



Diffusive transport of 2,4,6-trinitrotoluene (TNT) from contaminated soil to overlying water

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Abstract

Understanding the transport mechanism of 2,4,6-trinitrotoluene (TNT) and other compounds is necessary in order to implement an effective phyto- or bioremediation scheme for explosives-contaminated soil. The transport of TNT from two contaminated soils into overlying water was investigated using a laboratory sheet-flow leaching bed reactor (SLBR). Soil I had a low contamination of TNT ($11 \pm 1 \text{ mg kg}^{-1}$), whereas Soil II had very high contamination of TNT ($22874 \pm 518 \text{ mg kg}^{-1}$). The results showed a decrease in aqueous effluent TNT concentration and flux with time in both cases indicating the diffusive nature of the process ($t^{-1/2}$ dependence). The flux from the sediment to the water column was used to obtain the effective diffusivity of TNT by fitting the data on Soil I to a mathematical model. The average effective diffusivity value obtained was $1.18 \times 10^{-6} \pm 8.32 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$. This suggests slow diffusive transport of TNT through the soil. The initial flux from Soil II was approximately 50 times higher than from Soil I. The final steady state flux was larger than that from Soil I since the initial porewater TNT concentration was much higher for Soil II. From our experiments, we calculated that the characteristic time for transport is estimated to be ca. 7 years whereas the first order degradation rate by plant enzymes is ca. 70 min. The slow transport of TNT from the soil bed will be a limiting factor in the phyto- or bioremediation of explosives-contaminated soils. © 1998 Elsevier Science B.V.

Keywords: Trinitrotoluene; Diffusion; Soil; Phytoremediation

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1. Introduction

Contaminated soils are found at many inactive and abandoned federal munitions facilities in the US. Munitions facilities typically have recalcitrant nitro-aromatic compounds that have persisted for decades in surface and subsurface soils. These explosives-contaminated soils contain several compounds, a major component being 2,4,6-trinitrotoluene (TNT). TNT is a single ring aromatic compound with an empirical formula of $C_7H_5N_3O_6$ and a molecular weight of 227. It has limited solubility in water (ca. 100 mg l^{-1} at 298 K) [1]. The existing information on subsurface TNT fate and transport is meager [2]. Unlike many other organic compounds, TNT has little affinity for soils and can rapidly migrate to the groundwater [3–7]. The toxic and/or inhibitory effects of TNT to animals, fish, plants and microorganisms have been well documented [8–12]. For this reason, TNT has been the focus of intensive cleanup efforts at several facilities.

In order to select an appropriate remediation strategy, one needs to understand the transport and fate of TNT within contaminated soils. The remediation of TNT-contaminated soil in some cases involves TNT transport from the soil to water as one important and controlling step for the process. For example, phytoremediation is carried out in an aqueous medium containing plants and enzymes [13]. This is a topic of ongoing research within the EPA Hazardous Substances Research Center (South and Southwest) which is a consortium between the Louisiana State University, Rice University and Georgia Institute of Technology. This project was initiated in support of that effort. Fig. 1 is a conceptual reactor configuration that is being evaluated for phytoremediation. It consists of two-steps. First, the TNT is transported from soil to an overlying water column by diffusion and advection in a leaching bed reactor. Second, the TNT in the aqueous phase is delivered to a plant bioreactor where it is transformed to other reduced species by enzymatic reactions.

The primary objective of this research is to study the process of TNT transport from soil to water. The apparatus used in this study is called a sheet-flow leaching bed reactor (SLBR). It consists of a thin rectangular shaped stainless steel chamber containing a flat

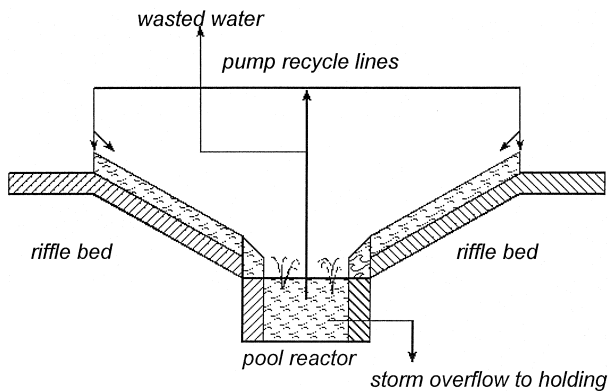


Fig. 1. Schematic of a pseudo-steady state operation for a TNT-plant enzyme bioremediation process.

bed of soil. A thin film of water flows over the soil surface simulating an inclined field lysimeter for delivering TNT-laden water to a bioreactor. The purpose of these experiments is to determine the bed-derived leaching rates, diffusivity, and the extractable amount of TNT from soil, all of which are needed for the design of a field extractor unit. The experimental data from the laboratory microcosms will be used in an appropriate mathematical model to obtain the necessary parameters. These laboratory investigations of the fundamental diffusion process will serve to support future field studies. In a further study the mass transport information on TNT could be integrated with the TNT biotransformation kinetics and used in the optimal design of a field-scale TNT phytoremediation scheme.

2. Experimental

2.1. Field soil collection and preparation

Two types of soil samples were used in this study. Soil I was a low contamination sample collected and processed from a contaminated site (Alabama Army Ammunition Plant, AAAP) located in Talledega county (near Childersburg) in east central Alabama. Soil II was acquired through the US Army Engineer Waterways Experiment Station, Vicksburg, MS, and had a high TNT concentration. The soils were screened to eliminate large debris. Soil characteristics such as porosity and particle size distribution were determined by standard methods for soil analysis. The bulk density was determined using the core method and the particle density was determined using the pycnometer method [14]. The pertinent physical and chemical properties of the soils are given in Table 1.

2.2. Experimental apparatus and procedure

Fig. 2 is a schematic of the apparatus, both top and side views. Several such reactors were available in the laboratory for each experiment. Each reactor was 50 cm long, 5 cm

Table 1
Characteristics of the soil

Parameter	Value	
	Soil I	Soil II
Porosity	0.46	0.42
Bulk density (g cm^{-3})	1.38	1.53
Particle density (g cm^{-3})	2.57	2.64
Percent organic matter (f_{om})	1.7	NA
Percent clay	5.3	29.5
Percent silt	10.8	35.9
Percent sand	83.9	34.6
Soil pH	6.5	NA
Cation exchange capacity (Meq/100 g)	7.4	NA

NA denotes not analysed.

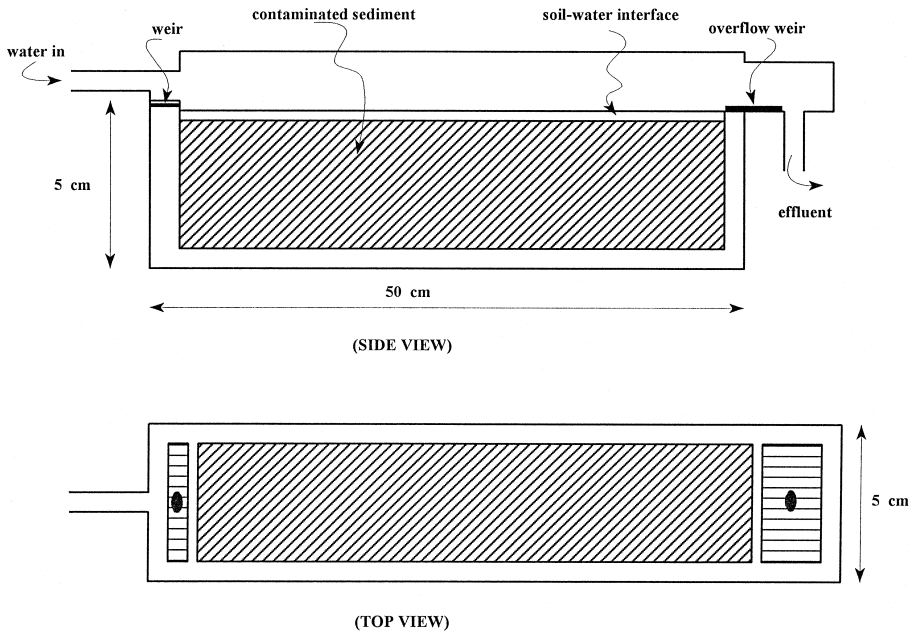


Fig. 2. Schematic of the sheet-flow leaching bed reactor (SLBR).

wide and 5 cm deep. Into the reactor the soil was loaded in a slurry form and levelled to a depth of 4.7 to 4.8 cm using special skimmer blades. In normal operation, distilled water would enter the inlet chamber, flow over the weir into the soil bed in sheet-flow (2 to 3 mm thickness) and to the overflow weir at the exit chamber. Continuous water flow across the soil bed was provided using a multicartridge peristaltic pump with average flow of 96 ml h^{-1} . Samples of water at the exit were collected at appropriate intervals analyzed for the explosives. The actual water flow rate was calculated from the total volume (ΔV) collected over the time interval. From the effluent water sample concentration (C), the soil-water interface area (A) and the duration of sample collection (Δt), the flux was calculated using the equation

$$N_A = \frac{\Delta VC}{\Delta t A} \quad (1)$$

At the conclusion of the experiment, the soil bed was drained overnight, the soil cored into 2 mm layers, extracted and analyzed for TNT.

2.3. Soil sample analysis

The determination of TNT load on the soil was carried out using either a sonication or a shaking method which involved the following steps: An accurately measured weight

(W_t) of the sample was placed in an amber bottle containing a known volume (V) of acetonitrile. It was capped and placed in a sonication bath for 5 h, or on a shaker bath for 18 h. After settling for 1 h, 5 ml of the supernatant was pipetted and combined with 5 ml of HPLC grade water to achieve a 50/50 ratio of acetonitrile and water. It was then filtered through a 0.2 μm PTFE filter. The first 2 ml was discarded and the remainder used to obtain TNT concentration (C , mg l^{-1}) using a high pressure liquid chromatography technique described later. The total TNT load on the soil (w , mg kg^{-1}) was obtained from CVN/W_t where N is the dilution factor, if any used. Both sonication and shaking gave comparable results.

2.4. Aqueous sample analysis

3 to 4 ml of the aqueous sample was filtered into an amber glass sampling vial using a 0.2 μm PTFE filter. An equal volume of acetonitrile was added for preservation and to achieve the 50/50 ratio of acetonitrile to water. The sample was then thoroughly shaken and 2 μl injected into a liquid chromatograph.

2.5. Analysis of TNT and other explosives

Although TNT was the main compound in this study, we have also analyzed the samples for all possible explosives commonly observed in these soils. A mixture of twelve analytical standards was obtained from Crescent Chemicals. The mixture contained the following compounds: HMX, RDX, trinitrobenzene (TNB), dinitrobenzene (DNB), tetryl, nitrobenzene (NB), 2,4,6-trinitrotoluene (TNT), 2,4-dinitrotoluene (2,4-DNT), 2,6-dinitrotoluene (2,6-DNT), 2-nitrotoluene (2-NT), 3-nitrotoluene (3-NT) and 4-nitrotoluene (4-NT) purchased from Crescent Chemicals Inc. The mixture was dissolved in a 50:50 acetonitrile–methanol solution with concentrations of about 1 mg ml^{-1} . The standard was diluted in acetonitrile to achieve a 40 ppm standard stock solution. Working standards were made by diluting the stock solution with HPLC-grade water. The HPLC was calibrated using these standards.

The EPA Standard method 8330 was slightly modified to analyze for TNT and related contaminants [15]. A Hewlett-Packard 1090 Series IIL high pressure liquid chromatograph (HPLC) equipped with a photodiode array multiwavelength UV detector and controlled by an HP Chemstation software with autosampler was used. Two mobile phases were initially tested, viz., a 30/70 water/methanol and a 50/50 water/methanol. Since the 50/50 case gave better separation of peaks, it was used at a flow rate of 0.9 ml min^{-1} .

A Hewlett-Packard BDS-5 Hypersil LC-18 reversed phase cartridge column (5 μm particle size, 250 \times 4 mm) was used. The injection volume was 2 μl and the oven temperature was set at 40°C. The UV detector was set at a wavelength of 254 nm. The total runtime was only 14 min as against the 30 min in the original EPA method. The separation obtained by this method was superior to that offered by the EPA method. Sample handling and QA/QC was ensured by regular analysis of system blanks and

comparison of results obtained by different extraction and quantitation methods. Duplicate analysis was employed for selected samples to assess method variability.

3. Results and discussion

The overall mass transfer from sediment to water is given by

$$N_A(t) = K_L(C_{pw} - C_w) \quad (2)$$

where C_{pw} is the porewater concentration of TNT (g cm^{-3}) in equilibrium with the soil and C_w is the TNT concentration in the overlying water (g cm^{-3}). Generally, for low contamination levels in the soil, $C_{pw} = w_A/K_d$, where w_A is the TNT load on the soil (g g^{-1}) and K_d is the soil–water partition constant for TNT ($\text{cm}^3 \text{g}^{-1}$). K_L is the overall mass transfer coefficient for TNT (cm s^{-1}).

In general, there are two resistances to mass transfer from the soil porewater to the overlying water column in an SLBR. These are: (i) a diffusional resistance to mass transfer within the soil pores and, (ii) a water-side boundary layer mass transfer resistance. When the concentration of TNT in the soil is ‘high’ (i.e. $w_A > w_c$, a critical soil loading at which the porewater TNT concentration is at its equilibrium water solubility) the porewater concentration is constant and hence the flux is solely controlled by the water-side boundary layer resistance and will be approximately constant. However, when the soil loading of TNT is ‘low’ ($w_A < w_c$), both resistances will be important. For the latter case, the overall mass transfer coefficient is given by [16]

$$\frac{1}{K_L} = \frac{1}{k_1} + \sqrt{\frac{\pi t}{D_e R_f}} \quad (3)$$

where k_1 is the water-side mass transfer coefficient (cm s^{-1}), D_e is the effective diffusion coefficient for TNT in the soil porewater ($\text{cm}^2 \text{s}^{-1}$) and R_f is the retardation factor given by $\epsilon + \rho_b K_d$. ϵ is the soil porosity, and ρ_b is the soil bulk density (g cm^{-3}). Each term in the above equation denotes a resistance. It should be obvious from Eq. (3) that at $t = 0$, the water-side resistance dominates whereas as t increases the second term (soil-side resistance) becomes important. Thus the mathematical models for flux from a typical contaminated soil require that we incorporate the changing characteristics of mass transfer resistances at different TNT concentrations in the soil porewaters. The value of k_1 in Eq. (3) can be estimated from an equation developed earlier [16,19]

$$k_1 = 0.449 \left[\frac{(gQ')^{2/3} D_A^2}{\nu L} \right]^{1/3} \quad (4)$$

where Q' is the volumetric flow rate per unit width of soil–water interface ($\text{cm}^3 \text{s}^{-1} \text{cm}^{-1}$), L is the length of the bed (cm), g is the gravitational constant (cm s^{-2}), ν is the dynamic viscosity and D_A is the molecular diffusivity of TNT in water ($\text{cm}^2 \text{s}^{-1}$).

For the analysis of the experimental conditions reported in this paper, we need an appropriate equation for flux of a non-reactive sorbing pollutant such as TNT from a

'finite' sediment bed to the overlying water column. This was presented earlier [17] and is given as follows:

$$N_A(t) = \frac{2D_e C_0 H^2}{L} \sum_{n=1}^{\infty} \frac{\exp(-\alpha_n^2 T)}{H(H+1) + \alpha_n^2} \quad (5)$$

where

$$H = \frac{Qk_1 L}{D_e(Q + k_1 A)}, T = \frac{D_e t}{R_f L^2}$$

and α_n are the eigenvalues of the equation $\alpha \tan \alpha = H$. C_0 is the initial porewater concentration of TNT (g cm^{-3}), Q is the water flow rate over the soil ($\text{cm}^3 \text{s}^{-1}$) and L is the depth of the soil layer (cm). This model is valid only for the case where $w < w_c$ (low contamination of TNT in the soil). In the above equation all the parameters are known except the effective diffusivity. By fitting the model to the experimental flux data the appropriate value of the effective diffusivity can be obtained.

The contaminated soil from the AAAP site (Soil I) had an average concentration of $11 \pm 1 \text{ mg kg}^{-1}$ of TNT. Apart from TNT several other explosive compounds were also observed in the soil. Table 2 lists the average concentrations of these compounds. Five separate experiments were conducted in the SLBR. A typical experimental profile is shown in Fig. 3. It is clear from the figure that the flux of TNT from the contaminated soil decreased with time indicating that the soil-side resistance is dominant in this case. The initial concentration of TNT in the aqueous phase was 1.4 mg l^{-1} which was much lower than the aqueous solubility. Within a short time after the water flow rate had begun a concentration gradient developed within the soil, which then limited the mass transfer of TNT to the overlying water column. A simple calculation using Eq. (3) shows that for the experimental parameters used in these experiments, the soil-side resistance becomes equal to the water-side resistance in about 2 h. Other explosive compounds (for example, Tetryl, DNT and TNB) also showed similar flux profiles (Fig. 4). The data for the other four experiments also showed the same behavior. The fluxes are in the order $\text{TNT} \approx \text{Tetryl} > \text{DNT} > \text{TNB}$.

The mathematical model for the flux required several experimental parameters such as the soil–water partition constant for TNT, the water flowrate, soil–water surface area,

Table 2
Concentrations of major explosives and other compounds in soil

Compound	Concentration (mg/kg dry soil)	
	Soil I	Soil II
TNT	11 ± 1	22874 ± 518
2,4-DNT+2,6-DNT	8.5 ± 1.3	13 ± 4
Tetryl	10 ± 1	< BDL
TNB	4.3 ± 0.1	25 ± 6
DNB	0.8 ± 0.1	< BDL
RDX	NA	$6,150 \pm 183$
HMX	1.2 ± 0.2	$1,416 \pm 19$

NA denotes not analysed.

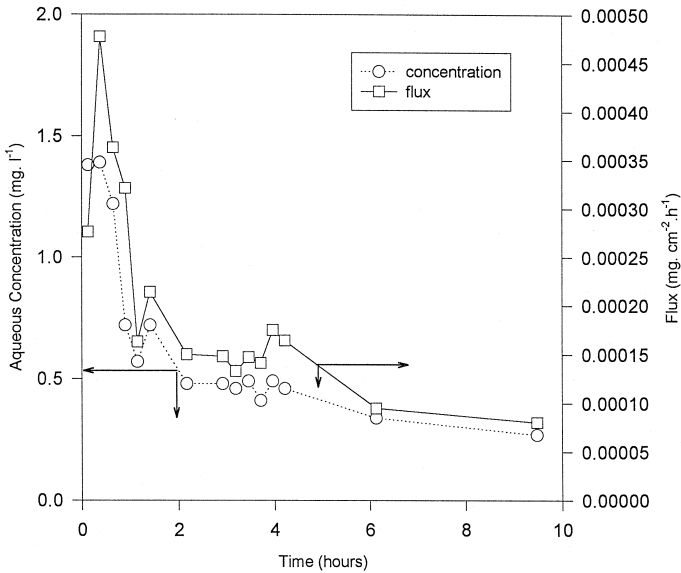


Fig. 3. A typical experimental profile of TNT flux from Soil I versus time at a water flow rate of 97 ml h⁻¹.

and the water-side mass transfer coefficient. A K_d for TNT of 2.9 l kg⁻¹ was obtained using a thin disc experiment [20]. Values of K_d ranging from 2 to 12 for TNT on a variety of different soils have been reported by others [6,7]. The water-side mass transfer

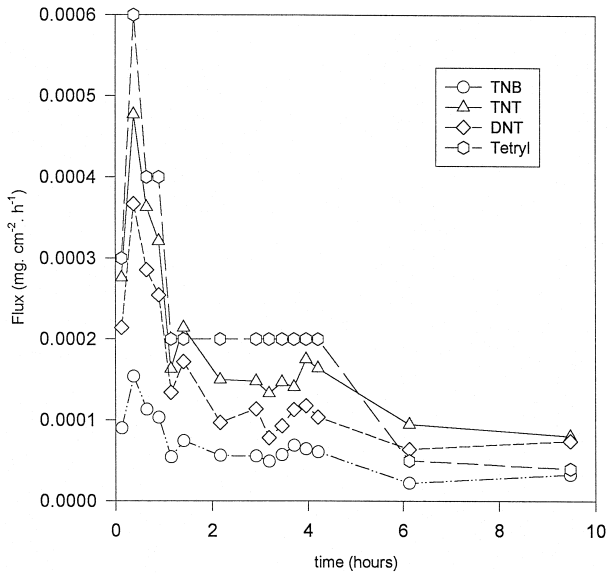


Fig. 4. Typical flux versus time curves for different contaminants from Soil I.

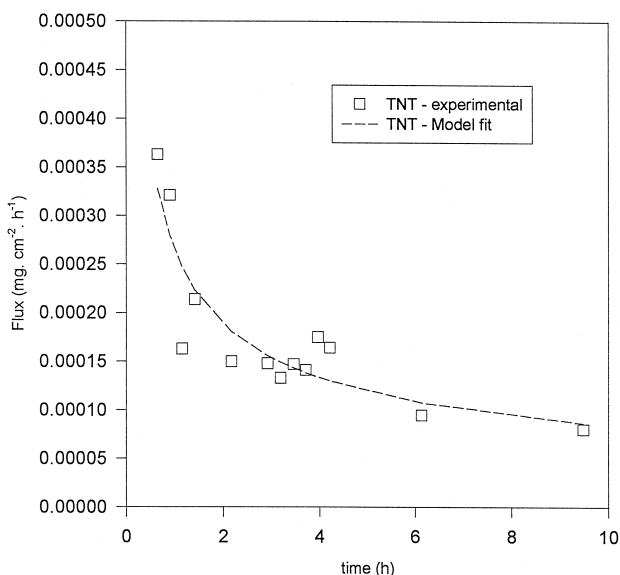


Fig. 5. Typical model and experimental profiles of flux versus time for TNT flux from Soil I.

parameter, k_1 was obtained from Eq. (4). The model was found to satisfactorily predict the removal of TNT from Soil I under these experimental conditions. A typical fit to the experimental data is shown in Fig. 5. The degree of fit suggests that the fundamental transport mechanisms appeared to be accounted for. The effective diffusivities obtained from the model for the five different experiments using Soil I are shown in Table 3. The average value of the effective diffusivity (D_e) was $(1.18 \pm 0.83) \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$. The molecular diffusivity (D_A) of TNT can be obtained from this, since $D_A = D_e \epsilon^{-4/3}$, and is $3.28 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ which compares well with the value of $6.71 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$ estimated using the Wilke–Chang correlation [2,18]. The magnitude of the effective diffusivity of TNT obtained from these experiments suggests very slow transport through soil and illustrates the fact that the diffusion of TNT is the limiting factor in the transport from Soil I to overlying water. The relatively large standard deviation in D_e indicates perhaps the variability in soil packing for the different experiments.

Table 3

Effective diffusivities obtained from experimental data for Soil I

Experiment number	Flow rate, Q ($\text{cm}^3 \text{ h}^{-1}$)	Fitted D_e ($\text{cm}^2 \text{ s}^{-1}$)
1	90	1.42×10^{-7}
2	84	1.60×10^{-6}
3	103	7.83×10^{-7}
4	108	1.06×10^{-6}
5	100	2.34×10^{-6}
Average	96 ± 9	$(1.18 \pm 0.83) \times 10^{-6}$

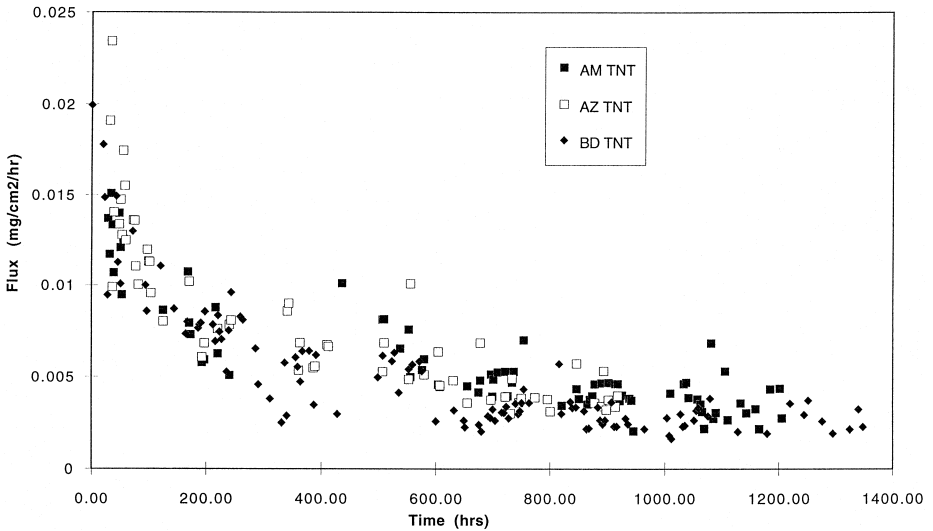


Fig. 6. Flux of TNT versus time for TNT from Soil II at a water flow rate of 110 ml h^{-1} .

Fig. 6 shows the results of three different experimental TNT flux to water from Soil II at a flow rate of 110 ml h^{-1} . The initial TNT concentration in the overlying water was 68 mg l^{-1} which is only 68% of its aqueous solubility at room temperature. This is expected since the TNT concentration on the soil is high (but less than the critical loading), and the porewater concentration should therefore be high. An important feature of the flux profile is that the initial TNT flux is fifty times larger than the case of Soil I. If the effective diffusivity is similar in both cases, one should expect that, based on Eq. (5), the flux for Soil II should be directly proportional to the initial TNT porewater concentration. This seems to be borne out by these results. It is also interesting to note that the final steady state flux from Soil II is 20 times larger than that from Soil I. If the TNT concentration in the soil were higher than the critical soil loading, one would have expected a constant initial flux for a significant period of time as a result of the water-side boundary layer mass transfer resistance. Only with significant surface TNT depletion over time will the process become soil-side diffusion controlled and a slowly decaying flux will then become apparent.

Consider the conceptual phytoremediation scheme for the field as shown in Fig. 1. The leached TNT from the bed will be delivered to a plant pool bioreactor where hydroponic plants will enzymatically degrade TNT to reduced products. It has been shown in work conducted at the US EPA Laboratory in Athens, GA that the rate of biodegradation is pseudo first order and given by

$$r_p = -k_p[P][\text{TNT}] \quad (6)$$

where r_p is the rate in mass of TNT converted per volume of water in the reactor per time ($\text{mg cm}^{-3} \text{ min}^{-1}$), k_p is the rate constant ($\text{min}/\text{wt fraction}$), $[P]$ is the weight fraction of plants (g g^{-1}) and $[\text{TNT}]$ is the concentration of TNT in the aqueous phase

(mg cm^{-3}). Data obtained from Wolfe and Carrera [21] showed that $k_p[P] \approx 0.01 \text{ min}^{-1}$ (a pseudo first order rate constant).

The overall schematic for the process thus involves two steps



A characteristic time for each step can be defined as the time it takes for the concentration to decrease to $1/e$ times its original value. The characteristic time for Step I is that for diffusion from a semi-infinite bed of thickness L as given by $\tau_{\text{diff}} \approx L^2 R_f / 4D_e$ [22]. Assuming a 1 m deep soil bed from which TNT is to be remediated, τ_{diff} is $9.7 \times 10^7 \text{ s}$ ($= 7.7 \text{ years}$). The characteristic time for a reaction is its half-life. For Step II since the rate constant is of the order of 0.01 min^{-1} , the characteristic time τ_{rxn} is 70 min. It is therefore clear that the rate of Step I, i.e., the rate of diffusion from the porewater to the overlying water will control the overall rate of TNT depletion from the soil.

4. Conclusions

It was demonstrated that the flux of TNT from contaminated soils to overlying water decreased with time indicating a strong resistance to mass transfer of TNT from soil porewaters. A mathematical model satisfactorily predicted the removal rate of TNT from soil under the conditions of the experiment. The effective diffusivity of TNT calculated by fitting the experimental data to the model was $1.18 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$, indicating very slow diffusive transport of TNT through soil. These results implied that diffusion of TNT is the limiting factor in the overall transport from the soil bed.

A further implication of the low diffusivity observed in these laboratory experiments is apparent in the context of remediation alternatives such as phytoremediation of explosives-contaminated soils. The plant biotransformation of TNT to other products is known to occur in a relatively short time frame (pseudo first-order rate constant ca. 0.01 min^{-1}). The success of the process will then be dependant on the rate of delivery of TNT from the soil bed to overlying water, and obviously it will be limited by the slow diffusive transport through the soil. For successful implementation of this remediation scheme, methods will have to be devised that accelerate the delivery of TNT to the hydroponic plants.

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