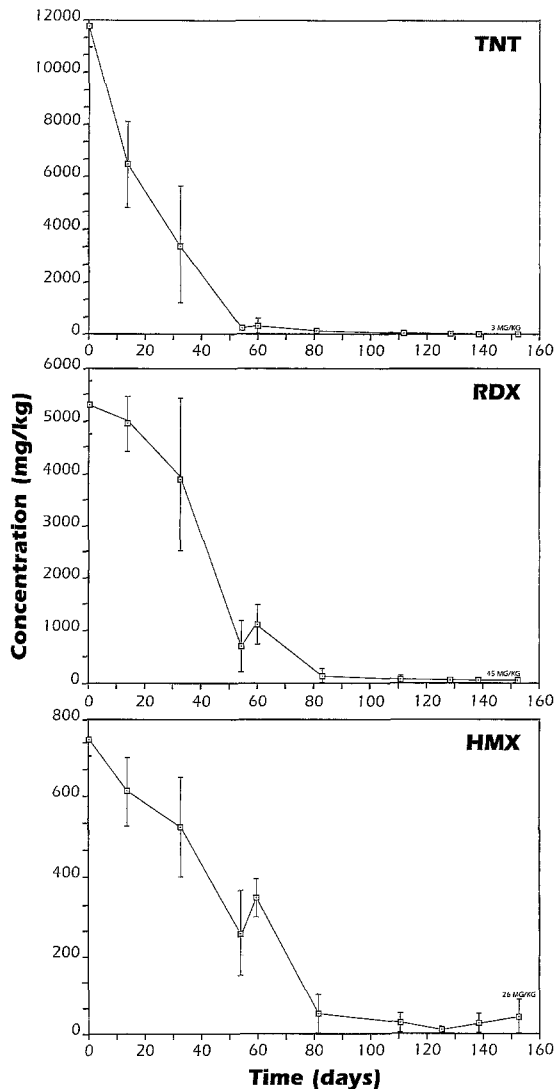


Fig. 3. Concentration of TNT, HMX, and RDX under thermophilic conditions (error bars represent standard deviation).

Total explosives in the thermophilic and mesophilic piles at LAAP were reduced from 17870 and 16460 mg/kg to 74 and 326 mg/kg, respectively during the 153-day test. The concentration of TNT, HMX, and RDX within the thermophilic pile at LAAP is presented in Fig. 3. No data are presented for tetryl since the compound was below detection limits in all compost samples (including time 0) analyzed. The mean and standard deviation (error bars for Fig. 3, 4, and 6) at each time point represent at least three and as many as nine samples. Calculated half-lives (days) were 12 for TNT, 17 for RDX, and 23 for HMX under thermophilic conditions and 22 for TNT, 30 for RDX, and 42 for HMX under mesophilic conditions.

LAAP compost samples were analyzed for the only TNT transformation products for which SARMS could be

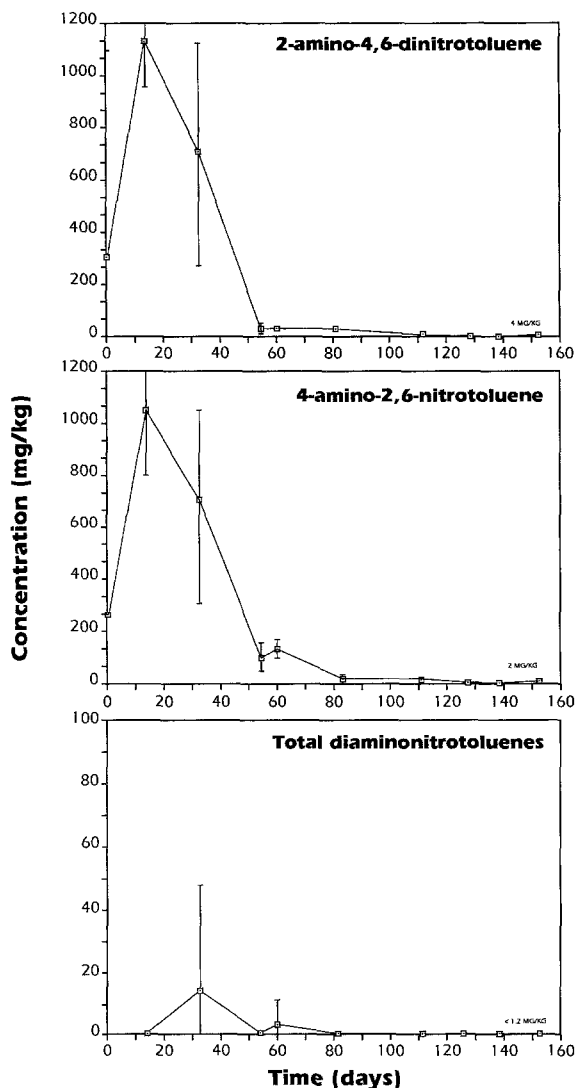


Fig. 4. Concentration of intermediates in LAAP pile 2.

obtained (2-amino-4,6-dinitrotoluene; 4-amino-2,6-dinitrotoluene; 2,4-diamino-6-nitrotoluene; and 2,6-diamino-4-nitrotoluene). Initial concentrations of transformation products in these samples ranged from nondetectable to 250 mg/kg. These individual concentrations increased over the first several weeks of the test period to a high of approximately 1500 mg/kg, and then decreased to low mg/kg levels thereafter. At day 153, the concentration of each transformation product was less than 22 mg/kg and in many cases nondetectable. Transformation product data for LAAP pile 2 are presented in Fig. 4.

Samples of water from the sump were analyzed for explosives and transformation products on days 0, 16, 22 and 153. The analytes were below detection limits in all sump water samples. Samples of sump water were analyzed for metals at day 153. Potassium, magnesium,

sodium, aluminium, iron, and calcium were the only metals detected at concentrations greater than 1 mg/l. No metals were detected in extracts of compost samples obtained using the EPA Toxicity Characteristic Leaching Procedure (TCLP).

The appearance of the LAAP compost changed considerably over the 153-day test period. When the materials to be composted were initially mixed, the mixture had a highly fibrous appearance, a rough texture, and it smelled conspicuously of the materials used to prepare it. After approximately 100 days, the compost had become more soil-like and less fibrous in appearance. At the end of the test period, the compost had both the appearance and smell of loamy soil. Although nonquantitative, visual examination of both central and peripheral samples indicated that fungal growth was considerably heavier in the mesophilic than in the thermophilic pile at LAAP. Fungal growth penetrated into the central regions of all particles examined.

The presence of microorganisms capable of growing at 35 and 55 °C was evaluated. Plate counts were performed using extracts of samples from the mesophilic and the thermophilic pile. Two sets of plates were prepared for each extract. One set was incubated at 35 °C and the second at 55 °C. As shown in Fig. 5, the maintenance of

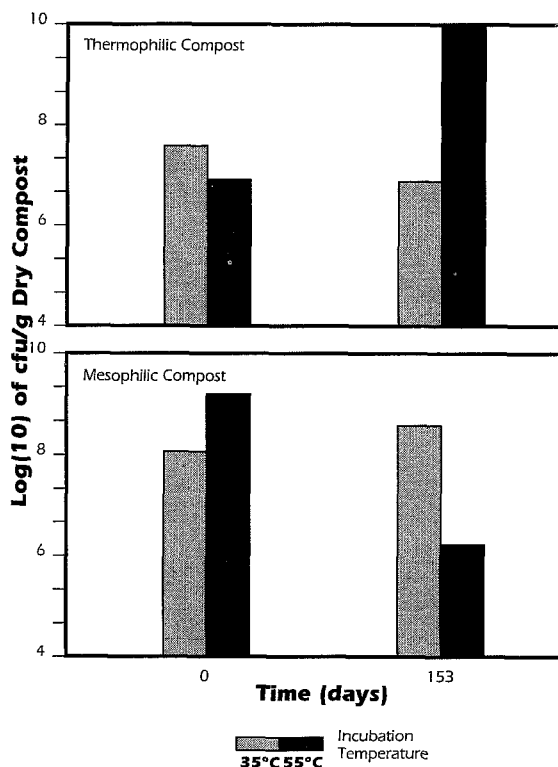


Fig. 5. Population density of heterotrophic microorganisms in thermophilic and mesophilic LAAP compost.

the compost pile temperature at 35 °C led to a selective increase in the number of microorganisms capable of growth at 35 °C and a decrease in the number capable of growth at 55 °C. Maintenance of temperature at 55 °C in the thermophilic pile resulted in an increase in the number of microorganisms capable of growth at 55 °C and a decrease in the population capable of growth at 35 °C.

BAAP data

The percentage of moisture of BAAP compost samples varied from 27 to 65 weight percent. The moisture at test initiation and following remoistening was in the 55–65 weight percent range. Moisture dropped to the 30–40 weight percent range after 20–30 days.

Temperature in BAAP piles 2, 3, and 4 reached 55 to 65 °C within five days of test initiation. Thermophilic temperatures then were maintained until approximately day 65, when temperatures began a gradual decline into the mesophilic range. BAAP pile 1 maintained a mesophilic temperature of approximately 35 °C until approximately day 57. After this time, temperatures climbed into the thermophilic range, and were between 60 and 65 °C between day 75 and 94. Temperatures then gradually declined to approximately 30 °C at day 150. Temperature control in pile 1 became ineffective after approximately day 55, presumably reflecting the inability of air to flow well through the compacting mixture.

NC was significantly destroyed in all tests conducted at BAAP. In piles 1 and 2, NC was reduced in samples taken from several locations in the pile from a high of 4933 mg/kg and 3039 mg/kg to 133 mg/kg and 54 mg/kg, respectively. Compost fortified with nominal concentrations of 3, 5, 7.5, and 10% NC was placed in mesh bags located in the central region of piles 1 and 2. After 98 days, NC was reduced from 23 600 mg/kg to 61 mg/kg in the 10% NC bagged compost sample placed in pile 2. Extensive NC reduction was observed in all other bagged samples placed in piles 1 and 2.

The soil fraction used in the mixture to be composted was increased from 19% (by mass) used in piles 1 and 2 to 22 and 33% for piles 3 and 4, respectively. In addition, NC was added to compost at higher concentrations (nominal of 5, 30, and 60%) for samples contained in mesh bags and placed in pile 3. Results from a minimum of five samples taken from several locations in piles 3 and 4 for each time point are presented in Fig. 6. NC concentration in compost contained in mesh bags was reduced from $14\,310 \pm 3068$ mg/kg to 1662 ± 1405 mg/kg; from $114\,500 \pm 26\,160$ mg/kg to 2455 ± 1028 mg/kg; and from $218\,600 \pm 23\,570$ mg/kg to $68\,800 \pm 12\,190$ mg/kg over the 101-day test period.

Samples of sump water were analyzed for NC at the end of the test periods for piles 1 and 2 and piles 3 and

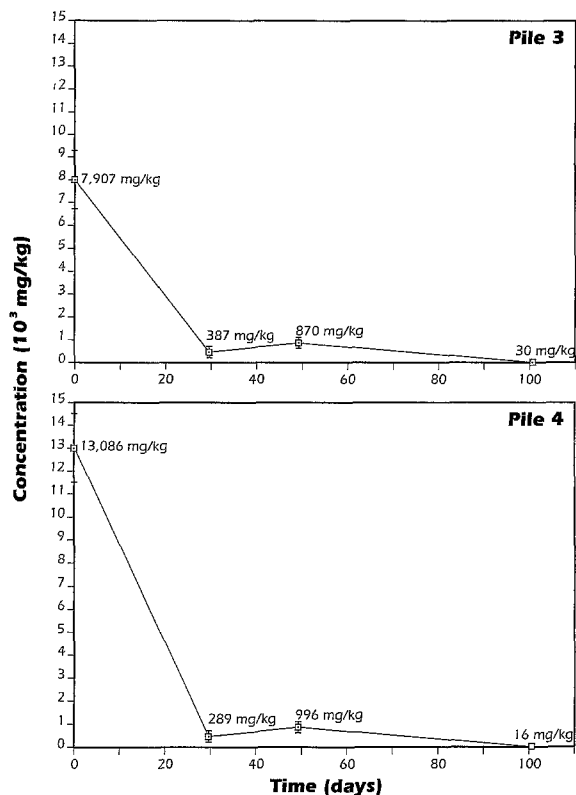


Fig. 6. Concentration of nitrocellulose in BAAP piles 3 and 4 (error bars represent standard deviation).

4. NC was below the detection limit for all sump samples. The appearance of the BAAP compost changed similarly to the LAAP compost over the test period. Enumeration of microorganisms in all four piles at BAAP consistently demonstrated population densities between 10^6 and 10^7 cfu/g.

DISCUSSION

Concentrations of solvent-extractable TNT, RDX, and HMX in LAAP piles and NC in BAAP piles were significantly reduced during the field tests. Fate mechanisms that may have been responsible for contaminant reduction include sorption of explosives and transformation products to the compost matrix, incorporation of explosives and transformation products into environmentally stable molecules such as humic acids, and mineralization of explosives to carbon dioxide, water, and other inorganics. Volatilization was unlikely since the explosives and their transformation products are non-volatile.

The field demonstration using explosives-contaminated soil at LAAP demonstrates the significant transformation potential (for example, extractable TNT reduction

from 11 840 mg/kg to 3 mg/kg) of the composting process. Consequently, composting may be a suitable technology for remediating explosives- or propellant-contaminated soil and sediment, as well as solid matrices contaminated with other biodegradable compounds. The degradation of high concentrations of NC enclosed in mesh bags indicates that composting may prove suitable for the destruction of NC production waste. Caution should be used in interpreting these mesh bags results, however, since an entire compost pile containing a high NC concentration may exhibit much different composting and NC degradation characteristics than the small quantities studied in bags in this program.

The utility of composting for explosives-contaminated matrices is still in doubt due to the need for additional data on the final transformation products and toxicity of the compost residue. An investigation of the LAAP compost residue [12] has revealed low ($\mu\text{g/ml}$) levels of leachable TNT, HMX, RDX, and two monoaminodinitrotoluenes. Leachates from the mesophilic compost residue were slightly more toxic than those from the thermophilic compost residue. The basis for this difference is unknown.

The LAAP program demonstrated superior contaminant destruction under thermophilic conditions. This difference could arise from greater contaminant solubility, higher rates of reaction, or the nature of thermophilic vs. mesophilic microbial communities. Both visual observation of the compost and enumeration of microorganisms indicate that differences exist between these microbial communities.

The microbial population size data presented in Fig. 5 are suggestive of the selective effect temperature has on the overall character of the microbial community. The effect of these community changes on the activity or function of individual populations, however, is unknown. It is likely that the significant activity of aerobic microorganisms within compost results in some localized areas of oxygen depletion and thus in the occurrence of anaerobic microsites. In particular, this would be expected within the central region of particles greater than 1–2 cm, or in regions where the moisture content exceeds approximately 60% of the water holding capacity.

The high variation observed in contaminant levels between individual samples from different sampling points early in the test period is reasonable and most likely reflects differences in microbial activity at different locations within the piles. Within any one of the several samples collected at one time, variation was low, as demonstrated by the analysis of several subsamples. The high variation observed in NC content within the BAAP mesh bag studies reflects the difficulty in obtaining a uniform NC distribution within a mixture to be composted.

A cost analysis [13] has indicated that the ability to economically process significant quantities of soil using composting is sensitive to the kinetic rate of contaminant destruction and, to a greater extent, soil concentration in the mixture to be composted. The BAAP study demonstrated that soil loadings as high as 33% by mass could be incorporated without diminishing the ability to generate heat and maintain thermophilic temperatures during winter in Wisconsin. For BAAP piles 3 and 4, more than half of the daily low temperatures were below 0 °C, with a maximum low temperature of –20 °C. For many days, the daily high temperature was also below 0 °C. If effective composting and contaminant destruction can be achieved with a soil volume of 20% and a total amendment cost of \$50/ton, composting can compete with incineration as a cost-effective remedial alternative.

While composting is a feasible bioremediation approach, certain cautions must be kept in mind. Volatile contaminants may be released during composting requiring additional treatment equipment and subsequent increased costs. Microorganisms have yet to be effectively utilized for the treatment of most heavy metals. Consequently, a careful evaluation is required when hazardous concentrations of metals or volatile organic compounds are present. Although composting is a water-consuming process, the potential for leachate formation should be considered. Finally, it is crucial to understand the final fate and toxicity of the contaminants prior to implementing composting for hazardous substance destruction. Treatability studies should be conducted to ensure that acceptable decontamination can be achieved.

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