Environmental fate and transport of nitroglycerin from propellant residues at firing positions in the unsaturated zone

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ABSTRACT

The surface soils from firing positions with M-72 66 mm antitank shoulder rockets at the Canadian Forces Base (CFB) Gagetown, New Brunswick, and those at the CFB Petawawa, Ontario were sampled. These soils were know to be contaminated with propellant residues and were spread at the surface of four columns containing uncontaminated soil from the CFB Valcartier, Quebec, and the CFB Petawawa, Ontario. The soils were watered in the laboratory following the groundwater recharge pattern of their respective region. The bottom of each column had nozzles to sample interstitial water output. The compounds of interest were NG (nitroglycerin), and its degradation metabolites, DNG (1,2-dinitroglycerine and 1,3-dinitroglycerine), MNG (1-mononitroglycerine and 2-mononitroglycerine), nitrates as well as perchlorate and bromides. Results presented high concentrations of nitrites, nitrates and perchlorates. An analytical method was also developed to monitor both NG and its degradation products. Samples will soon be processed.

RÉSUMÉ

Des sols de surface provenant des positions de tir anti-char à la roquette M-72 66 mm des bases militaires de Gagetown, au Nouveau-Brunswick, et de Petawawa, en Ontario ont été échantillonnés. Ces sols étaient contaminés par des résidus de poudre propulsive et ont été déposés à la surface de colonne de sols propres provenant de la base militaire de Valcartier, au Québec, et de Petawawa, en Ontario. Ces quatre colonnes ont été arrosées en laboratoire suivant la recharge de l'aquifère observée aux deux sites étudiés. Le fond des colonnes possèdait des orifices permettant l'écoulement et l'échantillonnage de l'effluent. Les contaminants suivis sont la NG (nitroglycérine), ses métabolites, DNG (1,2-dinitroglycérine et 1,3-dinitroglycérine), MNG (1-mononitroglycérine et 2-mononitroglycérine), le nitrate, et le perchlorate et les bromures. Les résultats présentent de fortes concentrations en nitrites, nitrates et perchlorates. Une méthode a également été développée afin de suivre la NG et ses produits de dégradation. Les échantillons seront prochainement analysés.

1 INTRODUCTION

In the last decade an increased environmental awareness has led the Canadian Forces to complete many studies and research projects in order to evaluate the impact of various military activities. Within this context, the topic of the environmental impacts of live firing on training ranges has increased in importance.

The main goal of this project was to study the fate of propellant residues on large soil columns, as no data were available in the literature on this topic, at the exception of two studies published by Hewitt and Mireki (2006). Firing positions sampled in various training ranges across North America have proven to be impacted with high levels of NG or 2,4-DNT from shoulder rocket or gun propellants (Dubé, Jenkins, Thiboutot, 2006) . The study was aimed at characterizing the impacts of military activities related to antitank firing position. The sites selected for the study were the antitank ranges at Garrison Valcartier, Québec, and those at the CFB (Canadian Force Base) Petawawa, Ontario. The shoulder rockets fired on those ranges were propelled by solid propellants based on a nitrocellulose matrix (NC) in which nitroglycerine (NG) and ammonium perchlorate (AP) are dispersed as oxidizer and energetic materials. The combustion processes in the rockets is incomplete, and resulted in the deposition and accumulation of propellant residues in the surface soils. The polymeric NC matrix protects the NG, as its environmental half-life has been determined to be less than a day (Jenkins, 2007) and NC leads to slow desorption processes of the embedded energetic materials. The compounds of interest were NG and its metabolites (1,2-dinitroglycerine, 1,3-dinitroglycerine, 1-mononitroglycerine, 2-mononitroglycerine, nitrates) and perchlorate. Nitroglycerin is a highly toxic substance used as an energetic plasticizer for both gun and rocket propellants (Christodoulatos , 1997). Perchlorates were also included in the study since the propellant formulation of the M72 66mm contains perchlorate as an oxydizer.

For the purpose of the study, the soils containing propellant residues were spread on the surface of four columns containing uncontaminated sand from the Arnhem training range of CFB Valcartier, and from CFB Petawawa. Those soils were watered following the precipitation patterns of their respective region, and interstitial water output of the columns was sampled in order to evaluate the contaminants transport through the unsaturated zone.

2 FIELD AND SAMPLING METHODOLOGY

The sampling was conducted in two steps; the first step consisted in collecting uncontaminated soil samples representative of the geological formations of the two sites (Valcartier and Petawawa), and the second consisted in collecting soils containing high levels of propellant residues behind antitank firing positions, which was later spread across the surface of the uncontaminated soil columns, and which were representative of the contaminated zone. The contaminated soils were collected at CFB Petawawa and CFB Gagetown.

2.1 Soil Sampling

The uncontaminated soil samples used in the column tests are representative of the firing positions on the two antitank ranges. The Petawawa uncontaminated soil sample was collected on November 2006, outside the impact zone and put into 200-liter drums. Drums were first washed using acetone and distilled water in order to eliminate any trace of contamination by energetic material (EM) having potentially occurred prior to sample collection. Moreover, in order to ensure a homogeneous sample, the soil was alternately distributed in the drums during filling. The Valcartier uncontaminated soil was collected in a sand pit next to the Arnhem training range. The drums collected on November 2006, were used to fill three columns.

2.2 Propellant residues sampling

The soils containing high levels of propellant residues were collected on training range A of CFB Petawawa, and at the Wellington training range of CFB Gagetown, New Brunswick. The residues from Wellington Range replaced those from Arnhem, as gravel was laid over the soil at CFB Valcartier firing positions in 2006, thus making their sampling impossible. Clean soil from the Valcartier area was still used in the soil columns, as the contaminated soils from Gagetown represented a similar contaminant source than the one in Valcartier.

The Gagetown propellant residues were collected from firing positions 1 and 2 of Wellington Range on September 2006. The residues were collected over a thickness of approximately 1 cm using a stainless steel spoon. Sampling was performed 1 meter behind the firing position. Sampling at firing position 1 was done within an area of approximately 3 m^2 (fig. 1), while sampling at position 2 was done over an area of approximately 7.5 m^2 . 20-L pails, previously washed with acetone and distilled water, were filled. The soil was sieved at INRS in order to eliminate the grain size fraction superior to 2 mm. The final volume of propellant residues mixed with soil was approximately 10 L.

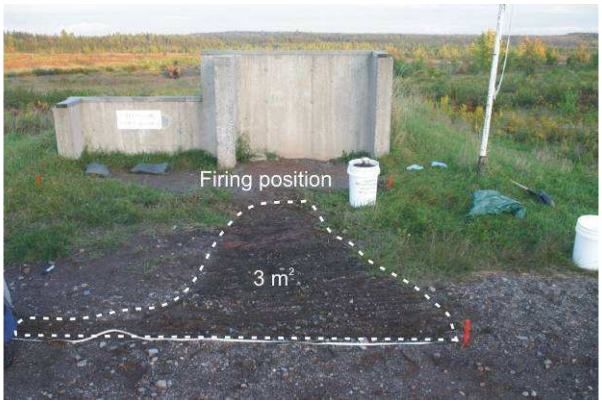


Figure 1. Firing position 1 of the Wellington antitank range, following propellant residue sampling.

The Petawawa propellant residues were collected at firing positions 4 and 5 of the antitank range A, in October 2006. The residues were collected across a thickness of 1 to 2 cm and over an area of 25 m^2, using a shovel, and were stored in 20-L pails. As the soil texture in this area is very coarse, a preliminary sieving with the objective of removing particles superior to 5 mm was achieved, followed by further sieving in the laboratory at INRS in order to remove the particles larger than 2 mm in diameter. The final volume of propellant residues and soil was reduced to one 20-L pail. The samples from the Petawawa and Gagetown firing positions were stored in darkness at 4℃, in order to prevent degradation and/or transformation of the compounds prior to lab test.

3 LABORATORY METHODOLOGY

3.1 Soil column

The columns were built from stainless steel and the inside was layered with Teflon^{MD} in order to prevent any interaction with the EM. The base of the columns consisted of a Teflon^{MD} plate held in place by a steel plate. The Teflon^{MD} plates at the base of the columns were machined with seven outflow holes. Each of these holes was located in the middle of a cone of approximately 1 mm depth and 20 cm diameter, in order to avoid the accumulation of a water lense at the base of the columns. Dimensions of columns are shown in fig 2. The columns dimensions were chosen as to avoid the wall effects, by respecting a height to diameter ratio inferior to 2 (Martel and Gélinas, 1996).

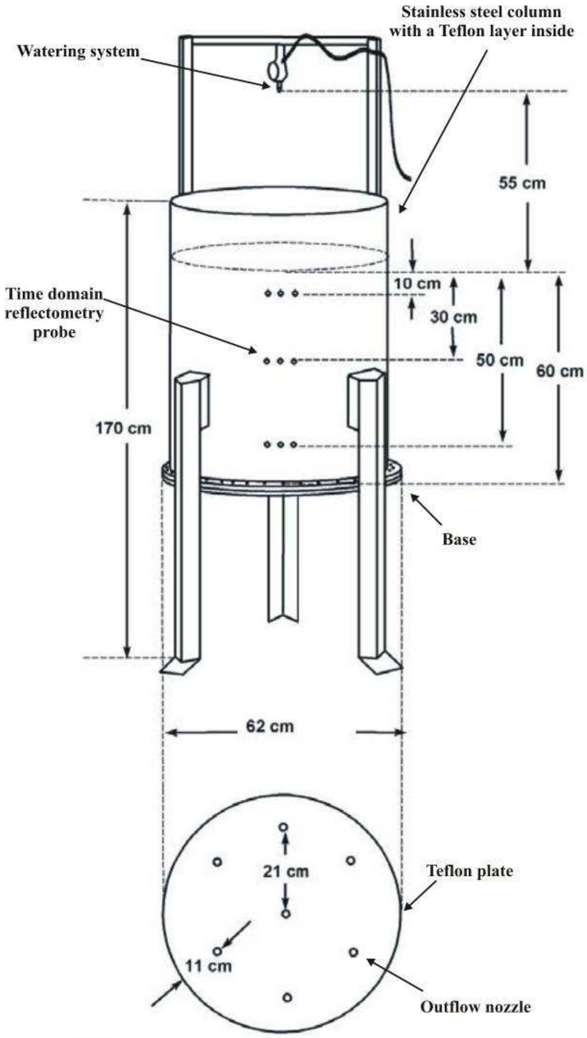


Figure 2. Dimensions of the columns

Fiberglass rods were inserted in each of the seven holes in order to maintain a similar minimum tension in all holes of the soil columns. The rods had an average length of 15.2 cm ± 0.2 from the base of the column to the opposite extremity of the rod. The rods covered the bottom of each cone depression at the base of the column.

The six columns were filled by successive layers of 1.6 cm in thickness, whose mass amount to 8 kg. The clean soil was previously sieved to remove the grain size fraction superior to 8 mm. The column diameter (59.6 cm) was at least 100 times greater than the d_{50} of the sand (0.277 mm for the Petawawa sand, and 0.375 mm for the Valcartier sand), and respected the criterion for avoiding preferential flow (Martel and Gélinas, 1996). The height of soil in each column was 60 cm. A group of three columns containing the same soil was simultaneously filled in order to ensure homogeneity. The water content was determined from the average of the water content values of the collected samples at each day of filling. Each soil layer was compacted using an electrical compactor with a 25-cm circular plate. Before adding the following layer, the soil was scarred with a comb to a depth of around 2 mm, so as to create a hydraulic contact between the soil layers.

The propellant contaminated soils from both sampling sites were applied at the surface of the columns. A mass of 5 kg of uncompacted soil contaminated with propellant residues resulted in a thickness of around 2 cm of residues on each column. The Gagetown residues were placed on the two columns containing the Valcartier soil (column B and C). Column A containing Valcartier soil was used for the application of sodium bromide (NaBr), a non-reactive solute which allows the determination of the retardation factor, and thus of the adsorption coefficient. The NaBr was diluted in distilled water contained in the column watering containers at a constant concentration of around 400 mg/L of Br^-. The propellant residues from Petawawa were placed on the two columns containing the Petawawa soil (column E and F). Column D containing the Petawawa soil was used for the application of NaBr.

The automated infiltration system allowed the exact measurement of the water volume applied to the columns. Each column was fed from an individual 30-L distilled water container simulating rainwater. The containers were connected in parallel to a pressurized air system, which ensures a 30-psi pressure. The flow rates at the nozzles used for watering were similar for each column. The watering rates applied to the columns corresponded to the specific recharge observed at Valcartier or Petawawa. The opening of the valves, and thus of the nozzles of the watering system, was controlled by a programmed CR-10X, which also allowed to measure and record basic parameters (battery voltage, programme signature, temperature) and the water content.

Time domain reflectometry (TDR) probes were placed at three levels in the columns in order to precisely measure the water content in the soil profile. The TDR probes were installed at 10 cm, 30 cm and 50 cm from the base of the columns.

3.2 Chemical analysis

The NG initial concentrations in the soils collected at both firing positions were analytically determined at the DRDC Valcartier laboratory using High Performance Liquid Chromatography (HPLC), following the EPA method 8330b (US EPA, 1996). A representative soil sample was collected from the two sources soils (from Gagetown and Petawawa) after careful homogenization. Neither the acetone slurry nor the grinding was conducted prior to analysis on the whole sample to avoid changing the physicochemical properties of the source. The source soils were homogenized through mixing and sub-sampled at various locations. The samples collected for analysis were then further homogenized with the use of an acetone slurry for homogenization instead of the grinding step. Pierced aluminium paper foil was used to minimize the risk of analyte lost while evaporating the acetone. The practical quantification limit for such analyses was 20 µg/kg in soil and 10 µg/L in water samples. The perchlorate concentrations were analytically determined at the Environment Canada laboratory in Burlington, Ontario, following the LC/MS/MS method, whose practical quantitation limit was 0.6 µg/kg. With these results and knowing the mass of residues deposed on columns (5 kg), the source term for NG and ClO_4^-, were calculated.

4 RESULTS AND INTERPRETATIONS

4.1 Propellant residues

The NG and perchlorate concentrations in the soils collected at firing positions were analytically determined. Also, the source term applied at the surface of the columns was calculated (Table 1). Columns containing soil from Petawawa had Petawawa residues and columns containing soil from Valcartier had Gagetown residues.

Table 1. Nitroglycerine (NG) and perchlorate (ClO_4^-) concentrations in the firing position surface soils and calculated source term at the surface of the soil columns B, C, E and F.

	Propellant residues		Source term	
	NG	$ClO4^-$	NG	$ClO4^-$
	mg/kg residues	µg/kg residues	g	µg
Gagetown (col. B and C)	3145	3.52	15.73	17.6
Petawawa (col. E and F)	5652	53.46	28.26	267.3

4.2 Hydrogeological Characteristics of Soils

The general physical properties of the two soils are listed in Table 2. Both soils consisted of sand with negligible silt and clay fractions. The cation exchange capacity (CEC) was therefore low, as cation exchanges take place on the fine grain size fraction. The carbon content was also negligible for both soils.

Table 2. Properties of the Valcartier and Petawawa soils.

Parameter	Valcartier	Petawawa
Cation exchange capacity ($meq/100g_{soil}$)	1,61	1,87
Total organic carbon (%)	0,1	< 0.1
Pebbles (%)	2	0
very coarse sand (%)	8	< 1
coarse sand (%)	18	5
medium sand (%)	40	49
fine sand (%)	24	33
very fine sand (%)	7	11
silt (%)	< 1	< 1
clay (%)	< 1	< 1
Classification (USDA)	Sand	Sand
d_{50} (mm)	0.375	0.277

The mean water content of each column, average values for pore volume, porosity and bulk density were determined for each soil (table 3). A solid density of 2650 kg/m^3 was used in the calculations. The Valcartier sand, which was coarser than the Petawawa sand, had a lower pore volume.

Table 3. Average values for porosity and density for each soil.

Parameter	Valcartier	Petawawa
Total soil volume (L)	168	168
Pore volume (L)	64	66
Effective porosity	0.259	0.328
Total porosity	0.383	0.390
Bulk density (kg/m^3)	1778	1757

The hydraulic conductivity of soils was measured using a Guelph infiltrometer. The average saturated hydraulic conductivity (K_{sat}) values obtained were $3.75 \cdot 10^{-2}$ cm/s for the Valcartier sand, and $4.55 \cdot 10^{-2}$ cm/s for the Petawawa sand.

The unsaturated hydraulic conductivity of the soil, which depends on the soil saturation level and the tension applied to each column, was also measured with the Guelph infiltrometer. The relatively finer Petawawa sand presented higher unsaturated hydraulic conductivity vs applied tension values than the coarser Valcartier sand (fig. 3). Those results were in agreement with theoretical notions, as finer sand possesses a higher capillarity, and has a lower risk of hydraulic break down.

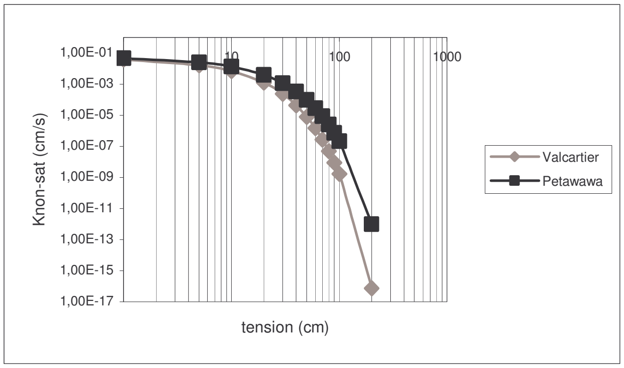


Figure 3. Average unsaturated hydraulic conductivity of the two soils as a function of tension.

4.3 Observed infiltration flow and outflow

The columns were brought to a steady state of flow prior to the experiments. The springtime infiltration period had two significant deviations for the inflow rate. The electric valve on column E jammed open on day 1 of the infiltration, and was stopped the day after. During this 24 hour period, column E was completely flooded. The infiltration for column E was stopped and restored on day 6. Then, the electric valve jammed again and flooded the column. The infiltration for column E was stopped the day after and restored on day 19. The valve was repaired and worked properly after.

The measured infiltrated water quantities were similar to the expected values from each precipitation regime for column A, C, D and F. Leaks in Containers B and E are however visible. Container B had important leaks at the base, which explains the high infiltrated water volume read on the container graduated scale during the first spring infiltration period. Because of the too important leaks, experience with container B was abandoned during first autumn infiltration period. The E container had leaks too, but those were discontinuous. These reservoirs were sealed again during the first summer drying period without noticeable improvements.

The outflow water volumes for the columns A and C (Valcartier) were similar. Column B is not shown on figure 4 because it was abandoned earlier during fall period. However, the outflow water volumes for the columns D, E and F (Petawawa) varied.(fig 4).

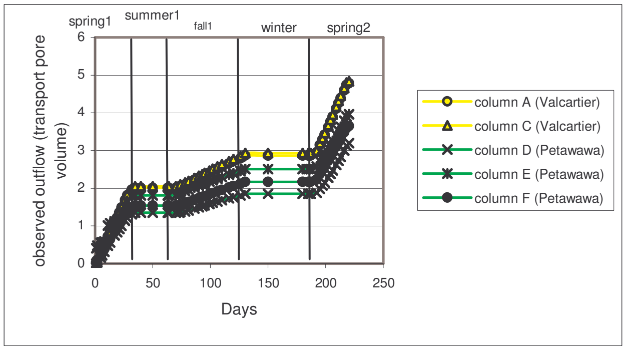


Figure 4. Cumulative measured outflow for each column

Column F was the most conform to infiltration flow. Outflow rate for column D was too slow because of the performance of nozzle. Rather, the watering system nozzle on column E was leaking slightly, increasing the amount of water applied on the column and the amount of water sampled at the bottom of the column. Also, the outflow water volumes for Column E were quite important following the two step flood.

4.4 Time domain reflectometry results

The moisture content in the columns was monitored using time domain reflectometers (TDRs) at 10 cm, 30 cm and 50 cm depths. The data from the TDRs in columns A and F during springtime infiltration is shown on figures 5 and 6. Precipitations were scheduled every 15 minutes for eight hours a day.

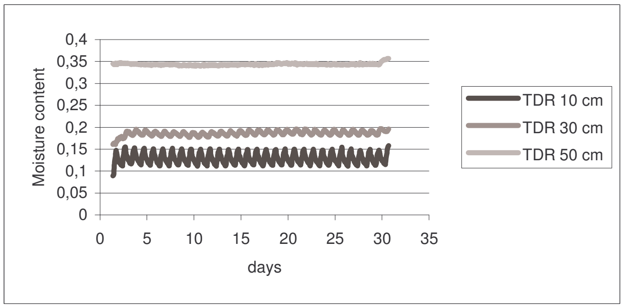


Figure 5. TDR data from springtime infiltration in column A – Valcartier

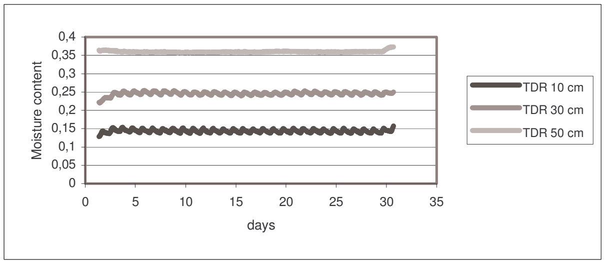


Figure 6. TDR data from springtime infiltration in column F – Petawawa

The transient daily fluctuations in moisture content were visible during this period. The flow, although transient, was at equilibrium. This means that from one day to the next, the overall moisture content did not increase or decrease during the springtime flow. In other words, the daily fluctuations did not show long-term upward or downward trends during the springtime infiltration period. However the magnitude of changes in moisture content between column A and F were quite different (Fig. 6-7). Two reasons explain this difference. First, the patterns of infiltration on the two sites were different. The recharge at Valcartier was more important than at Petawawa (369 mm/year compared to 237 mm/year). Also, the Petawawa soil texture was finer than Valcartier, therefore the water was more closely related to soil particles and the residual moisture content was higher.

The average moisture content at 10, 30 and 50 cm in column A are presented on figure 7. The water content in the soil decreased significantly at 10 and 30 cm after the first springtime infiltration. This drop occurred rapidly at the beginning of the drying period and moistures reached equilibrium afterwards. Also, at the beginning of the second springtime infiltration, the moisture quickly returned to its maximum value. The phenomenon of moisture variation was less pronounced following the first autumn infiltration since precipitations are less abundant than in the springtime. In contrast, at 50 cm, no variation of moisture was noted. Given the little difference between the values of moisture at 50 cm and the value of porosity, it is possible that the soil has reached saturation. Finally, due to a programming error, moisture data for the early autumn was not available.

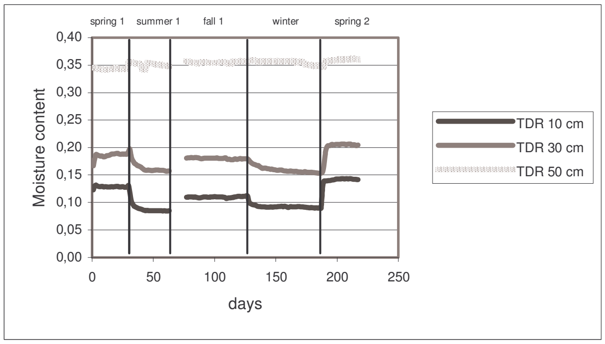


Figure 7. TDR data of column A – Valcartier

4.5 Solute transport results

The graphical data are presented with respect to transport pore volumes eluted. In the saturated regime, one pore volume is the quantity of water required to completely fill the pore spaces of a given quantity of soil. Most contaminant transport studies are performed in saturated conditions, and the concept of pore volume is commonly used in the literature to describe the quantity of fluid which has passed through a saturated soil column (Lewis, 2007). Thus, the breakthrough of fluid travelling at the average linear velocity is achieved when one pore volume has passed through a column of soil. Of course, in the unsaturated regime, the pores are filled with both water and air, so one pore volume will not represent the point at which all the water in a column has been theoretically replaced. Pore volume of transport in the unsaturated zone is found using a tracer (fig. 8).

Indeed, a transport pore volume, for a continuous source of solute, is attained when concentrations sampled at the bottom of the columns correspond to half of the initial concentration. Transport pore volume was 33 and 28 L for Valcartier and Petawawa respectively. The significantly higher concentrations corresponded to the first sample following a period of drying (fig. 8). The water in the fiberglass rods evaporated during the drying period, thus concentrating the Br-. This phenomenon was more pronounced for samples of the Petawawa soil since the volumes of water at each sampling period were less important due to the precipitation regime therefore, the dilution was lower.

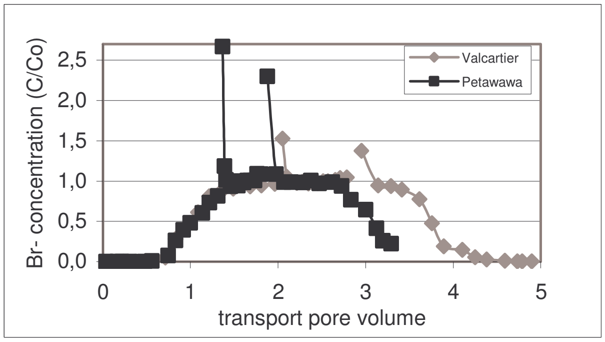


Figure 8. Tracer (Br^-) concentration vs. transport pore volumes from column A – Valcartier and D- Petawawa

Perchlorate concentrations versus transport pore volume were first plotted (fig. 9). This data will provide insight into the behaviour of a solute as it passes through the unsaturated soil columns, without any adsorption or transformation losses. Thus, perchlorate can almost be seen as a pulse of tracer (compared to a continuous source) in this experiment. Perchlorate concentrations were followed for column F taking into account the higher initial concentration of perchlorates for residues from Petawawa (53.46 µg/kg) compared to Gagetown (3.52 µg/kg). A sharp rise in concentration was observed even at 0.6 transport pore volume for this element. The maximum was observed at approximately 1,2 transport pore volume. The perchlorate curve was then compared to the bromide curve, and the results demonstrate that perchlorate was not retarded.

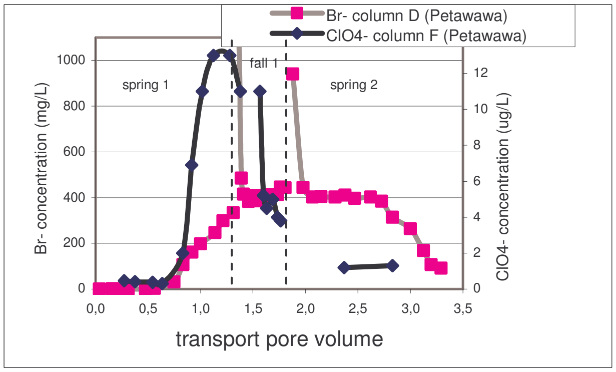
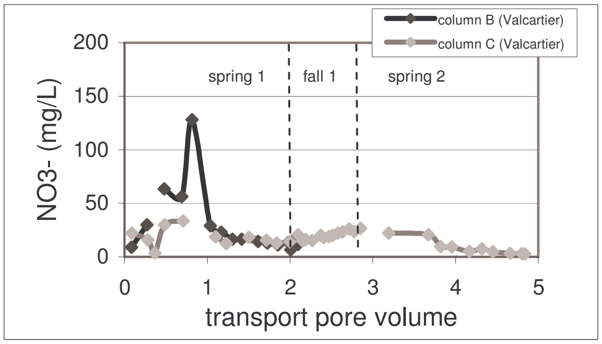


Figure 9. Perchlorate (ClO_4^-) leachate concentration vs. transport pore volumes from column F – Petawawa

The samples were also analysed for their nitrate concentration (fig. 10-11). In the literature, the degradation of NG does not produce nitrate because experiments were done under anaerobic conditions or under conditions where the optimal mass of bacteria capable of using nitrate as a source of energy were not reached (Wendt and al. 1978, White and al. 1996, Chrsitodoulatos and al. 1997, Accashian and al. 2000) . Results were different in the present study because the fate and transport processes of NG took place in aerobic condition.

Nitrate concentrations for columns B and C followed a similar trend (fig.10). The data collection for column B has been stopped at the beginning of the first autumn infiltration period, as the column was abandoned. The nitrate concentration results for column B have shown a significant nitrate production at the beginning of experiment. The nitrate results for column C have shown a slight nitrate production between 2.5 and 3.8 transport pore volume. The average nitrate concentration in samples from column A, the reference column, was 2.4 mg / L. This value can be used has the background.



FFigure 10. Nitrate (NO_3^-) leachate concentration vs. transport pore volumes from columns B and C (Valcartier)

Nitrate concentrations for column E were not regular, probably due to the two episodes of flooding at the beginning of experimentation (fig.11). However, nitrate concentrations for column F have shown the first nitrate production at the end of the first spring infiltration period. Then, a second phase of nitrate production was observed with a maximum at approximately 2.2 transport pore volume at the second spring. The average nitrate concentration in samples from column D, the reference column, was 6.4 mg / L. This value can be used has the background.

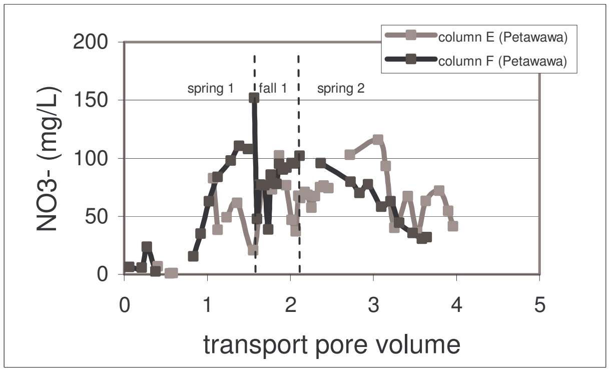


Figure 11. Nitrate (NO_3^-) leachate concentration vs. transport pore volumes from column E and F (Petawawa)

Finally, nitrate and perchlorate concentrations were compared (fig.12). Perchlorate is an ion which is neither retarded nor transformed during its transport through the unsaturated zone. The first maximum in concentration for nitrate has arrived shortly after the maximum for perchlorate. However, nitrate concentrations can only be explained by a nitrate production from NG. Indeed, the initial nitrate concentration in the residual propellant was not known, but was presumed to be low, as nitrate is a compound quickly washed through the unsaturated zone. Finally, the second nitrate peak maximum was clearly the result of a nitrate production from NG because no second maximum appeared on the perchlorate curve.

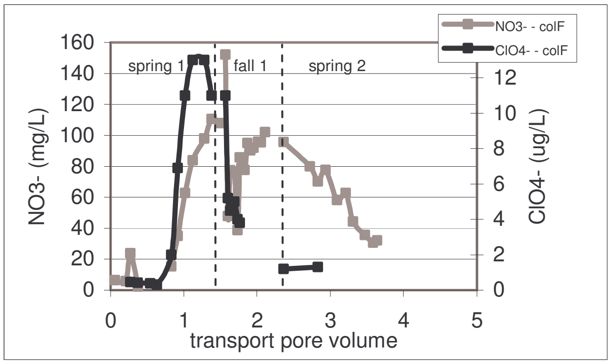


Figure 12. Nitrate (NO_3^-) and perchlorate (ClO_4^-) leachate concentration vs. transport pore volumes from column F (Petawawa)

Samples were also collected for general EM analysis, and they were frozen at -20°C due to analytical workload. They will soon be processed. An analytical method was also developed to monitor both NG and its degradation products. This will allow the better understanding of NG degradation pathways in aerobic conditions.

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