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Decomposition of Paper De-inking Sludge in a Sandpit Minesoil During its Revegetation

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DECOMPOSITION OF PAPER DE-INKING SLUDGE IN A SANDPIT MINESOIL DURING ITS REVEGETATION

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Summary—Paper de-inking sludge was used as an organic amendment for revegetating an abandoned sandpit in Québec, Canada. In situ patterns of sludge decomposition and of total nitrogen (N) and phosphorus (P) dynamics where characterized in a litter bag study. In a one-time operation, sludge was applied at a rate of 0 and 105 Mg dry matter ha^{-1}, along with N at 3, 6 or 9 kg Mg^{-1} sludge and P at 0.5 or 1.0 kg Mg^{-1} sludge. Sludge and fertilizers were incorporated into the top 0.21 m of the minesoil and tall wheatgrass (*Agropyron elongatum* (Host) Beauv.) was seeded. Mass loss was well described by a double exponential model when cumulative degree days (sum of daily temperature above 0 ° C) were used as the independent variable. Fifty one percent of the initial material decomposed with a half life of 0.4 year, whereas the remaining material had a much slower rate of decay with a half life of 13 years. The large size and slow decomposition rate of the recalcitrant pool of this material were attributed to the high lignin content and the presence of clay in the sludge. Both N and P in decomposing sludge presented a short accumulation phase followed by a long release phase wich likely contributed to the succesfull revegetation of this disturbed sandpit site.

Keywords : organic amendment, carbon mineralization, nitrogen, phosphorus, *Agropyron elongatum,* tall wheatgrass.

INTRODUCTION

In minesoils and other severely disturbed soils, decomposition of recently deposited plant residues or other inputs and associated processes are particularly crucial since they are closely related to nutrient availability and to the rate of soil organic matter accumulation. Such microbially-mediated processes are initially inhibited in minesoils compared to undisturbed soils, but the amelioration of soil functions is frequently observed as organic carbon (C) and nitrogen (N) accumulate through revegetation (Visser *et al.,* 1984). The use of organic amendments for reclamation of minesoils has been gaining popularity in recent years. However, general knowledge of their decomposition in such soils is still very limited, in spite of the critical nature of these processes to the viability of a plant cover.

Patterns of decomposition and of nutrient loss and retention of decaying substrate are primarily a function of the quality of the decaying substrate itself, and of the prevailing climatic conditions and soil properties (Berg and Agren, 1984; Polglase *et al.,* 1992; Vanlauwe *et al.,* 1997). In addition, decomposability of organic substrates can be contrastingly affected by the availability of N and phosphorus (P), either from endogenous or exogenous origin (Prescott, 1995; Recous *et al.,* 1995; Cheshire and Chapman, 1996). A prerequisite for optimal management of a particular organic amendment in a revegetation context, is a proper knowledge of its behaviour after its incorporation into minesoil. The ultimate goal is successful vegetation establishment and persistence, with minimum nutrient loss. In the present study, paper de-inking sludge was evaluated as an organic amendment for the revegetation of an abandoned sandpit. De-inking sludge is composed of wood fiber but also contains fillers, ink and the chemicals used to dissociate these materials from the pulp fiber (NCASI, 1992). We hypothesized that high application rates of de-inking sludge can restore nutrient cycling within disturbed soil ecosystems when sufficient N and P are provided. In a litter bag decomposition study, we investigated C, N and P dynamics fallowing sludge incorporation at high rates into a sandpit minesoil, during the establishment of a perennial grass cover. The specific objective was to characterize *in situ* patterns of sludge decomposition and of N and P accumulation and release, as affected by increasing rates of N and P.

MATERIAL AND METHODS

Study Site and Plot Establishment

The research was conducted in an abandoned sandpit located at St-Lambert-de-Lévis, Québec, Canada (46°34' N, 71°13' W). The mean annual temperature is 4 ° C and mean total precipitation is 1200 mm of which about one third is snow. The minesoil was recently exposed after removal of topsoil and mining of subjacent layers of sand down to a depth of about 2 m. The minesoil contained approximately 94% medium-textured sand with very little gravel; other properties are presented in Table 1.

De-inking sludge was obtained from the Daishowa Inc. paper mill (Québec, Québec, Canada) (Table 1). In a one-time operation, raw paper de-inking sludge was mechanically incorporated with a rotovator into the surface 0.21 m of soil at two rates (0 and 105 dry Mg ha^{-1}), supplemented for both treatments with N at three rates (315, 630 and 945 kg N ha^{-1}; i.e., 3, 6 and 9 kg Mg^{-1} sludge) and with P at two rates (52.5 and 105 kg P ha^{-1}; i.e., 0.5 and 1 kg Mg^{-1} sludge). Levels of sludge, N and P were selected based on the results of greenhouse trials, and were within ranges for adequate plant growth (Fierro *et al.,* 1997). The sludge application rate of 105 dry Mg ha^{-1} corresponds roughly to 30% sludge by volume over the incorporation depth. Nitrogen was applied as urea and P as single superphosphate that contains 10 to 12% of S. Thus, sulfur was not considered as a limiting factor for plants or microorganisms. In addition, de-inking sludge contains about 0.02% of S. Potassium chloride was applied uniformly to all plots at 85 kg K ha^{-1}. Tall wheatgrass (*Agropyron elongatum* (Host) Beauv., Canada No. 1; 42% germination) was drill-seeded at a density of 680 pure live seeds m^{-2}. The decomposition study was conducted on sludge-amended plots only. The experimental design was a split-plot with four replications, and with N treatments in main plots and P treatments in subplots. Each plot was 4 x 4 m.

Field Methods

Ten g of air-dry raw de-inking sludge was enclosed into each of 360 litter bags (< 0.01 m x 0.15 m x 0.15 m). A mesh opening of 0.5 mm was selected in order that losses of sludge, due to handling, would be minimized while allowing free access to microflora and microfauna decomposers (Swift *et al.,* 1979). On the day of sludge and fertilizer incorporation, 15 litter bags were randomly placed in each plot. No fertilizer was added to sludge in litter bags. They were buried vertically in the soil in the 3 to 18 cm soil layer. After approximately 1, 3, 10, 16 and 27 months, three litter bags per plot were retrieved and oven-dried at 50 °C for 72 h. This low drying temperature was used to minimize N volatilization. After drying, large roots were carefully removed and litter bag contents were weighed individually.

Soil temperature of each plot was recorded every 6 h at 8 cm depth with thermocouples and a datalogger system (CR10, Campbell Scientific Inc., Logan, Utah).

Analytical Procedures

Further analyses of decaying sludge were conducted on the pooled contents of the three litter bags retrieved from each plot at a given date. The pooled samples were ground to pass a 1-mm sceive and subsampled to determine water content (105 ° C for 24 h) and ash content (500 ° C for 8 h). Total N and C contents were determined by dry combustion (CNS-1000 analyzer, LECO Co. St. Joseph, MI), and total P by acid digestion and colorimetry (Tandon *et al.,* 1968) with a predigestion in HNO_3 (Olsen and Sommers, 1982).

Organic chemical composition of sludge was determined by the detergent fiber method (Goering and Van Soest, 1970) in which dried sludge was successively extracted with a neutral-detergent solution, an acid-detergent solution and sulphuric acid. This yielded the following organic fractions : cellulose, acid-detergent lignin, hemicellulose and neutral-detergent soluble fraction.

Data Analyses

The sludge analyses were corrected for soil infiltration in litter bags (ranged between 1 and 8% for all sampling dates) as follows for a given sampling date (Blair, 1988; Schuman and Belden, 1991):

$$M_r = M_f \, [(A_s - A_f)/(A_s - A_i)]$$

where M_r is the dry mass remaining of sludge (grams), M_f is the final dry mass of litter bag contents (grams), A_s is the original ash percentage of minesoil, A_f is the final ash percentage of litter bag contents, and A_i is the initial ash percentage of sludge. This correction roughly corresponded to calculating mass loss as percentage of ash free dry matter remaining since the soil had a very low organic matter content (0.2%). In addition, P and N concentrations of litter bag contents were corrected for indigenous soil P and N contents (Blair, 1988), respectively. However, contamination with soil N was negligible since the content in soil was extremely low.

Analysis of variance on repeated measurements were conducted using the GLM procedure and the repeated statement in the SAS statistical package (SAS Institute, 1988) on full data sets of percent mass remaining, N, P and C-to-element ratios. Sphericity test was rejected when $P<0.001$.

For kinetic analysis of decomposition, temperature was expressed in degree-days (Honeycutt *et al.,* 1988). These were obtained by summary daily mean soil temperature above 0

° C. A two-compartment first-order model (Voroney *et al.,* 1989; Jenkinson, 1977) was used to describe mass loss of sludge:

$$M_{remaining} = M_1 \exp(-K_1 T) + M_2 \exp(-K_2 T)$$

where $M_{remaining}$ is the mass of sludge remaining (%), M_1 and M_2 are the sizes of the two compartments (% of input), K_1 and K_2 are the decomposition rate constants of each compartment (degree-days^{-1}), and T is cumulative degree-days. Equation constants were obtained using a non-linear curve fitting software (Table Curve 2D, AISN Software Inc., Jandel Scientific, San Rafael, CA). The reconversion of degree-days to years was done using the two-year mean value of 3119 degree-days year^{-1}.

Linear regression analysis was used to relate mass loss to N and P concentrations, and to describe N release from sludge. Parameters of N and P dynamics were estimated from a series of linear regressions using the approach described by Aber and Melillo (1982). All data are reported on an ash free dry mass basis unless otherwise indicated.

RESULTS

Decomposition of Sludge and Changes in its Organic Fractions

Initially, cellulose comprised about half the ash-free dry mass of raw sludge, acid-detergent lignin accounted for about one quarter, and hemicellulose and neutral-detergent soluble fraction for the remaining (Table 1). Total mass remaining was not significantly affected by N or P treatments, at any time; therefore, only the overall means are presented (Fig. 1A).

Decomposition occurred rapidly in the first three months, with more than 30% of initial mass loss. Sixteen months after incorporation, sludge had lost more than half of its initial mass and reached a phase of slower decomposition. From that time to the end of the study which was almost 12 months later, only an additional 4% of initial mass was lost. After 27 months in the soil, the average total mass of sludge remaining was 43.4%.

Mass loss was almost negligible when soil temperature was below 0°C. The nearly horizontal sections of the mass loss curve corresponded well with the two winters through which this study was conducted (Figs. 1A and 1B). *In situ* net decomposition was well described by the double exponential model when degree-days were considered as the independent variable (Fig. 2). Fifty-one percent of the initial material was mineralized relatively quickly with a decay rate of $5.7\ 10^{-4}$ degree-days^{-1} which corresponds to a half-life of 0.4 year (where one year = 3119 degree-days^{-1}) and a mean residence time near 0.6 year. The remaining 49% of the initial material mineralized more slowly (by a factor of 30) with a decay rate of $1.7\ 10^{-5}$ degree-days^{-1} corresponding to a half-life of 12.9 years and a mean residence time of 18.6 years.

Nitrogen and Phosphorus Dynamics

Total N dynamics in the decaying sludge followed a two-phase pattern (Fig. 3A). A net accumulation of N occurred during the first three months (~1400 degree-days) which was followed by a release of N. Nitrogen dynamics were significantly affected by N treatments; net accumulation increased with N application rates. The inverse linear functions between mass loss and N concentration in remaining sludge were used to determine the variables of N dynamics (Table 2). The maximum amount of N accumulated per unit of initial sludge mass (N_{max}) varied from 1.4 to 2.1 mg g^{-1} from the lowest to the highest N application rate, respectively and occurred within the third month. The total N content of sludge at N_{max} was 5.8, 6.8 and 7.4 mg g^{-1} for the low, intermediate and high N rate, respectively. The onset of N mineralization (at N_{max}), occurred at 821 degree-days for the low N rate, at 1043 degree-days for the intermediate N rate and at 1225 degree-days for the high N rate.

Nitrogen release from sludge (after N_{max}) was adequately described by a linear function when degree-days was used as independent variable (Table 3). Nitrogen was released slightly faster and in larger amounts when N was applied at the two highest rates, compared to the lowest rate. After 27 months in the soil, when less than 44% of the initial sludge mass was left, the N content of the remaining material corresponded to 75, 84 and 92% of its original amount, when N was applied at 3, 6 and 9 mg g^{-1} sludge, respectively.

The C/N ratios declined throughout the first 16 months when they reached values near 65, thereafter they remained rather constant (Fig. 3B). The critical C/N ratio (at N_{max}), when net release began, varied from 82 to 70 for the lowest to the highest N rates, respectively. The initial ratios (61, 41 and 31), as calculated from initial C and N contents of sludge and N additions, were never attained. During the initial three months, the sharp decrease in the C/N was due to both net N accumulation and C mineralization. From that time, further decreases in C/N were due to C mineralization only since N was no longer accumulating in sludge (Fig. 3A).

The pattern of net P accumulation and release also presented two phases, but a moderate net re-immobilization was observed from month 16 (~4600 degree-days) onwards (Fig. 4A). In contrast to N, P dynamics were not affected significantly by P or N treatments, therefore, only the overall means are presented. As done for N, the inverse linear function between mass loss and P concentration was used to derive the variables of P dynamics. This regression was: Y = -

1171 X + 138 ($r^2 = 0.93$, $P<0.01$), where Y is mass remaining of sludge (% of initial) and X is P concentration of sludge (%).

The maximum amount of P accumulated (P_{max}) was 0.16 mg g^{-1} sludge. The total P content of sludge at P_{max} was 0.6 mg g^{-1}. The amount of P immobilized per gram of dry matter lost (P equivalent) was 0.5 mg g^{-1}. The onset of P release (at Pmax) was observed after about 1490 degree-days, which is slightly later than the onset of N mineralization. In contrast to N, the phase of P release was not linear and P release rates were not calculated. After the critical C/P (882) was reached and net P release began, ratio decreased more slowly (Fig. 4B). At the end of the study, the absolute amount of P in the remaining material was close to the original amount.

DISCUSSION

Net decomposition of sludge presented a two-phase pattern. The total mass remaining had a rapid early decrease followed by a much slower one. This recalcitrant pool of sludge decomposing in the minesoil was considerably larger than those reported for less lignified substrates such as straw decomposing in non-degraded soils (20-30% in Voroney *et al.,* 1989; 33% in Aita *et al.,* 1997). Its half-life was also longer, about 13 years compared to 6 to 9 years under various climatic conditions (reviewed by Voroney *et al.,* 1989) and about 3 years (Aita *et al.,* 1997) for wheat straw. The lignin content of the sludge could contribute to the large size of the recalcitrant pool. The slow decomposition of this pool may also be partially attributed to the clay present in the sludge, which is derived from paper fillers (NCASI, 1992). There is about 5 to 15% of clay, as kaolinite, in this sludge (C. J. Beauchamp, unpublished results). The clay is spread around wood fibers which could have been increasingly coated as decomposition proceeded, becoming physically protected from microbial attack. The role of clay in protecting residues and metabolites from decomposition has been recently reviewed by Ladd *et al.,* (1996). Skene et al. (1996) found that kaolin could slow the rate of mineralization of straw. Physical isolation of decomposing sludge in the litter bags might also have slowed their decomposition. However, Fierro et al. (1999c) have confirmed the slow decomposition of this material based on whole-soil C and physical fraction measurements.

Our results on mass loss provide no statistical evidence that increased N or P availability accelerated or delayed decomposition, as it has been contradictorily reported for N (Bremer *et al.,* 1991; Recous *et al.,* 1995; Cheshire and Chapman, 1996) and for P (Amador and Jones, 1993; Cheshire and Chapman, 1996). In fact, only a trend (not shown) was noticed as slightly more material (~ 6% of initial amount) remained with the highest N application rate. However, measurements of whole soil and particulate C confirmed the positive effect of N on C conservation in the same study (Fierro *et al*., 1999c)

Nitrogen immobilization was expected to be substantial in the early stages of decomposition, because of the high initial C/N ratio of sludge. Supplemental N was forethought to compensate for immobilization while allowing vegetation establishment. Anticipated microbial immobilization can be roughly estimated considering that fungi are the primary decomposers of sludge, with a C assimilation efficiency of 30% and C and N contents of 50 and 5% dry mass, respectively (Boyd, 1982). This yields a theoretical net N immobilization of 8.1 mg g^{-1} sludge which is far more than the maximum amount of N accumulated observed in this study (1.4 to 2.1 mg g^{-1} sludge). Indeed, less N may be required during decomposition when more C is unavailable or slowly available to microorganisms (Bremer *et al.,* 1991). Also, the populations of diazotrophic bacteria may be stimulated by the de-inking sludge and favor dinitrogen fixation as reported by Halsall and Gibson (1989) during residue decomposition. An increase in population of diazotrophic bacteria was noticed in the rhizosphere of plant grown in de-inking sludge compost (Lessard and Beauchamp, unplublished data). Moreover, a high nitrate leaching potential was noticed during the first three months (Fierro *et al.,* 1999b) and, therefore, significant amounts of N may have been lost prior to microbial assimilation. Values of N_{max} were, however, close to those reported for grassland (Seastedt *et al.,* 1992) and forest (Melillo *et al.,* 1989) litter, but no N was added to those residues. More N accumulated in decomposing sludge as the N rate increased, a similar response was reported with lentil and wheat straw (Bremer *et al.,* 1991). This may have been due to 1) more N immobilized by a growing microbial biomass with a lower C/N ratio (i.e. a lower microbial N efficiency, see N equivalent in Table 2), and 2) more N in solution (i.e. NO_3^-, NH_4^+) was initially absorbed by the sludge, which has a high water retention capacity, thus allowing more N to be taken up by microorganisms or absorbed by sludge.

Net N release began when C/N ratios were about 70 to 80. This critical ratio was higher than is commonly expected for less lignified substrates (Swift *et al.,* 1979). More lignified substrates like pine needles or wood chips, presented high critical ratios that were close to those reported here for sludge (McClaugherty *et al.,* 1985; Melillo *et al.,* 1989). After attaining their critical values, the C/N ratio of remaining sludge continued to decline until it reached a steady state at a value near 65. This ratio was reached within the slow decomposition phase and remained constant thereafter. Total net release of N from sludge increased with N rates and it certainly supported the better growth observed with the highest N rates (Fierro *et al.,* 1999a).

This higher amount of N released was caused by a higher accumulation (i.e. higher N_{max}) combined with a faster release rate.

In spite of a roughly similar two-phase pattern, P dynamics differed from N dynamics in some aspects. Various soils would have various retention of P fertilizer according to the quantities added to the soil, aluminum and iron contents, soil pH, interactions with other soil nutriments, etc., whereas N would be more mobile. Nevertheless, the amount of P remaining at the end of the study was close to the original amount, in other words, total net P release from sludge was equivalent to P accumulated (P_{max}), which was about 0.16 mg g^{-1} sludge. Loss of P was continuously slower than that of C, as indicated by generally decreasing C/P ratios. A similar trend was observed under a no-till system for wheat residues (high initial C/P ratio), but not for residues with lower ratios where no trend was apparent (Buchanan and King, 1993). Carbon and P losses were not expected to parallel each other during decomposition of sludge, since organic P is probably mineralized independently from C (McGill and Cole, 1981). As supporting evidence of uncoupled C and P losses from sludge during this study, the figures 4A and 1 show that the bulk of P release occurred between the third and fourth sampling when little decomposition was observed since it was the first winter.

In conclusion, sludge presented a particularly large recalcitrant pool decomposing slowly. After 27 months in the soil, the remaining mass of sludge was still 43 % of its initial amount. During decomposition, sludge was a continuous source of C, N and P in the sandpit that likely contributed to the successfull re-establishment of a vegetable cover. Nitrogen and P mineralizing from decomposing sludge can, therefore, regulate primary productivity in the early phases of revegetation of abandoned sands until organic matter derived from vegetation could accumulate sufficiently to support the restoration of nutrient cycling in the soil-plant-microbial ecosystem.

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Table 1. Selected initial characteristics of sandpit minesoil and paper de-inking sludge.

Parameter	Minesoil	Sludge
Total C (mg g^{-1})	1.3 ± 0.5^{z}	382 ± 1
Total N (µg g^{-1})	211 ± 65^{z}	3300 ± 290
Total P (µg g^{-1})	413 ± 4	252 ± 44
Water content at -33 kPa (g g^{-1})	0.06 ± 0.01	0.65 ± 0.01
pH ($CaCl_2$)	4.7 ± 0.1	8.6 ± 0.1
Ash (mg g^{-1})	992.6	250.4
Cellulose (mg g^{-1})^{y}	ND	474.4± 13.8
Acid-detergent lignin (mg g^{-1})^{y}	ND	273.5± 17.0
Hemicellulose (mg g^{-1})^{y}	ND	125.4± 18.4
Neutral-detergent soluble fraction (mg g^{-1})^{y}	ND	126.7± 18,7

[z] : means of 16 individual samples; all other values are means of four composite samples (± standard deviations).

[y] : Ash free dry matter.

Table 2. Inverse linear functions relating mass loss of de-inking sludge and N concentration in the remaining material, and derived variables on N dynamics, as affected by N treatments.

N application (mg g^{-1})	Slope	Intercept	r^2	N_{max}[z] (mg g^{-1})	N_{eq}[y] (mg g^{-1})	Critical[x] C/N
3	-136.5	159.4	0.99***	1.4	6.8	82
6	-111.7	151.3	0.99***	1.8	7.3	74
9	-98.3	145.7	0.98**	2.1	7.5	70

[z] : maximum amount of N immobilized per gram initial material

[y] : N equivalent or amount of N immobilized per gram dry matter lost

x : C/N ratio at which N release from sludge begins

, *: significant at $P < 0.01$ and 0.001, respectively

Table 3. Total N released from sludge, N release rates (slopes) and coefficients of determination from the linear regressions between N remaining and degree-days over the phase of net N mineralization (after reaching the critical C/N ratio), as affected by N treatments.

N application (mg g^{-1})	Total net N release (mg g^{-1})	N release rate (degree-$days^{-1}$)	r^2
3	2.5	0.0047	0.95*
6	3.0	0.0052	0.97*
9	3.3	0.0052	0.98*

*: significant at $P < 0.05$

LEGENDS

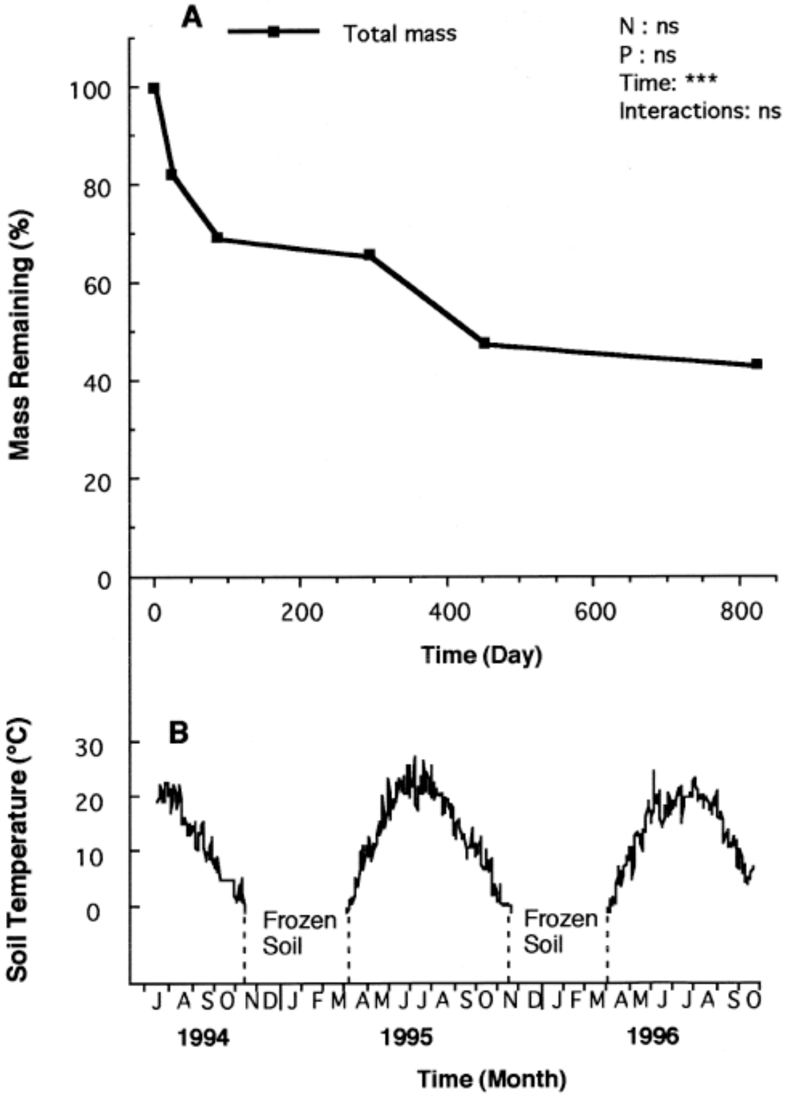

Figure 1. A) *In situ* net decomposition of de-inking sludge fractions; N and P treatments were pooled (n = 24). B) Mean soil temperature (8 cm depth) in sludge amended minesoil (n = 24). Sources of variation are presented for both total mass fractions; ns and ** : not significant and significant at *P*<0.001, respectively. Coefficient of variation (C.V.) was 6.4%.

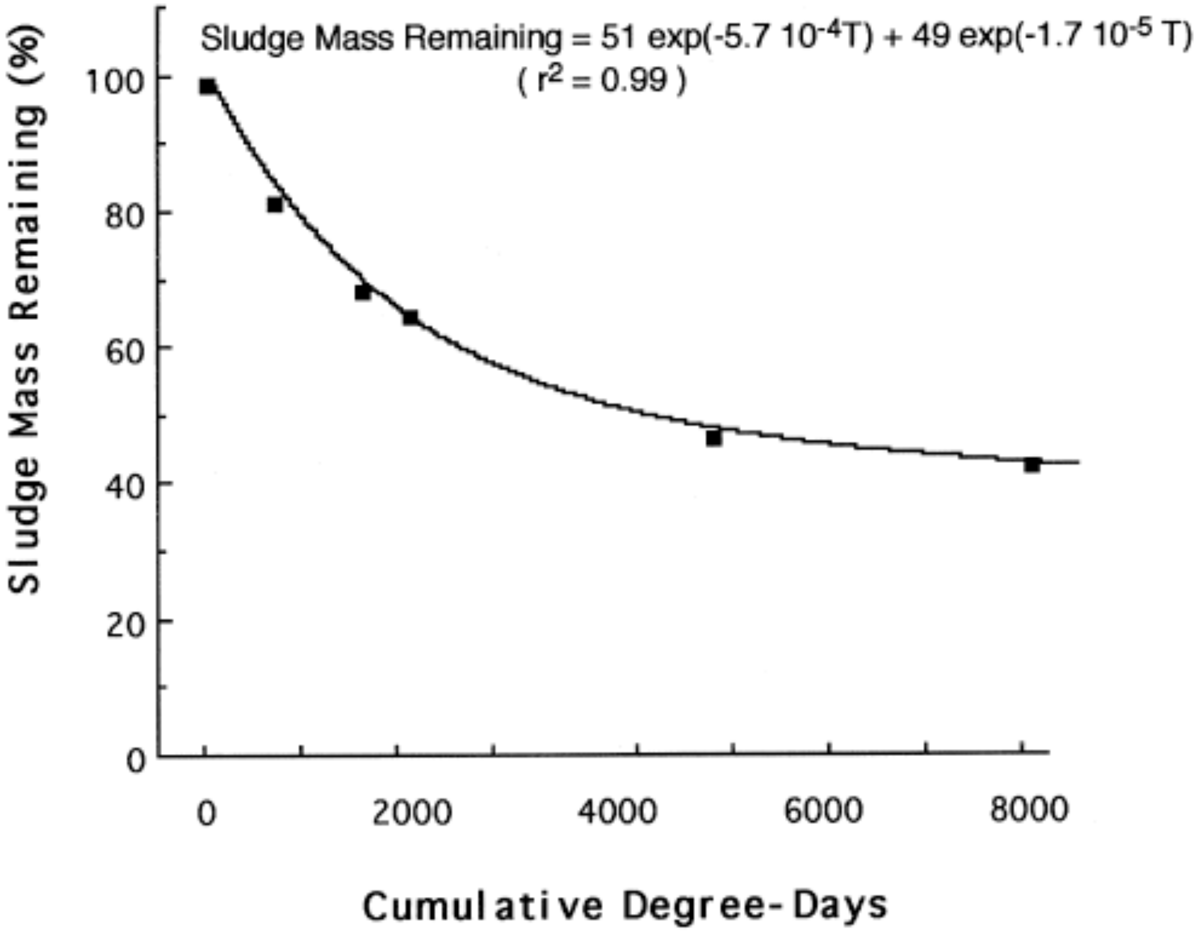

Figure 2. Mass of de-inking sludge remaining in litter bags as function of cumulative degree-days; N and P treatments were pooled (n = 24). T : degree-days, n : observed, — : simulated.

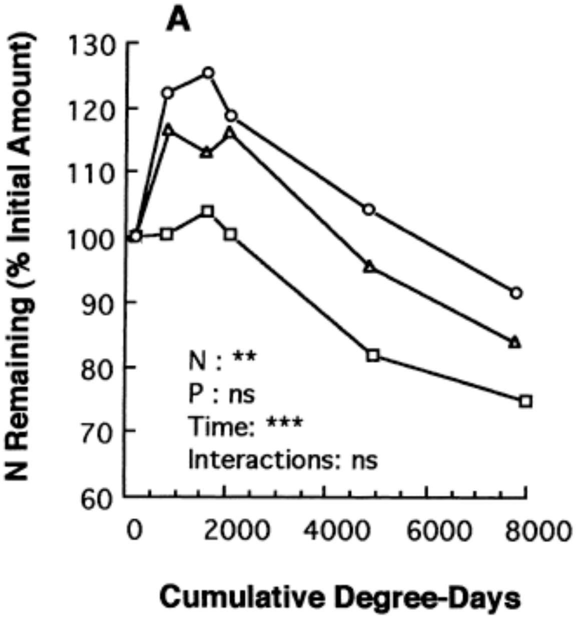

Figure 3. A) Changes in de-inking sludge N, as function of cumulative degree-days. B) Changes in mean C/N ratio of decomposing sludge during the 823-day period; P treatments were pooled (n = 8). ns and *, **, *** : not significant and significant at *P*<0.05, 0.01 and 0.001, respectively. C.V. = 8.3%.

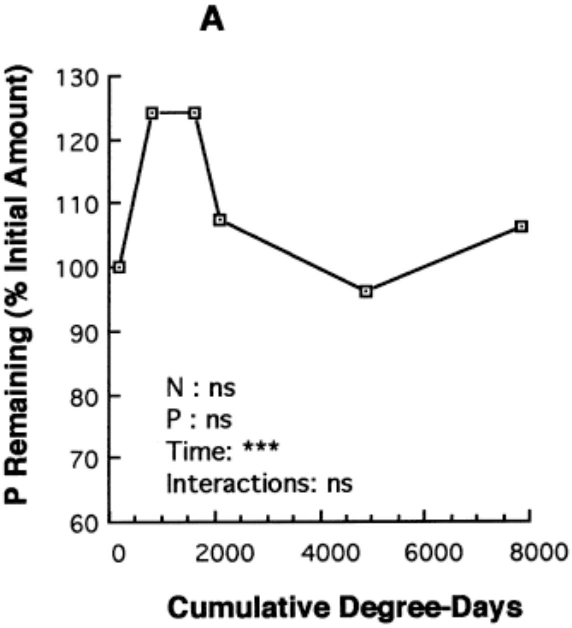

Figure 4. A) Changes in de-inking sludge P, as function of cumulative degree-days. B) Changes in mean C/P ratios of decomposing sludge during the 823-day period; N and P treatments were pooled (n = 24). ns and *** : not significant and significant at $P<0.001$, respectively. C.V. 22.7%.