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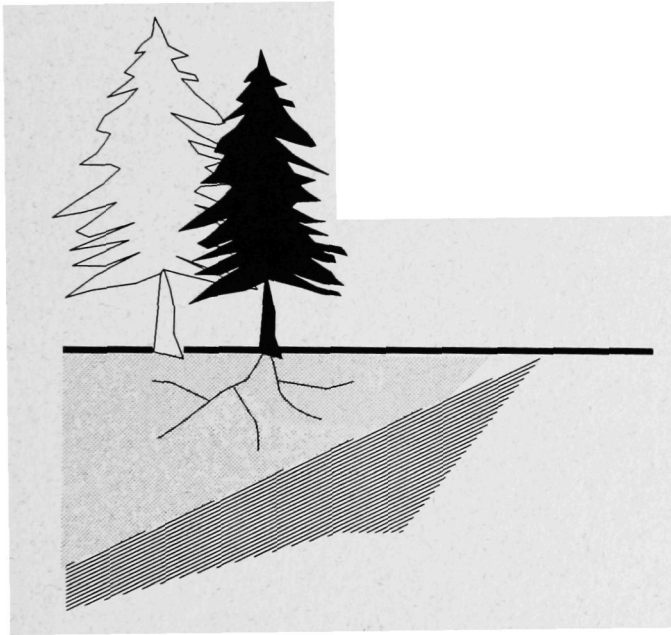
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Variability Factors Involved with Land Application of Papermill Sludge

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MAINE AGRICULTURAL EXPERIMENT STATION

VARIABILITY FACTORS
INVOLVED WITH LAND APPLICATION OF
PAPERMILL SLUDGE

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INTRODUCTION

Large amounts of wastewater sludge are produced annually by the paper industry in Maine. Land application of this material on forest sites is one method of sludge disposal that is currently being studied. The chemical and physical quality of a sludge to be spread is of prime consideration in any land application program (DEP 1988). Therefore, sludge must be carefully analyzed prior to application for a land application permit. Such analyses include, but are not limited to, pH, percentage of dry solids, total volatile solids, macro- and micronutrients, and heavy metals.

Sludge that is to be land applied on forested sites is commonly placed in temporary storage facilities, such as on-site winter field stacking areas (DEP 1988). Sludge may be held in these facilities for no more than 180 days. However, the chemical and physical properties of sludge may change within this time frame, due to microbial activity, mechanical and chemical weathering, and leaching of nutrients within sludge piles. These changes in composition may considerably increase the variability of actual sludge and chemical loading rates.

In addition to variability of sludge properties within a temporary stockpile, there may be considerable spatial variation in actual loading rates of sludge applied to a site. Factors such as topography, weather conditions, machine performance, operator experience, and sludge characteristics can influence the uniformity of sludge application.

The goal of this case study was to assess two components of variability associated with the land application of papermill sludge. These included (1) changes occurring within a temporary sludge stockpile, and (2) spatial variability of application to the site. Understanding the magnitude of such changes will assist in accounting for variability inherent in such land application operations.

METHODS AND MATERIALS

Sludgepile Sampling

On January 19, 1989, a large stockpile (approx. 7500 yds³ or 5735 m³) of papermill sludge located in Letter E Township, Franklin County, Maine, was sampled. The sludge had originated at the International Paper mill in Jay, Maine, and had been stockpiled from December, 1988, to early January, 1989. Sampling was accomplished by running three transects across the pile, with ten samples being taken along each transect.

At each sample point, samples were taken at three depths using a shovel: surface, two feet, and five feet. For each transect, the ten samples for a specific depth were combined, mixed thoroughly, and subsampled to obtain the material used for chemical and physical analyses.

Sludge Analysis

All sludge samples were air-dried, ground through a 2 mm sieve, and stored in Whirlpack bags. Sludge pH and Loss-On-Ignition were determined by the methods of Robarge and Fernandez (1986). Extractable Ca, Mg, K, Na, P, Al, Fe, Mn, B, Cu, and Zn were determined by extraction with unbuffered 1 N NH_4Cl (Robarge and Fernandez 1986). Total elemental concentrations in the sludge were determined using a nitric-perchloric digestion (Thornton et al. 1985). All elements were measured by inductively coupled plasma emission spectrometry (ICP), except K and Na, which were measured by atomic absorption.

Loading Rate Measurement

In early September, 1989, sludge from the stockpile was spread on a clearcut site in Letter E Township in western Maine by private contractors. A forwarder outfitted with a spreading unit was used to apply the material. Application of sludge to four previously established study plots (20 m by 20 m) was measured using black plastic trays (61 cm by 46 cm). Trays were sufficiently deep to prevent sludge from spilling out. One tray was placed at each corner of the plots for a total of 16 trays. Following application, sludge from each tray was collected and placed individually in labelled plastic bags. In some cases, application rates were so heavy that only a portion of the sample could be collected. Correction factors were applied to account for this subsampling. Samples were then dried in a greenhouse and weighed. From the data collected, loading rates at each of the four corners of each plot were calculated.

Statistical Analysis

For stockpile data, descriptive statistics, analysis of variance, and mean separations using Duncan's New Multiple Range Test were calculated using the Statistical Analysis System (SAS Institute 1982) on the University of Maine mainframe computer facilities.

RESULTS AND DISCUSSION

Stockpile Variability

Considerable variability in chemical properties was seen throughout the sludge stockpile, both among

transects and at varying depths. Transects represent different times of stockpiling, therefore differences between total elemental concentrations of sludge from the transects may reflect variability in the papermaking process that produced the sludge. Sludge pH in water and concentrations of N, Ca, P, Mg, Al, Na, and Cu are presented in Figures 1 through 7. Table 1 shows pH in dilute salt solution, organic matter estimated by Loss-On-Ignition, and concentrations of K, Zn, Fe, and B in sludge. Concentrations of total N, P, Ca, and Na were significantly different among transects. Total N differences may also be due in part to mineralization of sludge organic N, with subsequent losses over time due to denitrification, volatilization, and leaching. Transect C is the oldest material, which supports this explanation of aging effects on total N concentrations.

Both increasing and decreasing trends were seen for total and extractable elemental concentrations. Few clear trends for total elemental concentrations were evident with depth, except for total Na which increased with depth, possibly due to its soluble nature and susceptibility to leaching (Figure 6). In the relatively short period of time the sludge had been stockpiled, sufficient Na may have been leached from the surface layer of the pile to greater depths such that total concentrations were significantly increased. Extractable Ca, Mg, K, Na, and Mn all increased significantly with depth (Figures 2, 4, 6, and Table 1). While many metals tend to complex with organic materials, these base cations appear to remain relatively mobile within the sludge. Preliminary results from soil solution measurements at the site where this sludge was applied (unpublished data) suggest that SO_4 may be the dominant counter anion providing electroneutrality for the leading cations. Manganese becomes more soluble with decreasing pH and more reducing conditions. Therefore, increased levels of extractable Mn at greater depths may reflect the decrease in sludge pH (Table 1), and lower redox potential deeper in the stockpile. In all cases, the source of greater concentrations with depth is likely from cation release following decomposition at the surface of the stockpile.

Total N, P, Al, and sludge pH and extractable P, Al, and Cu all decreased with increasing depth in the stockpile (Figures 1, 3, 5, and 7). One possible explanation of higher surface concentrations of these elements may be the "rind effect". As the outermost layer of the stockpile settles (i.e., the "rind") and begins to decompose, elements in this layer can be concentrated in the decreasing sludge mass. Low Al and Cu solubility due to high sludge pH and complexation by organic matter would inhibit leaching of these elements to lower depths. As the outer layer of sludge decomposes and C is released through microbial respiration, much of the N would be immobilized in the form of microbial biomass given the relatively high C:N ratio (approx. 30:1) of

the sludge. This aggrading microbial biomass in the surface layers of the stockpile would result in higher total N concentrations.

Table 1. Selected sludge pH-salt, LOI, and elemental concentrations (mg/kg)¹

Depth	A	Transect	
		B	C
		<u>pH-salt</u>	
1	7.18 aC	7.30 aB	7.39 aA
2	7.13 aA	7.07 bB	7.15 bA
3	6.85 bC	6.98 cB	7.18 bA
		<u>LOI</u>	
1	54.7 aA	48.7 aB	50.1 abB
2	53.1 aA	49.5 aAB	48.3 bB
3	54.7 aA	47.2 aB	53.4 aA
		<u>Total K</u>	
1	1510 aA	1597 aA	1643 aA
2	1339 aB	1136 bB	1793 aA
3	1389 aAB	1193 bB	1508 aA
		<u>Extractable K</u>	
1	446 bA	392 bB	427 cA
2	462 bB	421 aC	500 bA
3	503 aA	417 abB	527 aA
		<u>Total Mn</u>	
1	975 abB	1086 aA	1131 aA
2	881 bB	877 bB	1026 bA
3	1003 aA	948 bA	964 bA
		<u>Extractable Mn</u>	
1	179 cA	177 cA	202 cA
2	428 bA	418 bA	404 bA
3	549 aA	457 aB	446 aB
		<u>Total B</u>	
1	8.84 aA	10.66 aA	11.10 aA
2	8.75 aA	9.69 aA	10.32 aA
3	9.94 aA	9.98 aA	11.91 aA
		<u>Extractable B</u>	
1	0.30 bB	0.85 aAB	1.03 bA
2	1.14 aA	1.23 aA	1.67 aA
3	1.60 aA	0.93 aB	1.36 abAB

¹ Lower and upper case letters denote statistically significant differences among depths and transects, respectively, at the 0.05 confidence level.

Extractable P (Figure 2) also decreased significantly with depth in the sludge pile. An additional explanation for this trend, aside from the rind effect, may be the result of Ca-P solubility. Calcium-phosphate species are likely the dominant forms of P at the pHs and Ca concentrations measured for this sludge (Lindsay and Moreno 1960). Therefore, more P may be extractable, with NH_4Cl , from the outer rind since the lowest extractable Ca was found there. However, at greater depths increased Ca concentrations may result in more P

being combined as Ca-P compounds, thus reducing the more labile extractable P pool. Similar trends were evident for Mg, which behaves like Ca with regard to P solubility.

Little variation existed among transects or depths for Zn and Fe concentrations. The mean total and extractable Zn concentrations for all samples were 183 and 2.54 mg kg⁻¹, respectively. Overall total Fe concentrations were 3932 mg kg⁻¹ but all extractable Fe measurements were below the instrumental detection limit for this study.

Figure 7 shows few differences existed in the total Cu data, with extractable Cu showing the "rind effect" phenomenon proposed above, except for total Cu in transect A at depth 2. This anomalous high concentration of total Cu likely represents a piece metal from wire or machinery in the sample. Although not characteristic of the sludge itself, this data graphically underscores the need for adequate replication and quality control in sludge characterization.

Loading Rate Variability

The target loading range for sludge on this site was 18 to 20 dry tons/acre (40-44 T/ha). However, actual loading rates varied considerably among plots and also within plots. Among all plots, mean loading rates ranged from 9.4 tons/acre (Plot I) to 63.6 tons/acre (Plot IV) Table 2. Within plots, the largest range was seen in Plot IV (3.1 to 124.7 tons/acre) as shown in Figure 8.

High spatial variation in sludge application was readily evident upon visual examination of the plots. The actual loading rate of sludge applied to plots appeared to vary with terrain features, machine operation, and operator experience. The data presented here is from a case study of a specific site, the range of loading rates seen in this study highlights the need for further investigations to determine realistic capabilities and potential. Steps should be taken to account for such variability in future monitoring or research studies of sludge land application operations. In addition, investigations to determine responses to land application of papermill sludge should include individual tree, plot level, and ecosystem or watershed level research.

Table 2. Mean Actual Loading Rates (air-dried tons/acre).

Plot	Mean Loading Rate
I	9.4
II	33.0
III	18.7
IV	63.6
Overall Mean	31.2

CONCLUSIONS

Significant variability in the chemical characteristics of a temporary stockpile of papermill sludge was observed at a forest land application site in western Maine. These differences were evident despite the fact that the stockpile had been in place at the time of sampling for approximately one month during the winter. Significant differences were observed both among transects taken across the length of the stockpile and at different depths within the stockpile. Therefore, it is evident that the temporary sludge stockpile was in a dynamic state. Longer time periods and warmer climatic conditions would be expected to magnify the variability characterized in this study. Considering the dynamic conditions within the stockpile, it should not be assumed that subsequent land application of this sludge will utilize material of uniform composition. Calculating loading rates and acceptable ranges for land applied papermill sludge would ideally require consideration of stockpile variability and not be based solely upon elemental concentrations of a limited number of sludge samples taken directly at the mill. Stockpile variability, combined with the high spatial variability in loading rates on the site, makes quantification of the range and mean of actual loading rates a challenging task. However, understanding the characteristics of these components of variability in land application operations provides the necessary foundation for designing adequate evaluation and monitoring systems which will ensure sound waste utilization practices.

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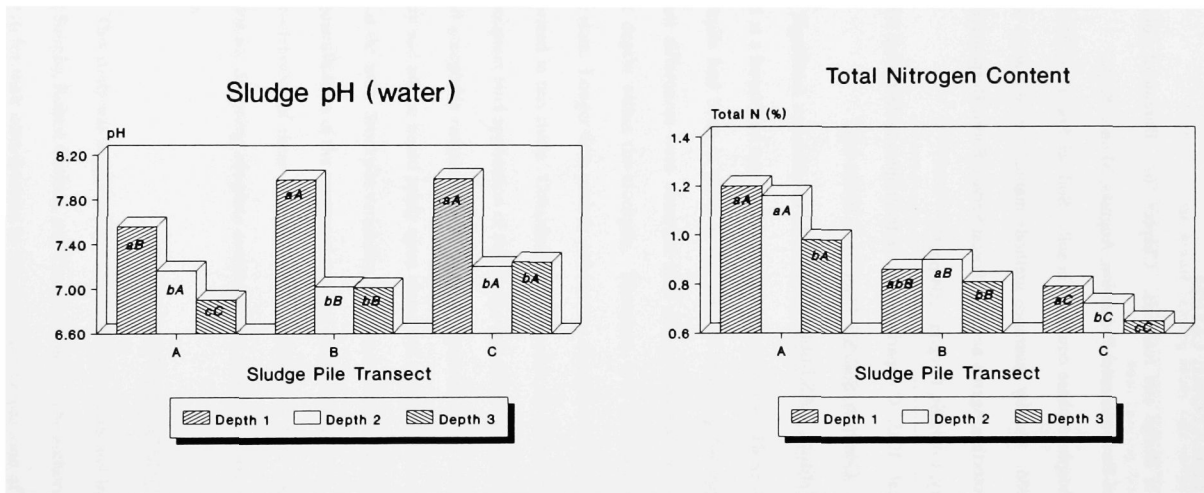


Figure 1. Sludge pH and total nitrogen content. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively, at the 0.05 level.

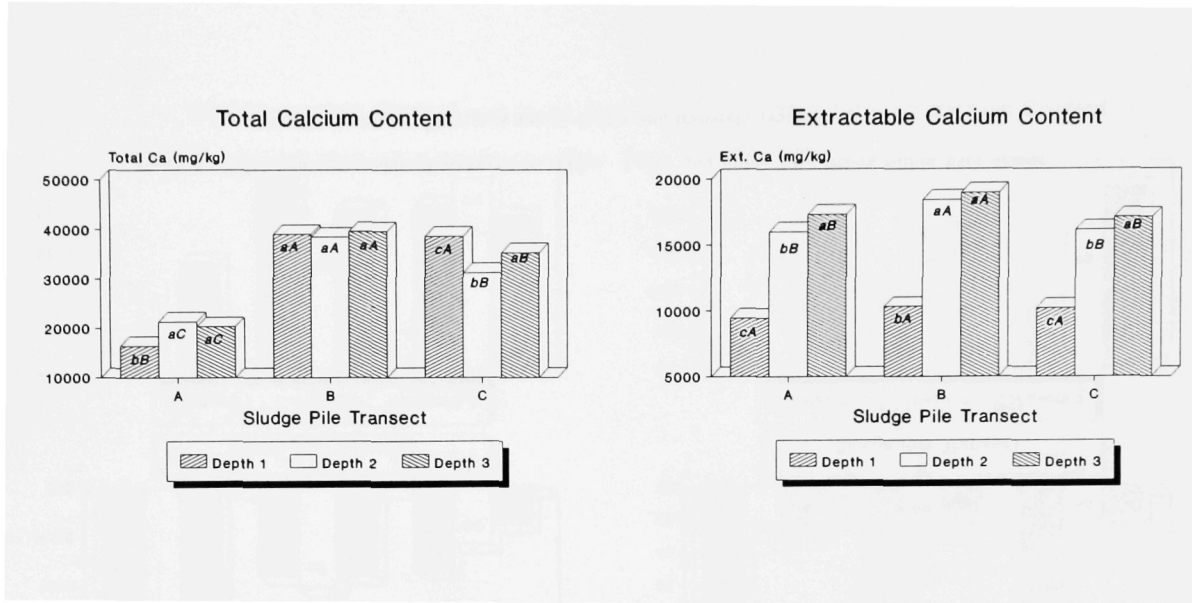


Figure 2. Total and extractable calcium in sludge. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively.

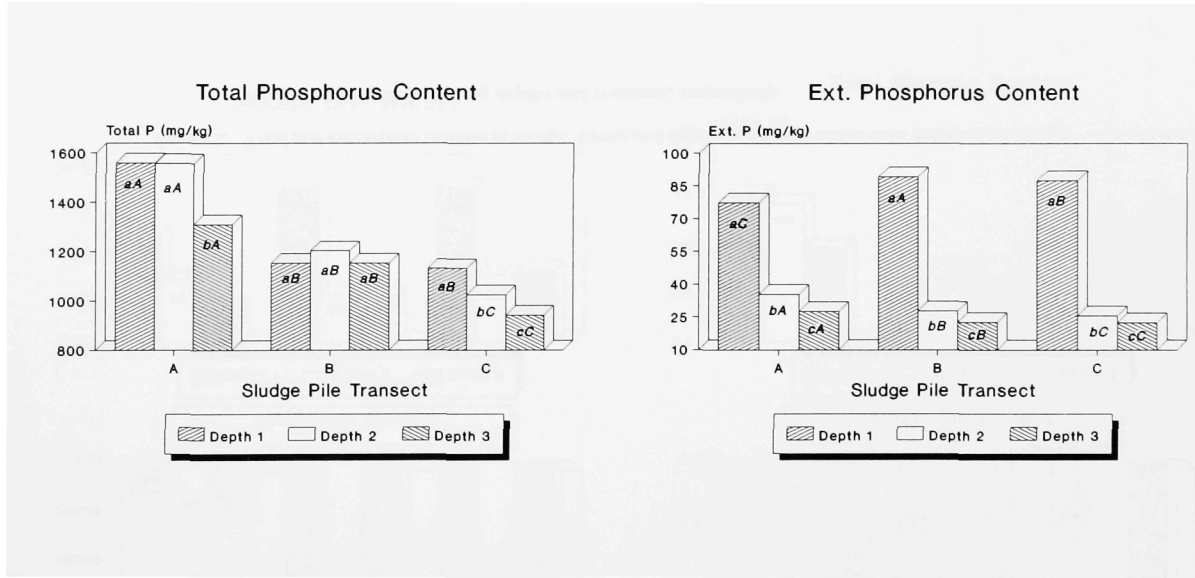


Figure 3. Total and extractable phosphorus in sludge. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively.

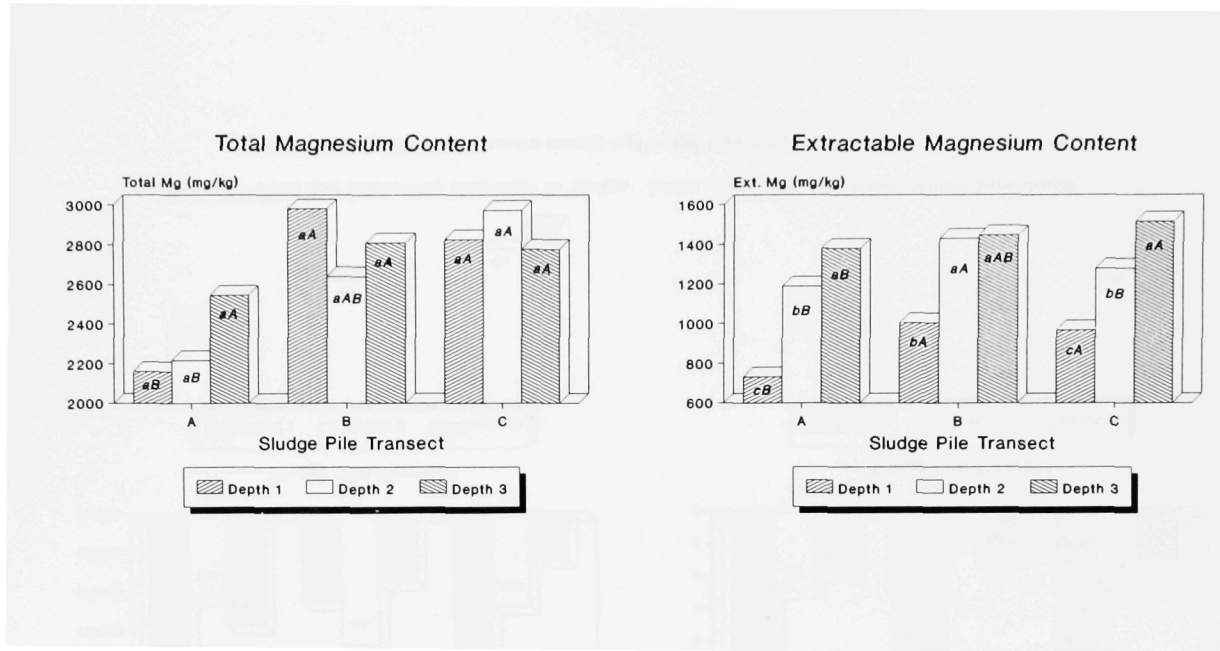


Figure 4. Total and extractable magnesium in sludge. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively.

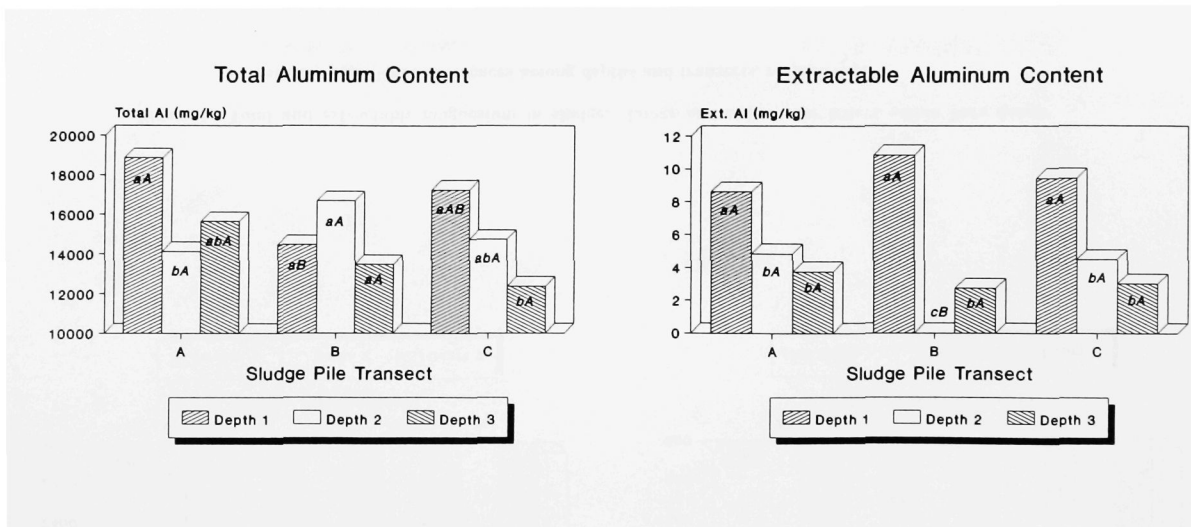


Figure 5. Total and extractable aluminum in sludge. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively.

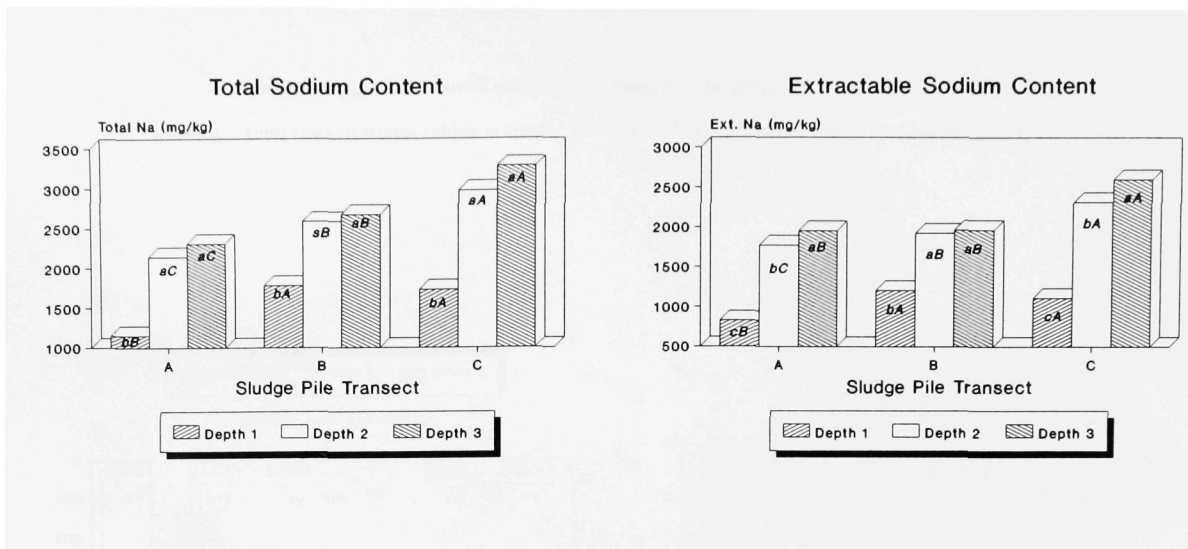


Figure 6. Total and extractable sodium in sludge. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively.

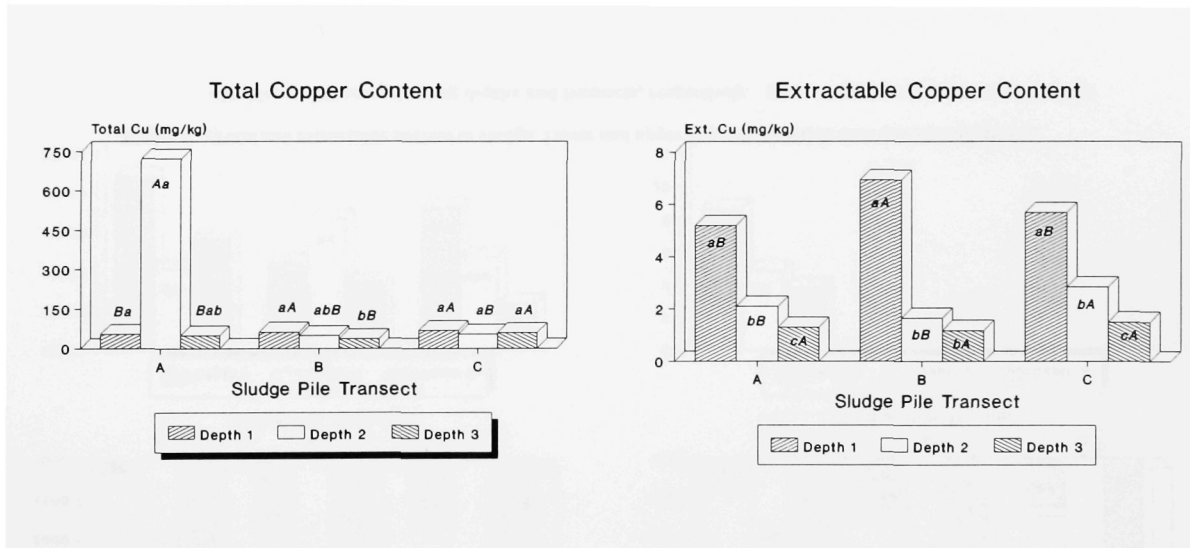


Figure 7. Total and extractable copper in sludge. Lower and upper case letters within bars denote statistically significant differences among depths and transects, respectively.

Sludge Loading Rates (tons/acre)

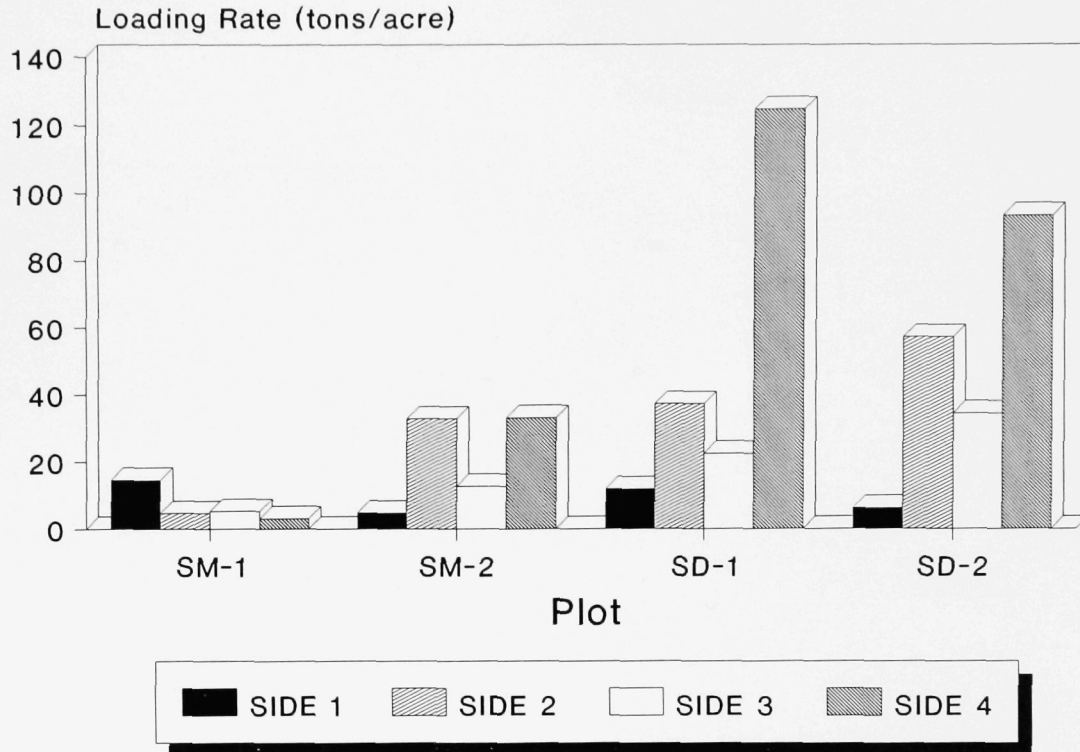


Figure 8. Individual measurements of actual sludge loading rates to the treated plots.