MICROANALYSIS OF WASTEWATER-AMENDED VADOSE ZONE SOILS

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Abstract

Microanalytical techniques were utilized as part of an investigation of soil pore clogging phenomena in wastewater-amended vadose zone soils. Undisturbed samples of Plano silt loam soil were obtained from a field research site where different treatments comprised of wastewater effluent type and loading rate had been intermittently applied to pilot-scale infiltration cells over a five-year period. As a result of the wastewater infiltration, soil pore clogging had developed to variable degrees as a function of treatment, in some cases it was substantial while it others it was nil. Sixty-four soil thin sections were prepared from the undisturbed soil cores and analyzed for micromorphometric features utilizing standard petrographic and dark field microscopy. A limited number of polished thin sections were also analyzed for microchemical properties utilizing an electron microprobe. These microanalyses enhanced the data derived from general morphological inspections and physical and chemical analyses of bulk soil samples. Amorphous organic materials were found to be correlated with soil pore clogging and the microanalyses provided valuable information regarding the nature and extent of their association with the natural soil.

Introduction

Infiltration of wastewater effluents into soil is known to result in pore clogging and dramatic reductions in natural soil infiltrability properties. Some degree of clogging can be beneficial by enhancing purification. However severe clogging can cause soil system anaerobiosis and diminished effluent purification as well as premature hydraulic failure. Knowledge of the causes of soil clogging is essential to help ensure that the design and operation of wastewater infiltration systems results in acceptable system performance.

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A field investigation was initiated at the University of Wisconsin in 1979 to study soil clogging and the effects of wastewater effluent composition and loading rate on its development. In the first of two major phases of the experiment, a field research facility was established to facilitate the study of soil clogging by means of a replicated, two-variable, three-level factorial design. The independent variables included three effluent types and three hydraulic loading rates with each treatment applied to replicate 0.9 m diameter soil cells installed in situ in a silt loam soil. During Phase I the infiltration rate response to each treatment (effluent type/hydraulic loading rate) was monitored over a five-year period. Soil clogging was observed to develop to variable degrees as a result of the different treatments. In some cases clogging was substantial, in others it was nil.

During a second phase of the experiment, undisturbed soil samples were collected from selected infiltration cells of each treatment to identify the nature and extent of any changes in soil physical and chemical properties that could have accounted for the soil clogging observed during Phase I. Physical and chemical analyses were conducted on bulk soil samples with depth below the infiltrative surface of each cell. Microanalyses were conducted on soil thin sections prepared from undisturbed soil samples to enable determination of micromorphometric and microchemical characteristics.

The purpose of this paper is to describe the methods and results of the microanalysis portion of the work. For details of the other aspects of the experimental work, readers are encouraged to contact the senior author of this paper. Information regarding forthcoming publications may be obtained from the University of Wisconsin (1985).

Materials and Methods

Soil Clogging Development

To facilitate the study of soil clogging as affected by wastewater infiltration, a research facility was established to include replicated 0.9 m diam. infiltration cells (pilot-scale subsurface soil absorption systems) installed in situ in a Plano silt loam soil (fine-silty, mixed, mesic, typic argiudoll). The wastewater infiltration surface of each cell was placed at about 70 cm depth in the B2 horizon, a zone of silty clay loam with 9% sand, 51% silt and 40% clay. Hydraulic conductivity properties included a Ksat of 223 cm/d dropping off sharply to less than 1 cm/d at 25 mbar tension.

A total of 24 infiltration cells were established and one of nine treatments was applied to each. Nine cells were loaded with domestic septic tank effluent (DSTE) applied to triplicate cells at 1.3, 2.6 and 5.2 cm/d (normal application rate for the Plano soil is 2 to 3 cm/d). An additional nine cells received graywater (domestic wastewaters less human waste) septic tank effluent (GSTE) also applied to triplicate cells at the same three rates. The final three treatments consisted of clean tapwater applied to duplicate cells at the three rates.

Intermittent daily application of the wastewater effluents and tapwater caused soil clogging, the occurrence and extent of which was a function of treatment. Soil clogging did not occur as a result of tapwater infiltration over five years at any of the three rates studied. For those cells receiving the two wastewater effluents, soil clogging development was significantly affected by effluent composition and loading rate, with the effect of composition more influential than that of loading rate. Domestic septic tank effluent (DSTE) application stimulated substantial clogging as evidenced by dramatic losses in soil infiltration rates. There was a variable initial period of operation at near initial infiltration rates followed by a steady decline to rates less than the daily loading rate. The length of the initial period was inversely related to daily hydraulic loading. Application of graywater STE stimulated soil clogging only at the highest loading rate studied, 5.2 cm/d. The infiltration rate responses of these cells were substantially the same as those of the 1.3 cm/d domestic STE cells. In all soil cells which experienced substantial soil clogging, the development of the clogging occurred over an extended time period (i.e. 18 to 24 months). As a expression of the clogged soil condition of some cells, continuous ponding of effluent developed. In all 2.6 and 5.2 cm/d DSTE cells ponding ensued after approximately 2.5 years of operation, while in the 1.3 cm/d DSTE and the 5.2 cm/d GSTE cells ponding did not occur until after five years of operation. With continued operation of a ponded cell, the depth of ponding gradually increased.

Soil Macroanalyses

In light of the differential soil clogging observed between treatments, further research was conducted to investigate the the characteristics of the wastewater-amended soils and to determine the nature and extent of the agents responsible for the soil clogging observed. This work involved the excavation into and extraction of undisturbed soil samples from selected soil cells to which the different treatments had been applied for 62 and 70 months. Undisturbed natural soil adjacent to the research site was also sampled.

Replicate undisturbed soil cores (10 cm diam. by 30 cm length) were extracted from a total of 11 soil cells and 1 undisturbed natural soil location. Soil samples were taken from the cores at various depths and analyses were conducted for morphological characteristics, moisture content and loss on ignition, total organic carbon, acid-extractable organic carbon, carbohydrate carbon, nitrogen forms, inorganic phosphorus and sulfur, exchangeable cations and total bacteria. Among the many results derived from these analyses, most striking were the substantial accumulations of organic matter at the wastewater infiltrative surfaces in those cells that had experienced substantial soil clogging. In each clogged soil cell, organic carbon and nitrogen concentrations were at least an order of magnitude higher at the infiltrative surface as compared to 10 mm below it where concentrations similar to natural soil existed.

Soil Microanalyses

Undisturbed soil cores were also extracted from seven infiltration cells and one natural soil location. From these, soil thin sections were prepared to enable micromorphometric and microchemical analyses. Undisturbed soil cores were obtained by implanting 15 cm diameter by 20 cm long sections of plastic pipe vertically through the wastewater infiltrative surfaces. The penetrating end of each core was beveled to aid penetration into the soil. Each core was first manually pressed into the soil surface and then a truck-mounted hydraulic soil probe was used to fully implant and extract the undisturbed core. After extraction, the soil cores were placed in polyethylene bags and stored in refrigerated containers. Within 8 hours of extraction, all eight cores were transported to the University of Wisconsin where they were stored at 4 C pending further sample preparation.

Soil thin sections were prepared in accordance with conventional procedures (e.g. Cady, 1983) with the possible exception of the drying and impregnation. The cores were dried under an argon atmosphere at 5 to 10 C to minimize the oxidation of organic matter and hydrous iron oxides as well as to limit the dessication of the soil during drying. After 133 days of drying, each of the cores was impregnated with Epo-Tek 301 epoxy. This epoxy was used because it had a very low viscosity, cured at room temperatures and had a refractive index of 1.54. After impregnation the cores were slabbed vertically through the center thus providing two equal halves, one of which was utilized for the preparation of vertical thin sections and the other for horizontal thin sections.

A total of 64 thin sections were prepared from the eight soil cores. From slabs taken from the central portion of each core, sequential vertical and horizontal sections (25 by 50 mm nominal size) were taken with depth beginning above the cell infiltrative surface and extending through it down to a depth of approximately 10 cm. Several selected sections were made in replicate and the replicates were polished for subsequent electron probe analysis.

The covered thin sections were analyzed utilizing standard petrographic and dark field techniques (Thresher, 1982; Cady, 1983). Standard petrographic techniques yield information concerning non-opaque materials but tend to yield very limited information with regards to amorphous organic matter or hydrous iron oxides, materials believed to be important from a soil clogging perspective. Dark field techniques, however, usually yield significant information regarding these semi-opaque materials (Thresher, 1982). The dark field colors of selected materials are: organic matter = light gray to black; geothite = light yellow to dark yellowish brown; lepidochrosite = light orange to dark orange brown; and hematite = light red to dark reddish brown. The dark field colors of the hydrous iron oxides are the result of selective absorption of light and internal reflections while those of organic matter are the result of absorption of white light. The darkness or lightness of the color is related to the relative concentration and grain size of the material responsible for the color. Since pedogenically produced organic matter and hydrous iron oxides are usually very fine grained, the depth of color of these materials in soils is generally related to the type and relative concentration of the material giving rise to the color.

Electron microscopic analyses of polished thin sections from the wastewater infiltrative surfaces of the 5.2 cm/d DSTE and GSTE cells were conducted to determine the weight percent composition of nine elements within selected locations of each thin section. Qualitative and semi-quantitative analyses were conducted for Si, Al, Ca, Mg, K, Na, Fe, S and P using energy dispersive and wavelength dispersive X-ray analysis in accordance with conventional practice (e.g. Keil, 1967; Kittrick, 1983). Precise quantitative analyses were not possible in the present work due to the epoxy impregnation agent and the difficulties in generating a relief-free surface in a soil thin section.

All electron probe analyses were conducted with the electron beam adjusted to scan an area, approximately 30 um square. Initially analyzed were selected minerals and compounds within standards to determine the intensities of the characteristic X-ray lines which would subsequently be determined on points within the two thin sections. Within each section were analyzed, five locations along a transect beginning at the wastewater infiltrative surface and extending several cm below it. Within each location were analyzed 10 points set on a 2 by 5 grid with each point separated from the adjacent points by approximately 200 um. Thus an area of approximately 0.2 mm by 0.8 mm was analyzed within each location of the transect.

Results

Soil Cell Morphology

In those soil infiltration cells that had experienced substantial clogging and a dramatic loss of soil infiltrability but which had not been continuously ponded with effluent, a thin black layer (<10 mm) was observed at the wastewater infiltrative surface while the underlying soil appeared similar to natural soil in color, structure and consistency. In contrast, in those cells that had been continuously ponded for an extended time period (e.g. >24 mo.), a substantial accumulation of similarly appearing black solids were observed to be present at the infiltrative surface but also to considerable height above it. Generally these black solids could be readily scraped away to reveal a structured soil underneath, although highly reduced as evidenced by gleyed soil colors (e.g. 5Y6/1).

Micromorphometric Features

Microscopic examination of the soil thin sections revealed various features concerning the zone of clogging and the materials responsible. While the nature and extent of specific features varied between thin sections of a given soil cell core, in large measure the observations in thin section were consistent within a given core and with the macromorphology observed in the field.

Microscopic examination of the thin sections from the unclogged cells, that is those that had exhibited no loss in infiltration rate, revealed conditions at the wastewater infiltative surface that were substantially similar to those of natural soil. The application and infiltration of tapwater and graywater septic tank effluent had little if any measurable effect on the micromorphology of the soils.

In sharp contrast, microscopic examination of the thin sections from the severely clogged soil cells revealed an accumulation of amorphous organic matter at the wastewater infiltrative surface. A representative thin section and photomicrograph therefrom are presented in Figure 1. As shown in Figure 1, there were two distinct zones of alteration of natural soil properties. There was a thin surficial zone where amorphous organic matter was present within soil pores within the natural soil matrix. The overall thickness of this surficial zone was typically less than 1 mm. A second zone was present above the first and was characterized by a matrix of mineral materials enriched with organic matter. Both of these features were nonexistent at locations where the gravel aggregate was in direct contact with the natural soil (Figure 1).

The features observed along the horizontal infiltrative surfaces of the clogged soil cells were also present along the vertical sidewalls of soil macropores (Figure 2). The section and photomicrograph of Figure 2 reveal a sealing of the soil matrix as well as the apparent blockage of the entry to the soil macropore. The vertical macropore sidewalls were observed to develop the same diffuse penetrational zone of organics but a lesser depositional zone. The latter effect could have been due to vertical orientation of the sidewall and/or blockage of the pore entrance.

Microchemical Properties

The results of the electron probe analyses revealed that to a first approximation the concentrations of the elements measured (Si, Al, Ca, Mg, Na, K, Fe, S, P) were substantially the same within and between the soil thin sections for the 5.2 cm/d DSTE and GSTE cells. Even in the organically enriched zones of the DSTE section, the concentrations of most elements were similar to those of the underlying natural soil zone and to all locations within the GSTE section. The levels of phosphorus and sulfur did appear to be elevated in the organically enriched zones of the DSTE section as compared to the underlying soil and that of all locations in the GSTE section. These results were consistent with those of the bulk soil chemical analyses and the micromorphometric observations, the results of which indicated a mineral matrix in both the surficial zone of diffuse organic matter penetration as well as the depositional zone above the surficial zone.

Summary

A field study of the effects on soil clogging development of wastewater effluent composition and loading rate revealed significant differences in clogging associated with these factors. Results of observations of macromorphology and macroanalyses for various physical and chemical parameters revealed an enrichment of organic matter at the wastewater infiltrative surface of the clogged soil. Microanalyses involving both microscopic and microchemical methods proved to be useful as supplementary tools to provide insight into the clogging materials. These

analyses revealed that in the wastewater infiltration cells that had clogged, there was a thin surficial zone at the wastewater infiltrative surface where amorphous organic matter had diffusely penetrated pores within the natural soil matrix. Upon this surficial zone was a deposit of considerable thickness characterized to consist of a largely mineral matrix enriched with organic matter. The latter deposit appeared to have developed during prolonged periods of continuous inundation of the wastewater infiltrative surface. Further research into the genesis of the clogging materials is in progress.

Acknowledgments

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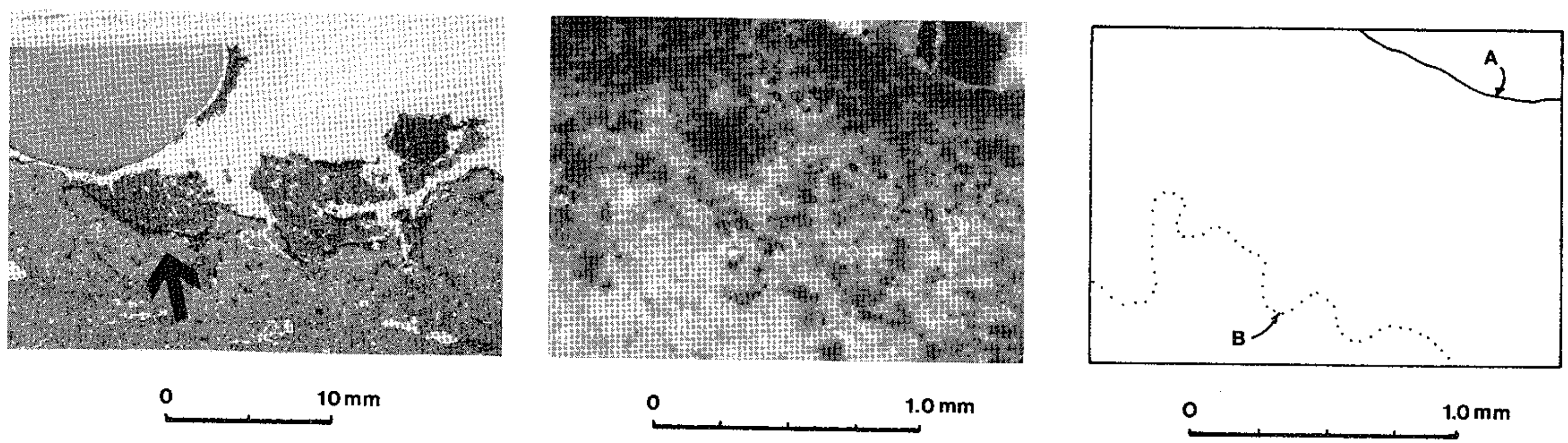

Figure 1. (LEFT) Photograph of a thin section prepared from a clogged and ponded domestic STE cell (5.2 cm/d application rate) showing a layer of accumulated organic and mineral material above the original soil penetrative surface (above arrow). Note the lack of accumulated material beneath the grain of gravel in the upper left corner of the photo.

(CENTER and RIGHT) Photomicrograph and drawing of the area above the arrow in the above photo showing the interpreted original penetrative surface (A) and diffuse penetration of organic material into the soil (B-lower limit of penetration).

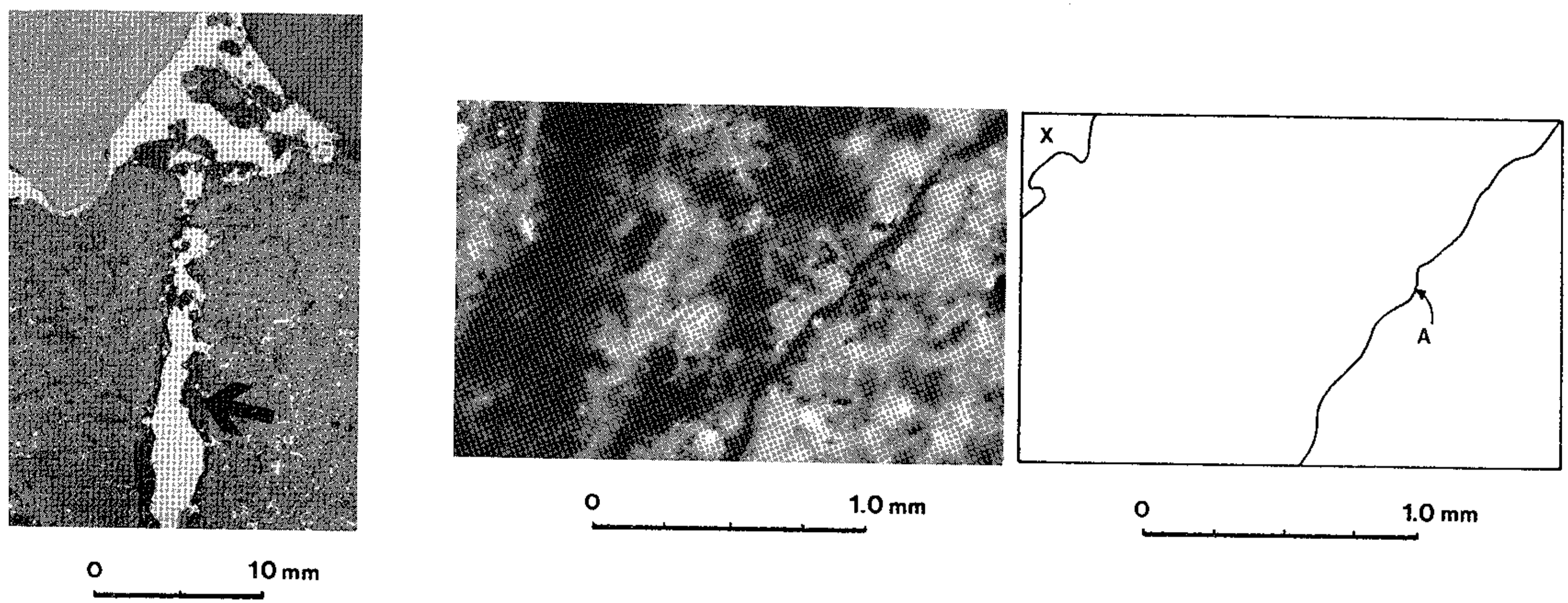

Figure 2. (LEFT) Photograph of a thin section prepared from a clogged and ponded domestic STE cell (5.2 cm/d application rate) showing sealing and blocking of a vertical soil macropore (next to arrow).

(CENTER and RIGHT) Photomicrograph and drawing of the area to the left of the arrow in the above photo showing the interpreted original penetrative surface (A) and accumulated organic and mineral material containing reduced hydrous iron oxides (X-open vertical pore space).