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I am submitting herewith a thesis written by Tony Layne Arnold entitled "Response of Corn and Fescue to Land Application of Industrial Waste Arising from Illmenite Ore Processing." I recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Plant and Soil Science.

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RESPONSE OF CORN AND FESCUE TO LAND APPLICATION
OF INDUSTRIAL WASTE ARISING FROM
ILLMENITE ORE PROCESSING

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville

Tony Layne Arnold

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ABSTRACT

Industrial sludge, a by-product of Ti extraction from illmenite ore (FeTiO_3), was applied to corn and fescue plots at rates of 0, 22, 45, and 90 metric tons/ha. Each rate of sludge, which characteristically has a pH value <2 , was applied at two rates of lime, 9 and 18 metric tons/ha, except for the 0 rate which was maintained as a control. The application of the waste material and lime did not affect the yield of either corn or fescue.

Tissue samples were taken at three stages of growth from each species and analyzed for selected heavy metals. Concentrations of As, Cd, Fe, Na, Ni, Pb, Se, Ti, and Zn in the first and second harvests of fescue were not affected by any treatment. Manganese concentrations in the fescue samples were significantly increased as a result of the highest rate of sludge. The concentrations ranged from 183 to 358 $\mu\text{g/g}$ of tissue in the first harvest, and from 231 to 410 $\mu\text{g/g}$ in the third harvest. The Ni concentration in the second harvest was significantly increased by applying sludge at the rate of 22 metric tons/ha while the 90 metric tons/ha rate increased in Ni concentration in the third harvest.

Manganese concentration in the corn fodder and grain stages were not affected by any treatment. However, corn ear-leaf analysis gave a significant increase in Mn as a result of treatment with 22 metric tons/ha sludge with 18 metric tons/ha lime. Concentration of Cd, Na, Fe, and Zn showed considerable variation among the stages of growth. Titanium in both species was not influenced by the treatments.

Soil water samples were collected at four depths in both corn and fescue plots (30, 61, 92, and 122 cm). Analysis of soil water samples indicated that the conductivity, chloride concentration, and Ni content increased due to the 90 metric ton sludge application. The pH of the groundwater, however, was not decreased by the applied treatments.

Analysis of the soil from each treatment showed no tendency for the pH, CEC, and organic matter being altered by the sludge-lime treatments. Heavy metal analysis indicated a tendency for the concentration of Mn and Fe to be increased in the 0-15 cm layer while Ni concentrations increased in the subsurface layers.

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CHAPTER I

INTRODUCTION

In recent years there has been an increased awareness of environmental pollution. As a result of this awareness, the 1972 Amendments to the Federal Water Pollution Control Act (Public Law 92-500) was enacted. This law has set standards and/or goals that all discharge of pollutants into navigable waters will be eliminated by 1985.

The enactment of this law has required that all viable alternatives be investigated in waste management or treatment for both municipalities and industries. Failure to comply to this law will result in the loss of federal funding.

The advantages of applying animal waste to the land as a source of plant nutrients have been known for many years. In more recent times, researchers have been applying municipal effluent to the land not only as a means of disposal, but also as a source of nutrients. As a result of Public Law 92-500, industry has begun searching for the best means by which they can dispose of their waste material. Industrial wastes vary considerably in their composition. Due to this variation, it is necessary for researchers to investigate the specific application of this material to the land and monitor factors such as chlorides, conductivity, heavy metals, other potentially toxic elements, pH, organics, and nitrogen to name a few.

The primary purpose of this research was to investigate the feasibility of applying an industrial waste material, obtained from E. I. DuPont de Nemours and Co., Inc., to the land and to determine its effects on both corn and fescue. The final conclusions were based on field plots that were conducted near New Johnsonville, Tennessee, on a DuPont plant site.

CHAPTER II

LITERATURE REVIEW

The application of waste to land or land treatment technology is an alternative approach to waste management. Overcash and Pal (1979) stated that "Land application is the intimate mixing or dispersion of waste into the upper zone of the soil-plant system with the objective of microbial stabilization, adsorption, immobilization, selective dispersion or crop recovery, leading to an environmentally acceptable assimilation of the waste."

Utilizing land treatment technology has received considerable attention in recent years. When applying waste to land, the main problem encountered is that the material and the disposal site vary widely. Because of these variations researchers must outline a specific site for applying a waste, containing an identified concentration of constituents.

The specific site in question should be defined according to several parameters. Soil properties such as texture, hydraulic conductivity, soil moisture, cation exchange capacity, heavy metals, organic matter and organisms should be specified to derive the possible limitations the site may have for loading capacities. As the data obtained from an area and specific for that site, every location must be investigated individually.

Waste materials vary greatly in constituents due primarily to its origin. Knapp (1970) indicated that industrial wastes are characterized by high biochemical oxygen demand (BOD), suspended solids, and inorganic and organic constituents. Waste material to be applied to a soil-plant system should be characterized for the following constituents:

phosphates, sulfides, oil, grease, specific organics, salts, acids, bases, anions, heavy metals and nitrogen (Overcash and Pal 1979). Care should be exercised in amount applied depending on the data obtained from these analyses so as not to exceed plant nutrient needs or apply toxic concentrations of heavy metals and/or other substances.

Applying waste material to land containing high amounts of "heavy metals" and other "potentially toxic elements" has received considerable interests from many researchers due to the possibility of reaching toxic levels in soils, contamination of groundwater, plant uptake and the risk of terrestrial food chain contamination. The term "heavy metals" generally refers to elements such as cadmium, cobalt, chromium, copper, iron, mercury, manganese, molybdenum, nickel, lead and zinc (Leeper 1972). "Potentially toxic elements" include arsenic, barium, selenium, sodium and silver. Each of these elements vary in its level of potential toxicity, uptake by plants and soil availability (Leeper 1972). These elements will be discussed regarding their potential for creating problem situations in a biological system.

Cadmium

Cadmium levels in the environment have received considerable attention in recent years as it has shown to cause detrimental effects

on both animals and humans with only moderate increases in dietary levels. These detrimental effects have not been established for Cd contained in organic materials, but many implications have been made. Some researchers have found that Cd in the human diet may be associated with such factors as hypertension (Schroeder and Balassa 1963), itai-itai disease (disease of the bones), respiratory diseases, cardiovascular diseases, and shortened life span (Fassett, 1975). Cadmium added to the diets of animals resulted in higher concentrations being found in the liver and kidneys as opposed to muscle and fat tissue (Doyle et al. 1974; Baker et al., 1975). Due to uncertainty of the concentrations of Cd in the environment that presents potential hazards to health, it is important that the amounts contained in sludges or other materials applied to the land be monitored so as not to pose a health threat (Sandstead, 1974).

Sludges vary considerably in Cd concentration depending upon the origin of the material. Sewage sludges have been found to contain from 3 to 3,000 ppm Cd depending on the amount of industrial effluent that is mixed with the domestic wastes (Sommers, 1977). Industrial wastes usually contain between 40 to 200 mg/kg Cd on a dry matter basis.

While sludges often contain high amounts of Cd, the amount found in soils usually range from 0.01 to 8.1 ppm Cd (Allaway, 1968; Frank et al. 1976). Therefore, applying sludge to the soil usually results in an increase in the Cd concentration in the soil.

The concentration of Cd found in the soil that influences plant uptake has been found to depend on several factors; soil pH, organic

matter, cation exchange capacity, and the presence of competing ions (John et al. 1972; Street et al. 1978).

Thies and Richter (1979) determined that the pH of a sludge treated soil affects both the extent of absorption as well as the degree of precipitation of Cd. They found a soil with pH 5 resulted in the greatest Cd concentration and plant Cd uptake. While raising the pH of the sludge-treated soil will not reduce the content of Cd in plants to the same level as that of an untreated soil, the content will be significantly lower at pH 8 as opposed to pH 5 (John, 1976; Miller et al. 1976; Boggess et al. 1978).

Cation exchange capacity is thought to control metal availability through the process of electrostatic and covalent bonding of the heavy metal cations to the soil colloids (Keeney et al. 1977; Sims and Boswell, 1978). Griffin et al. (1976) concluded that removal of Cd from solution is due to a cation exchange-adsorption reaction affected by pH and ionic competition. It was also noted that montmorillinitic type clays adsorbed five times more total metal ions than did kaolinitic type clays. Sims and Boswell (1978) indicated that there was a reduction in mobility of Cd due to the application of bentonite (2:1 montmorillinitic type clay) to the soil. This reduction in soil availability and plant uptake was related to the cation exchange capacity and pH, but the individual effects could not be separated due to both the high cation exchange capacity and the alkaline nature of bentonite, which confounded the determination.

The adsorption of Cd was also related to the organic matter content (John et al. 1972; Haghiri, 1974). Haghiri (1974) suggested that organic matter retards plant uptake due to its high cation exchange capacity rather than chelating ability. The net concentration of Cd in the soil will increase as long as the organic matter input is maintained. When the input of organic matter is ceased, the concentration of Cd in the soil solution will increase to toxic levels for plants (Haghiri, 1974; John, 1972). Hinesly et al. (1977), however, disagreed with this protective capability of organic matter.

They reported a decrease in the organic content in a soil after ceasing the addition of sludges. This resulted in a decline in the concentration of Cd in the plant tissue. The fate of Cd when the input of organic matter has ceased has not been completely determined, but the fact that the amount of extractable Cd increases with the amount of organic matter or organic carbon has been cited by several researchers (Maclean, 1976; John et al. 1972; Sims and Boswell, 1978).

Some significant relationships have been shown involving the uptake of other heavy metals with Cd. Baker et al. (1975) found that by applying Zn, Se, Mo, or Zn plus Cu reduced the Cd content of soybeans. Webber and Beauchamp (1979) obtained a high correlation between Cd and Zn concentrations in corn stover which suggests a definite relationship between the uptake and the storage of Cd and Zn.

John (1976) noted that as the amount of P in the soil solution was increased from 25 to 125 ppm, there was a decrease in the Cd concentration of both the leaves and the stems of oats. Boggess et al. (1978) showed

a reduction of 50% or more in the Cd content of maize shoots with the addition of P to the soil. This reduction in Cd in a P deficient soil may be due mainly to the increase in plant growth as opposed to a decrease in the uptake of Cd by the shoots (John, 1976; Boggess et al., 1978). Other interactions with Cd have been observed with elements such as: Cu, N, K, Pb, Ca and Fe (Cunningham et al., 1975; Giordana and Mortvedt, 1976; John, 1976; Hassett et al., 1976; Iwai et al. 1975).

The uptake of Cd is not only affected by the previously mentioned factors, but research has shown that different species, varieties and types of plant tissue contain different Cd concentrations with similar application rates. The concentration of Cd in legumes and cereal crops is usually lower than that of leafy vegetables (Bingham et al., 1975). Vegetables, such as spinach, lettuce, curlycrese and radishes, that are grown on sludge-amended soils contained 75, 70, 80, and 21 $\mu\text{g/g}$ Cd respectively in the edible portion while corn grain, rice and soybeans contained 2, 2, and 7 $\mu\text{g/g}$ Cd respectively.

Chaney and Giordana (1977) reported corn leaf concentrations of Cd as 27 $\mu\text{g/g}$ and 1.4 $\mu\text{g/g}$ for corn grain. Cadmium concentration in corn grain has been found to vary from 3 to 15% less than the Cd content in the leaf (EPA 430/09-76-013). Jones et al. (1975) found the highest levels of Cd in corn grain at a pH 6.0.

Forages such as bermudagrass, alfalfa and tall fescue contained 9.4, 8.2, and 17.3 $\mu\text{g/g}$ Cd respectively (Chaney and Giordana, 1977). Many crops may contain relatively high concentrations of Cd and not show symptoms of toxicity.

John and Van Laerhoven (1976) found differences in yield and leaf Cd concentrations among lettuce cultivars. This indicates that Cd uptake and translocation may be due to genetic control and proper selection of cultivars may aid in reducing the chance of contaminating food chains.

Mercury

Mercury occurs widely in low concentrations in the biosphere and has been recognized as a toxicant for many years. Mercury (Hg) enters the atmosphere as a result of burning coal and the smelting of Cu, Pb and Fe ores (Frank et al., 1976). By settling with the particulate matter in the atmosphere, Hg will contaminate both plants and soil. Mercury can also be added to the soil and to plants by being applied as a constituent of some herbicides, fungicides and pesticides (Ormrod, 1978; Estes et al., 1973). Application of sludges to a soil will also increase the Hg content due to Hg concentrations in sludges ranging from 0.6 to 11.2 mg/kg (Galloway et al., 1977).

Soils generally contain relatively low quantities of Hg, ranging from 0.02 to 0.2 ppm with an average concentration being approximately 100 ppb (Potter and Standiford, 1975). But Frank et al. (1976) found that surface soils of orchards in Canada ranged from 0.03 to as much as 1.14 ppm Hg and accounted the high concentrations to the previous applications of pesticides that contained Hg. Almost all of this increase was found in the top 5 cm. of the soil (Ross and Stewart, 1962).

When Hg is added to the soil, it may either volatilize, react with the exchange complex of the clay and organic matter, be converted by bacteria to more toxic forms, or be taken up by plants (Lipsey, 1975; Rogers, 1979; Gracey and Stewart, 1974). Rogers (1979) found that Hg was volatilized after being applied to a soil in different compounds. The most water soluble Hg compounds, $\text{Hg}(\text{NO}_3)_2$, HgCl_2 , and $\text{Hg}(\text{C}_2\text{H}_3\text{O}_2)_2$ lost from 12 to 38% of the applied Hg within 144 hours. With a less soluble compound, HgO , losses varied from 6 to 19% loss, while HgS , an insoluble form, lost only 0.2 to 0.3% of the applied Hg. The amount lost in each case depended on the form added, moisture content and the soil texture. In every instance, the loss rates were smallest with soils having large amounts of clay. Maclean (1974) found that with added Hg, 41% of the total applied was retained by a sandy soil, 84% for a clay, 67% for a sand-clay mixture, and 97% for a peat.

Mercury compounds have a high affinity for soil surfaces and organic matter; therefore, the highest concentration of Hg is found in the surface horizons of soils (Loehr et al. 1979; Gracey and Stewart, 1974; Mills et al., 1975).

Plant content of Hg generally increases as the amount of applied Hg increases. Mercury content has been found to also increase due to the application of Hg containing pesticides to the leaves of plants. Irrespective of the mode of entry, Hg is readily translocated throughout the plant (Ross and Stewart, 1962). Lipsey (1975) found significant amounts of Hg being absorbed and translocated by maize seedlings germinating in methyl mercury hydroxide. Root growth inhibition

occurred when the roots contained a Hg concentration in excess of 10 ppm, while shoots were inhibited at 1 ppm. Methyl mercury completely inhibited photosynthesis by chloroplasts at 1 ppm and blocked the oxidation of malate at a concentration of 2.5 ppm.

John (1972) suggested that, generally, the highest concentration of Hg was in the roots of vegetables as compared to the remainder of the plant, except in the case of spinach leaves and pea vines. Most plants range in Hg content from 0.01 to 0.20 ppm except when high levels of Hg have been added and then the concentration may exceed 0.5 ppm (EPA 430/9-76-013, 1976).

Since the above ground parts of the plants generally contain low quantities of Hg, there is little hazard of contamination of terrestrial food chains (Chaney, 1973). With such an high affinity for clay surfaces and organic matter, Hg proposes little threat of polluting the groundwater.

Lead

Lead is considered to be a potentially toxic element to humans and animals. Plants, however, exhibit a low potential toxicity for this nonessential element. Lead (Pb) is found to be a natural component of uncontaminated soils due primarily to the content of Pb in the earth's lithosphere (Lindsay, 1979). Typically, soils contain on the average of 20 ppm Pb, but many range in concentration from a trace to 1,200 ppm (Aubert and Pinta, 1977).

The origins of Pb as a pollutant have not been clearly determined, but several studies have been conducted to try to determine the relative importance of various Pb sources. Dedolph et al. (1970) conducted an experiment with perennial ryegrass and radishes to establish the source of tissue Pb. It was found that Pb was of both soil and air origin, but not from simulated rainfall. The amount of Pb uptake from the soil remained relatively constant, while the Pb concentration in the leaves is related to the Pb concentration in the atmosphere.

Traffic density affects Pb concentration in the soil and vegetation (Goldsmith et al., 1976; Lagerwerff and Specht, 1970; Page and Ganje, 1970; Motto et al., 1976). High concentrations of Pb in soils are associated with the greatest traffic volume and decrease significantly with increasing distance from the highway as well as with depth throughout the profile. The major effect of traffic along the highway has been found within 32 meters (Lagerwerff and Specht, 1970; Motto et al., 1970). Traffic densities below 30 vehicles per km² resulted in no Pb accumulation, while higher densities, greater than 200 motor vehicles per km² surface Pb has been found to be two to three times higher than normal (Goldsmith et al., 1976). Page and Ganje (1970) found that over a 40 year period in Los Angeles Pb had accumulated in the top 2.5 cm of the soil and ranged from 15 to 36 ppm. This is considerably lower than the amount thought necessary to be toxic to plants.

Plants absorb Pb through the leaves and roots. But this element exhibits little retranslocation. The flowering and fruiting parts of

the plant usually contain the lowest Pb concentration. Plants that are grown near highways have high concentrations of Pb in the aerial portion of the plant as opposed to the roots, and this concentration decreases with distance from the highway (Motto et al., 1970). Goldsmith et al. (1976) reported that grasses grown 6 meters from the highway contained 69.76 $\mu\text{g/g}$ dry weight, while at 50 meters, 24.85 $\mu\text{g/g}$ was found. Vegetables that are grown within 8 meters of the road may contain 80 to 115 ppm Pb (Cannon and Bowles, 1962).

Lead content of soils has been found to be increased by the addition of industrial and municipal sludges (Gaynor and Halstead, 1976; Streng, 1976). Domestic wastewater contains from trace amounts to as much as 500 mg/kg, while industrial wastes may contain up to 8,300 mg/kg (Galloway and Jacobs, 1977). Lead added with sludge reduces its potential hazard of toxicity when it is incorporated into the soil (Loehr et al., 1979). This decrease in potential hazard is due to Pb reacting with clays, phosphates, hydroxides, sesquioxides, carbonates, and organic matter after being added to the soil. All of these reactions with Pb greatly reduce its availability (Baker et al., 1975).

Lead tends to concentrate near the soil surface due to it being strongly fixed by the soil. Lead fixation by soil is accomplished by Pb^{2+} being hydrolyzed and polymerized, and is less active as pH increases (Leeper, 1972; Loehr et al., 1979). John (1971) found that soluble Pb was immobilized in the surface horizons and ranged from 0.5 to 180.4 ppm in agricultural soils. Subsurface soil horizons ranged from 1.9 to 17.3 ppm Pb. Chisholm (1972) noted an increase in Pb concentration in

the 0 to 15 cm depth when PbHAsO_4 was added to the soil, but did not note an increase in the Pb concentration in the subsoil.

Organic matter has been found to form complexes with Pb thus reducing the amount of Pb in solution (Mortenson, 1963). Several researchers have reported an inverse relationship between organic matter and the Pb concentration in solution (Maclean et al., 1969; Zimdahl et al., 1976).

Lead uptake by plants decreases with an increase in pH, cation exchange capacity, temperature, and available P (Miller et al., 1975; Arvik et al., 1974; Cox et al., 1972). Miller et al. (1975) observed an inverse relationship between the uptake of Pb by corn and soybeans and the Pb sorption capacity of the soil. Addition of lime can both alleviate root inhibition and restrict the translocation of Pb to the shoot.

Lead affects some plants differently than others. Corn growth and yield have been found unaffected by the addition of Pb to the field (Baumhardt and Welch, 1972). The content in corn grain has been found not to be altered, while the leaf and stem contents are increased. This indicates that translocation occurs from the roots to the plant tops, but not from the stover to the grain which would result in an accumulation of Pb in the stover (Malone et al., 1974).

The uptake of Pb by corn is directly proportional to the transpiration rates with a reduction of photosynthesis. Rolfe et al. (1975) found that when concentrations reached levels above 60 ppm Pb in loblolly pines, photosynthesis was reduced by 11% and the transpiration rate steadily declined.

Nickel

Nickel is commonly found in plant tissue in concentrations ranging from 10 to 100 ppm (EPA 430/9-76-013). Vaneslow (1966) suggested that nickel (Ni) concentrations between 50 and 100 ppm on a dry matter basis would result in Ni toxicity. Toxicity problems, as well as high concentrations of Ni, have been primarily associated with plants grown on soils derived from serpentine. Serpentine contains approximately 500 ppm Ni and arises from metamorphosed ultrabasic rocks (Aubert and Pinta, 1977). Soils generally contain from 10 to 1,000 ppm Ni with a typical value being 40 ppm (Bohn et al. 1979).

The concentration of Ni in soils is affected by organic matter, Fe and Al sesquioxides, texture and parent material. Nickel is characteristically noted for its ability to be held tightly by negatively charged surfaces, especially as the pH increases (Leeper, 1972; Aubert and Pinta, 1977). Therefore, by increasing the organic matter content in the soil there will be a reduction in the amount of soil-extractable and plant Ni (Halstead et al., 1968). Nickel content throughout the soil profile decreases with depth (Korte et al., 1976) in well drained agriculture soils (Swaine, 1960). In a poorly drained soil with a pH 5, Ni would be most mobile or available for plant uptake and there is less variation of concentration with depth in the profile (Hinesley et al., 1972). Flooding of soils will also increase the mobility of Ni within the soil (Siew Kee, 1962).

Availability of Ni can be greatly reduced if the pH of the soil is maintained above 6.5, reducing the chance of plant toxicity (Galloway

and Jacobs, 1977). Halstead et al. (1969) found that by pretreating acid soils with Ni and by varying the amount of applied lime resulted in the amount of soil-extractable and plant Ni being reduced at higher pH levels. They also noted that Ni uptake may be enhanced by the addition of phosphate. Crooke (1956) observed that soon after adding a Ni salt to a soil, 50% of the Ni could be recovered by NH_4OAc extract. However, if the soil pH was raised from 5.2 to 7.0 with the addition of lime, only 20% of the added Ni could be recovered.

Nickel content in plants has been found to be increased by factors such as sludge applications and their proximity to smelters and highways. Sludges vary greatly in their Ni content ranging from 110 to over 2,800 mg/kg dry weight (Galloway and Jacobs, 1977). Application of 502 metric tons/ha of sludge increased the Ni content in the plant from less than 4.5 to 16.3 ppm (Cunningham et al., 1975). Kirkham (1975) found that an application of 28 metric tons per hectare per year of sludge for 35 years increased the Ni content in the soil as well as the concentration in the corn plant, but the amount of Ni found in the corn grain remained unchanged.

Fescue grown at 8, 16 and 32 meters from a highway showed a decrease in Ni concentration with increasing distance from the highway (Lagerwerff et al., 1970). The soil content not only declined with distance from the highway, but also with depth throughout the profile.

The quantity of Ni in soils and plants can be altered substantially by the application of sludges as well as by other means. However, it is not expected that the concentration of Ni in plants will be sufficiently

increased to cause a toxicity or pose a threat to the food chain, but it is still imperative that proper management be applied.

Titanium

Titanium is a transition metal that has not been found to be essential for plant growth. It is, however, important due to its large quantity in soils. Soils typically range in concentrations between 1,000 and 10,000 ppm with the average falling around 4,400 ppm (Swaine, 1955). As soils weather, they become enriched with oxides and hydroxides of Al, Fe, and Ti (Bohn, 1979). Due to the stability of titanium oxides during the weathering process, they are often used as indicators of soil formation. Titanium oxides are the most common form of titanium found in soils and rock.

The most common Ti minerals that are found in soils are: rutile, anatase, brookite, leucoxene, illmenite, pseudorutile, and sphene (Hutton, 1977). Rutile, anatase and brookite are polymorph forms of titanium dioxides (TiO_2) with the most common of the three in nature being rutile (Mason and Berry, 1968). Illmenite (FeTiO_3) and rutile are generally found in igneous rocks and are mined as a source of titania that is used for paint pigments and production of metals (Mason and Berry, 1968).

Titanium generally does not exist in soluble form in weak acids or in water and is thus not very mobile throughout the soil profile (Hutton, 1974; Aubert and Pinta, 1977).

Plants generally contain low concentrations of Ti according to Bohn et al. (1979). They reported that plants typically contain 0.0008 percent Ti on a fresh weight basis.

Silver

Silver is widely distributed in nature but commonly occurs in small amounts. Natural waters usually contain less than 0.002 $\mu\text{g/mL}$ and soils contain on the average 0.05 ppm Ag (Lindsay, 1979; Ediger, 1973). Due to its insolubility, Ag rarely occurs in toxic amounts to plants, of which it is not essential (Vanselow, 1966). He indicated that Ag usually ranges in concentrations below 0.5 ppm in plant tissue, but may go as high as 1 ppm.

Manganese

Manganese is an essential element for the growth of plants and is commonly found in the soil at rather high concentrations, ranging from 20 to 3,000 ppm (Lindsay, 1979). Manganese (Mn) occurs in soils in three oxidation states: Mn(II), Mn(III), and Mn(IV) (Page, 1962). Acid soils usually contain the manganous (MnII) form, while most neutral to alkaline soils contain the higher valence forms, Mn(III) and Mn(IV). The divalent cation Mn(II) is soluble, mobile, and easily available to plants. The trivalent Mn(III) and the tetravalent cations Mn(IV) are almost insoluble, nonmobile, and unavailable to plants (Aubert and Pinta, 1977; Sauchelli, 1969).

Soil Mn has been found to be more available to plants grown in soils with a low pH as compared to those soils of higher pH. Page (1962)

suggested that the decreasing availability of Mn at pH's higher than 6.0 has been more attributed to the complexation of Mn by organic matter and not due to either the biological oxidation of Mn or the formation of higher oxides of manganese. Soil pH is the most important factor concerning Mn availability and must be maintained below 6.0 to be available to plants, but if the pH drops to 4.0, toxicity to the plant may occur.

The concentration of Mn in plants varies with plant species and location. The normal range for plant Mn is from 10 to 100 ppm (Bohn, 1979). The availability of Mn to the plant is primarily due to soil pH, unless application of sludges are made to the soil. Industrial sludges generally range in Mn content from 640 to 6100 mg/kg of dry matter (Galloway and Jacobs, 1977). It was found that when Mn was applied to the soil at rates of 0, 26, 54, and 113 μg , Mn per gram of soil contained in industrial sludge, resulted in wheat leaves contained 188, 113, 197, and 339 $\mu\text{g/g}$ tissue respectively (Sims et al., 1978).

Manganese applied to a Mn deficient soil with a high pH resulted in an increase in the Mn concentration in maize and barley (Singh and Steenberg, 1974). The distribution within each species differed. The barley contained the highest concentration of Mn in the roots followed by the blades, and then the sheaths. Maize, however, contained the most Mn in the blades, followed by the roots and the sheaths. When Mn was applied at a rate of 50 and 100 ppm, the maize blades contained 272 and 318 ppm Mn, the sheaths 106 and 124 ppm Mn, and the roots contained 169 and 248 ppm Mn respectively. The older leaves and nodes of the plants

contained higher concentrations of Mn than did the younger plant tissue. The Mn content of the individual leaves increased from the stalk to the leaf tips.

Barium

Barium is neither essential nor beneficial to the growth of plants or animals. It is, however, present in soils in concentrations ranging from 100 to 3,000 ppm (Vanselow, 1966; Bohn, 1979). Barium generally does not concentrate in plants that are utilized as a food source (Leeper, 1972). Vanselow (1966) noted Ba concentrations in plant tissue range from 3 ppm to as high as 150 ppm.

Due to its potential toxicity, standards have been set for maximum levels of Ba that can be found in drinking waters, 0.1 mg/l (Train, 1979). Application of waste material containing Ba must therefore be monitored carefully, especially since industrial waste may contain from 2,600 to 6,400 mg/kg barium, while domestic waste may contain between 600 and 1,000 mg/kg (Galloway and Jacobs, 1977).

Sodium

Sodium is required for normal growth of some plants, such as saltbush (Atriplex vesicaria) (Brownwell and Wood, 1957) and Halogeton glomeratus (William, 1960), but it is not essential for all plants (Lunt, 1966). Plants absorb Na as the Na^+ ion from the soil solution. Soils generally range in concentration from 750 to 7500 ppm Na (Lindsay, 1979). Sodium is one of the more loosely held ions in the soil and is easily lost in leaching water (Tisdale and Nelson, 1975).

Selenium

Selenium is not an essential element for plants, but is essential for the proper growth of animals (Sauchelli, 1969). Plants can, however, contain large quantities of selenium (Se) that may be toxic to animals. Concentrations of Se in animal diets are recommended to be 0.04 ppm, but if the level in the diet is in excess of 4 to 5 ppm, toxicity to the animal generally occurs (Allaway, 1968).

The accumulation of Se in plants depends on the species of plant, environmental factors, age, and the stage of growth of the plant (Rosenfeld and Beth, 1964; Sauchelli, 1969). The nature of the Se compound in the soil also affects plant uptake (Allaway, 1968). Soils generally contain Se in the range of 0.09 to 2.0 ppm Se (Lindsay, 1979; Gupta et al., 1975).

The most common forms of Se found in soil are the selenates (SeO_4^{2-}) and selenites (SeO_3^{2-}) (Bohn, 1979; Allaway, 1968). Selenate ions are commonly taken up by plants and are usually at higher concentrations under well aerated alkaline conditions. Selenium ions are not usually available in neutral to acid soils due to the formation of ferric selenites (Allaway, 1968; Cary, 1967). The amount of Se throughout the soil profile has been highly correlated with organic matter, Fe and Al oxides, and with clay content under certain conditions (Le'vesque, 1974). Plant content of Se has been related to the amount found in the soil. Kubota (1967) found that 80% of the plants sampled from the regions known to be low in Se resulted in concentrations of less than 0.05 ppm. In areas where soil Se has been found to be adequate, more than 80% of the samples contained greater than 0.1 ppm Se.

The desired range for Se in plant material to be consumed by animals is narrow, but if the concentration is too low, additions of Se to the soil will increase the plant concentration. Hartly (1967) found by adding Se to the soil, the content found in the forage increased, but the first harvest contained in excess of the tolerated range for animals. After the first harvest, the concentration declined to below toxic levels. By applying Se in the form of selenite as opposed to selenates, may reduce this chance of toxicity.

Selenium has been found to be contained in sludges from sixteen U.S. cities in concentrations ranging from 1.7 to 8.7 ppm (Furr et al., 1976). The Se contained in sludges is low in availability to plants and is usually in the form of elemental Se, selenites that are bound to hydrous iron oxides, heavy metal selenides, or as trimethylselenonium salts (Loehr et al., 1979).

Zinc

The content of Zn commonly found in soil ranges in concentration from 10 to 300 ppm, with an average of 50 ppm (Lindsay, 1979). This content could be significantly increased with the addition of waste material to the land.

Zinc is an essential element for both plants and animals. Zinc (Zn) is taken up by plants as the Zn^{2+} ion and if excess quantities exist, toxicity will occur. Leeper (1972) indicated phytotoxic levels of Zn in the plant at concentrations of 500 ppm. Factors such as phosphates, soil pH, organic matter, and type of clay minerals have been

found by several researchers to influence the uptake and availability of Zn.

The most common form of Zn found in soils are ZnS , ZnCO_3 , $\text{Zn}(\text{PO}_4)_2$, ZnO , $\text{Zn}(\text{OH})_2$ and Ca zincates. These compounds are more commonly found at pH values greater than 7.2 (Clarke and Graham, 1968) and are more soluble as the pH decreases.

When the amount of available P is increased in soils, Zn deficiencies often occur, especially if the natural Zn content of the soil is low. Ellis et al. (1964) reported a 50% reduction in field bean yields after previous applications of high levels of P. With 48.9 kg/ha P and no added Zn, the Zn content of the plant leaves grown on a low residual P treatment was 19.3 ppm, while on the high residual P treatment the leaves contained 14.7 ppm Zn.

Zinc has been found to react with organic matter in the soil resulting in the formation of both soluble and insoluble Zn complexes. Hodgson et al. (1966) found that under most circumstances, 60% of the soluble Zn in soil occurs in the form of Zn-organic complexes.

Galloway and Jacobs (1977) reported zinc concentrations in domestic waste material to be 100 to 1800 mg/kg, and 3200 to 14000 mg/kg dry matter in industrial waste. Schaver et al. (1980) found that with the addition of sludge at a rate of 0, 20, and 60 dry metric tons per hectare, resulted in a significant increase in the Zn content in the edible portion of vegetables for increases in sludge over a two year period. Similarly, Sidle et al. (1976) found higher concentrations of Zn in sludge treated areas on both corn silage and reed canarygrass.

Corn silage contained 45.1, 43.2, and 32.5 $\mu\text{g/g}$ dry matter Zn in 1972 for 7.5, 3.75 and 0 cm of effluent applied per week.

Arsenic

Arsenic has long been noted for its effects on humans and animals, but more recently has been credited with toxicity to plants that are grown on soils that have been contaminated by pesticides, lead smelters and industrial and organic wastes. In the 1930's and 1940's, inorganic arsenicals were extensively used in apple orchards, cotton fields and as soil sterilants (Bishop and Chesholm 1972; Dorman and Coleman 1937). With the prolonged use of large quantities of inorganic arsenicals, there resulted a distinct accumulation of As in soils of some areas. Bishop and Chesholm (1962) reported 10 to 125 ppm As in Canadian orchard soils while on areas that had not been contaminated, the concentration ranged from a trace to 8 ppm.

In the 1950's, organic arsenical herbicides such as methanearsonic acid (MSMA) and disodium methanearsonate (DSMA) were synthesized. These compounds were found to be largely inactivated by soils (Dickens 1967).

Porter and Peterson (1977) reported that plants had evolved a tolerance to the arsenate ion as opposed to the arsenite which was found to be more toxic to plants.

Arsenic has been found to closely resemble the phosphate ion as far as its activity in soil. Jacobs and Keeney (1970) found that arsenic uptake may be enhanced by phosphate displacing the adsorbed arsenate ion is increased as the pH ranges from 3 to 13, with phosphate being adsorbed only slightly more.

Iron

Iron has been established as an essential micronutrient for all plants and animals. It comprises about 5% of the earth's lithosphere, ranking fourth in abundance among chemical elements behind O, Si, and Al. Iron occurs in soils as oxides, hydroxides, phosphates and in the lattice structures of primary silicate and clay minerals. Primary and secondary minerals such as biotite, hornblende and chlorite weather resulting in additions of small amounts of Fe to the soil that may be absorbed by plants. Iron in these minerals is primarily in the ferrous, Fe^{2+} , state and after weathering changes to the ferric, Fe^{3+} , ion (Lindsay 1979; Krauskopf 1972).

Plants may obtain Fe by root absorption of ionic forms and organic salts, and by absorption through the leaves if foliar applied. Ferric iron, Fe^{3+} , may be absorbed by plants, but the metabolically active form is ferrous iron, Fe^{2+} (Krauskopf 1972).

The ability of a root to reduce Fe^{3+} to Fe^{2+} has been found to be related to Fe absorption. This reduction occurs at the outer surface of the plasmalemma and it is suggested that the source of electron is from within the cell (Brown et al. 1972).

The major factor that influences the solubility and availability to plants of Fe is soil pH. At soil pH values above 5.5, plants will not accumulate Fe to toxic levels due to its reduced solubility. At pH values below 5.0, Fe is more available but is rarely accumulated in plants to toxic levels. Deficiencies of Fe are more prevalent than are toxicities.

Domestic waste materials have been reported to contain 2300 to 12,600 mg/kg Fe, while industrial wastes contain between 64,500 and 225,000 mg/kg Fe on a dry weight basis (Galloway and Jacobs 1977).

CHAPTER III

MATERIALS AND METHODS

Field Methods

Two experiments were conducted at a DuPont plant site located near New Johnsonville, Tennessee. The experimental plots were established on a Paden Silt Loam (fine-silty, mixed, thermic Glossic Fragiudult) 6-10 percent slope in 1978 and 1979. The sludge material was obtained from E. I. DuPont de Nemours and Company, Inc., Pigment Division and was applied to both corn and fescue plots.

All plots were arranged in a randomized complete block design with four replications. Sludge applications were made at four rates, 0, 22.0, 45.0 and 90.0 metric tons per hectare, each at two levels of lime, 9.0 and 18.0 metric tons per hectare, except the 0 rate. The sludge and lime material was applied to the corn plots prior to planting in 1978 and 1979. The sludge-lime treatments were applied using a flail-type spreader. Fescue plots were established and the treatments were applied in the fall of 1978.

Corn plots consisted of six rows of corn (Zea mays L. cv. "Pioneer brand 3147 F-11") 9.1 m in length. The outside two rows served as guard rows, while 3 m of the center two rows were harvested at the milk stage as fodder. All plots were equally fertilized with 170 kg N, 40 kg P and 110 kg K per hectare prior to planting.

Ear-leaf samples were taken at silking, and dried in a forced air oven at 70 C. Fodder was cut by hand, weighed, subsampled and dried in

gas-heated dryers. Grain samples were taken in late fall for laboratory analysis. All tissue samples were ground in a Wiley mill to pass a 2.0 mm sieve and stored in plastic bags for analysis.

Fescue plots were established in the fall of 1978 and the sludge-lime treatments were applied. Tall fescue (Festuca arundinacea Schreb cv. "Kentucky 31") plots were 3 m by 6 m in size and were sampled in June, July and August, 1979, by cutting a 50.8 cm strip at random through each plot. The samples were weighed, subsampled and dried in a forced air oven at 70 C. The samples were ground in a Wiley mill to pass a 2.0 mm sieve and stored for analysis.

Ceramic cup lysimeters were buried in plots in both corn and fescue at 30, 61, 91 and 122 cm depth approximately 1 m apart within each treatment across one replication. Samples were taken by applying approximately one bar tension on each lysimeter with a hand vacuum pump in the fall and early spring of 1978, 1979 and 1980. The water samples were placed in plastic bottles and stored until laboratory analysis could be conducted.

Soil samples were taken from each corn plot at 0-15 and 15-30 cm depth and 0-8 and 8-15 cm depth from the fescue plots. Samples were taken at the 30, 61, 91 and 122 cm depth from within the same plots as the lysimeters were placed. All soil samples were allowed to air dry and were ground to pass a 2.0 mm sieve and stored.

Laboratory Methods

Laboratory analyses were conducted in the Plant and Soil Science laboratories at The University of Tennessee at Knoxville. Elemental

analysis was conducted on a Perkin-Elmer, Model 5000, Atomic Absorption Spectrophotometer. The elements Ag, Ba, Cu, Fe, Mn, and Zn were determined by flame atomic absorption throughout this research. Sodium was analyzed by flame emission. Cadmium, Ni, Pb and Ti were analyzed using a graphite furnace attachment. A deuterium lamp was used in the analysis of Ni and Pb for the correction of background interferences. To obtain higher sensitivity readings, L'vov platforms were used with the graphite furnace for Pb determinations (Weltz et al. 1980). A Perkin-Elmer Mercury Hydride System, MHS-10, was utilized in the determination of As, Hg, and Se. Absorbance readings and instrument settings maintained were those recommended by the manufacturer.

Determination of Ba, Cd, Fe, Mn, Ni, Na, and Zn were conducted by dry ashing plant material in a muffle furnace at 500 C for 4 hours and dissolving the ash in 3 N HCl. The supernatant was filtered through Watman No. 42 filter paper and brought to a constant volume of 25 ml.

Copper, Pb and Ag concentrations in plant tissue were determined by dry ashing the tissue in a muffle furnace. The ash was dissolved in 3 ml of 2 N HNO₃, filtered, and diluted to 25 ml.

Tissue samples for the determination of As and Se were dried at 85 C. One tenth gram of tissue was placed in a 25 ml Parr Acid Digestion Bomb with 3 ml of HNO₃, heated for one hour at 165 C and made up to 10 ml volume. The samples were then analyzed by hydride generation.

Soil samples that were obtained at four depths within each treatment were analyzed for heavy metals by extracting the samples with

0.005 M DTPA (diethylenetriaminepentaacetic acid), 0.1 M CaCl_2 , and 0.1 M TEA (triethanolamine) according to the procedure outlined by Lindsay and Norvell (1978) (see Appendix A).

Surface soils were analyzed for organic matter by the Walkley-Black method (see Appendix B) and soil pH was measured on a 1:1 soil/water ratio. Cation exchange capacity of the soil was determined for each treatment by the NH_4OAc procedure (see Appendix C).

Water samples were analyzed for all previously mentioned elements with the addition of pH, chlorides (see Appendix D), and conductivity.

Statistical Analyses

Data were analyzed at The University of Tennessee Computing Center on an IBM 370/3036 utilizing the Statistical Analyses System procedures outlined by Barr et al. (1979).

CHAPTER IV

RESULTS AND DISCUSSION

Sludge Analysis

The sludge material obtained from E. I. DuPont de Nemours and Co., Inc., is a by-product of Ti extraction from illmenite ore (FeTiO_3). As a result of this extraction process, the sludge has a $\text{pH} < 2.0$. As shown in Table 1, there are also various concentrations of heavy metals as well as a high concentration of chlorides found in the material. By utilizing the metal loading limits as summarized by Overcash and Pal (1979), the potential years that the material could be applied to the land could be calculated according to previous research. Based on the sludge analysis (Table 1), the number of years that it would be permissible to apply this sludge to a soil with a cation exchange capacity in the range between 10 and 15 meq/100 g is summarized in Table 2. From the calculations, it is apparent that the major limiting element that the sludge material contains is Mn. Manganese could be applied at the rate of 90 metric tons/ha (40 ton/ac) for 15 years before the maximum loading limit would be reached.

Yield

The application of various rates of sludge and lime to fescue plots resulted in no significant differences in the yields obtained in either June, July or August cuttings (Table 3). The total yield obtained from

Table 1. Total Analysis of the sludge material.

Properties	Concentration
pH	1.7
Moisture	21.8 %
Chlorides	1.5 %
Total carbon	30.0 %
Ag	0.14 µg/g
As	0.39 µg/g
Ba	9.3 µg/g
Be	0.2 µg/g
Ca	7510.0 µg/g
Cd	0.08 µg/g
Cr	28.8 µg/g
Fe	2.9 %
Hg	0.08 µg/g
K	92.0 µg/g
Mg	587.0 µg/g
Mn	608.0 µg/g
N	102.0 µg/g
Na	92.0 µg/g
Ni	2.1 µg/g
Pb	25.0 µg/g
Se	0.27 µg/g
Ti	1.3 %

Table 2. Estimated number of years of sludge application based on concentration of elements and the soil cation exchange capacity.

Metal	Maximum Metal Loading	Concentration in Sludge	Years of Application at Rate of
	(kg/ha/yr)	(μ g/ml)	(90 metric tons/ha)
Ag	200	0.14	17857
Cd	2	0.08	312
Hg	9	0.078	1442
Mn	750	608.	15
Ni	100	2.07	604
Pb	1000	25.3	494

Table 3. Mean yield for fescue harvested June, July, and August, 1979.

Treatment	Sludge	Lime	June	July	August	Total
	--metric tons/ha--		-----dry matter tons/acre-----			
1	0.0	0.0	0.85a*	0.52a*	0.25a*	1.63a*
2	22.0	9.0	0.88a	0.47a	0.25a	1.61a
3	45.0	9.0	0.89a	0.41a	0.27a	1.57a
4	90.0	9.0	0.89a	0.55a	0.26a	1.70a
5	22.0	18.0	0.91a	0.49a	0.25a	1.66a
6	45.0	18.0	0.73a	0.37a	0.20a	1.30a
7	90.0	18.0	0.71a	0.52a	0.28a	1.51a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

these three cuts was also found not to be significantly different. The yields ranged from 1.3 to 1.7 tons dry matter per acre.

Despite the applications of sludge, corn fodder yields were found not to vary significantly due to any treatment. Yields ranged from 6.1 to 7.9 tons dry matter per acre (Table 4).

Fescue Composition

Laboratory analyses of each cut of fescue resulted in a variety of concentrations. The concentrations of Fe, Ni, Cd, Pb, Nz, and Ti in the first cut (Table 5) were not affected by any treatment. The concentrations of Mn in the plant tissue, however, was significantly increased by the application of 90.0 metric tons/ha sludge. No concentration differences could be accredited to the level of lime, but fescue grown with 9.0 metric tons/ha lime contained more than the fescue grown with 18.0 metric tons/ha level of lime. These values were 357.8 and 258.8 $\mu\text{g/g}$ Mn respectively. No differences were found in Mn levels with any other treatment. Sodium concentrations were not affected by the treatments when compared to the control. However, a difference was noted between treatments 4 and 6. As shown in Table 6, Mn and Na concentrations in plant tissue of the July harvest were somewhat similar to the June harvest except treatment 3 was found to be no different than the check. The concentrations of Fe, Cd, Pb, and Ti were not affected as a result of the treatments. Iron and Cd concentrations in the plant tissue were lower than those values found in the tissue of the first harvest. Titanium, however, was more variable in its concentration when compared to the first harvest, but the check remained about the same.

Table 4. Mean height of corn and fodder yield for 1979.

Treatment	Sludge	Lime	Fodder Yield	Corn Height
	--metric tons/ha--		--tons/acre--	-----cm-----
1	0.0	0.0	6.62a*	224a*
2	22.0	9.0	7.18a	229a
3	45.0	9.0	6.89a	229a
4	90.0	9.0	7.22a	221a
5	22.0	18.0	7.91a	247a
6	45.0	18.0	6.87a	241a
7	90.0	18.0	6.06a	211a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

Table 5. Iron, Ni, Cd, Mn, Zn, Na, Ti, Pb, Se, and As concentration of fescue harvested June 1979 as affected by levels of sludge and lime.

Treatment	Sludge	Lime	Fe	Ni	Cd	Mn	Zn	Na	Ti	Pb	Se	As
---metric tons/ha---ppm---												
1	0.0	0.0	152.5a*	3.91a*	1.08a*	183.8c*	42.9a*	207.1ab*	0.85a*	0.08a*	0.014a*	0.136a*
2	22.0	9.0	137.3a	3.28a	1.08a	209.1bc	46.0a	193.5ab	0.85a	0.07a	0.008a	0.067a
3	45.0	9.0	134.1a	3.01a	1.05a	264.6bc	47.9a	249.6ab	1.13a	0.09a	0.011a	0.125a
4	90.0	9.0	138.3a	3.39a	1.17a	347.8a	48.4a	322.6a	0.92a	0.07a	0.008a	0.042a
5	22.0	18.0	147.6a	3.38a	1.10a	229.6bc	45.6a	234.7ab	0.88a	0.08a	0.006a	0.092a
6	45.0	18.0	119.1a	3.04a	1.01a	212.4bc	42.1a	137.1b	0.94a	0.08a	0.008a	0.100a
7	90.0	18.0	156.0a	3.94a	1.25a	285.8ab	49.2a	261.1ab	1.63a	0.08a	0.009a	0.117a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance according to Duncan's Multiple Range Test.

Table 6. Iron, Ni, Cd, Mn, Zn, Na, Ti, and Pb concentrations of fescue harvested July 1979 as affected by levels of sludge and lime.

Treatment	Sludge	Lime	Fe	Ni	Cd	Mn	Zn	Na	Ti	Pb
			---metric tons/ha---ppm---							
1	0.0	0.0	127.4a*	1.28b*	1.00a*	183.9b*	50.9bc*	232.8ab*	0.86a*	0.04a*
2	22.0	9.0	116.3a	1.55ab	0.56a	195.1b	50.1bc	151.8a	0.80a	0.04a
3	45.0	9.0	133.7a	1.33b	0.75a	265.7ab	59.8a	194.2ab	1.32a	0.05a
4	90.0	9.0	112.5a	2.20ab	0.82a	336.9a	56.8ab	266.5a	0.77a	0.04a
5	22.0	18.0	118.0a	2.46a	0.65a	210.1b	49.8c	175.8ab	0.72a	0.04a
6	45.0	18.0	146.1a	1.98ab	0.63a	192.9b	51.1bc	137.5b	1.35a	0.05a
7	90.0	18.0	117.1a	2.06ab	0.84a	306.8a	57.0ab	277.6ab	1.26a	0.03a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

Treatment 5 caused increase of Ni in the second harvest over both the check and treatment 3. The Ni concentrations found in the tissue of the second fescue harvest were lower than those found in the tissue of the first harvest. Contrastingly, Zn concentrations increased in the tissue of the second harvest. Plant Zn content was higher due to treatment 3 than the levels found in all treatments except 4 and 7.

Analysis of the plant tissue harvested at cut three showed an increase in concentrations of Ti, Zn, and Mn above those values obtained in the previous harvest (Table 7). The concentrations of Cd, Fe, Zn, Na, Pb, and Ti were not affected by the rates of sludge or lime applied at this harvest time. As in the previous harvests, Mn concentration again increased at the 90.0 metric tons/ha level of sludge while the Ni content found in the fescue treated with 90.0 metric tons/ha of sludge was increased over that in the check.

When comparing the concentrations of each element between each harvest, Na, Fe, and Cd decreased from June to August while the concentrations of Mn and Zn were increased.

Corn Composition

Treatment 4 was the only one to cause a significant increase in Mn levels in the ear-leaf at silking (Table 8). With the exception of treatments 2 and 7, the concentration of Na in the ear-leaf decreased below the 113.7 $\mu\text{g/g}$ found in the check. Zinc concentrations decreased with increasing sludge rates at the two high lime levels except for treatment 4, which increased to 104.3 $\mu\text{g/g}$. There was a trend to higher

Table 7. Iron, Ni, Cd, Mn, Zn, Na, Ti, and Pb concentration of fescue harvested August 1979 as affected by levels of sludge and lime.

Treatment	Sludge	Lime	Fe	Ni	Cd	Mn	Zn	Na	Ti	Pb
	---metric tons/ha---ppm---									
1	0.0	0.0	96.0a*	0.72b*	0.32a*	230.7b*	51.0a*	155.5a*	1.17a*	0.06a*
2	22.0	9.0	98.9a	1.20ab	0.35a	237.5b	56.5a	152.2a	0.91a	0.07a
3	45.0	9.0	100.9a	2.13ab	0.32a	272.3b	59.7a	162.3a	1.25a	0.06a
4	90.0	9.0	102.2a	2.15a	0.34a	388.6a	56.7a	191.3a	0.68a	0.06a
5	22.0	18.0	119.4a	1.36ab	0.36a	245.9b	52.3a	152.9a	1.04a	0.06a
6	45.0	18.0	101.4a	1.18ab	0.30a	249.1b	57.3a	121.0a	1.52a	0.05a
7	90.0	18.0	106.9a	2.32a	0.40a	409.9a	51.3a	146.8a	1.62a	0.05a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

Table 8. Iron, Ni, Cd, Mn, Zn, Na, Ti, Pb, As, and Se concentrations of corn ear-leaf sampled in 1979 at silking.

Treatment	Sludge	Lime	Fe	Ni	Cd	Mn	Zn	Na	Ti	Pb	Se	As
---metric tons/ha---ppm---												
1	0.0	0.0	83.2b*	0.78a*	0.29a*	264.3ab*	94.3ab*	113.7a*	2.18a*	0.07a*	0.008a*	0.063a*
2	22.0	9.0	93.5ab	0.58a	0.12a	216.3b	90.1ab	97.8ab	1.90a	0.07a	0.006a	0.056a
3	45.0	9.0	109.1ab	0.68a	0.24a	274.2ab	82.6ab	91.0b	2.12a	0.07a	0.010a	0.050a
4	90.0	9.0	136.4ab	0.74a	0.31a	345.0a	104.3a	91.0b	2.88a	0.06a	0.010a	0.038a
5	22.0	18.0	168.4a	0.76a	0.11a	234.5b	79.9ab	82.0b	2.39a	0.08a	0.007a	0.050a
6	45.0	18.0	103.0ab	0.68a	0.14a	194.2b	68.1b	83.6b	1.99a	0.09a	0.012a	0.086a
7	90.0	18.0	88.9b	0.69a	0.25a	252.4b	70.5b	94.0ab	1.47a	0.07a	0.008a	0.056a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

Fe concentration as a result of all treatments, but only treatment 5 resulted in a significant increase above the control.

Fodder samples as shown in Table 9 were analyzed and all elemental concentrations decreased below those values from the ear-leaf samples except for Cd. Especially dramatic were the decreases in Ti concentrations. Treatment 3 increased the Cd concentrations above that of other treatments. No differences were found in the concentrations of Ni, Pb, Ti, Zn or Mn in the fodder at any level of sludge-lime treatment.

Concentrations of Na detected in the fodder tissue did not vary significantly from the concentration of the control but treatment 3 contained more Na than treatment 7 with values of 41.4 and 22.3 $\mu\text{g/g}$ respectively. Iron concentrations were increased above the 21.5 $\mu\text{g/g}$ found in the control by treatment 3.

Corn grain analysis (Table 10) resulted in a decrease in the concentrations of all elements from those concentrations found in the fodder except in the case of Ni which increased. Application of treatment 3 resulted in a significant increase in Cd concentration between the other treatments.

Soil Water

Soil water samples were collected from ceramic cup lysimeters from four depths across each treatment. The moisture tubes were placed in the plots of fescue after establishment, while the corn plots were tilled and planted prior to inserting the tubes. Samples were taken

Table 9. Iron, Ni, Cd, Mn, Zn, Na, Ti, and Pb concentrations of corn fodder harvested in 1979.

Treatment	Sludge	Lime	Fe	Ni	Cd	Mn	Zn	Na	Ti	Pb
---metric tons/ha---ppm---										
1	0.0	0.0	21.5b*	0.11a*	0.20b*	149.1a*	65.9a*	34.4ab*	0.07a*	0.04a*
2	22.0	9.0	28.7ab	0.11a	0.19b	142.9a	77.6a	32.5ab	0.13a	0.05a
3	45.0	9.0	36.4a	0.15a	0.29a	150.6a	82.3a	41.3a	0.09a	0.03a
4	90.0	9.0	19.7b	0.10a	0.20b	105.2a	58.9a	37.1ab	0.01a	0.04a
5	22.0	18.0	23.5b	0.08a	0.17b	79.9a	56.9a	32.4ab	0.09a	0.05a
6	45.0	18.0	27.2ab	0.09a	0.18b	119.7a	68.6a	29.8ab	0.12a	0.04a
7	90.0	18.0	21.0b	0.06a	0.17b	108.7a	56.6a	22.3b	0.05a	0.04a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

Table 10. Iron, Ni, Cd, Mn, Zn, Na, Ti, and Pb concentration of corn grain sampled in 1979.

Treatment	Sludge	Lime	Fe	Ni	Cd	Mn	Zn	Na	Ti	Pb
---metric tons/ha---ppm---										
1	0.0	0.0	11.9a*	0.33a*	0.05b*	5.1a*	36.6a*	39.0a*	0.00	0.02a*
2	22.0	9.0	12.1a	0.67a	0.07b	4.8a	40.1a	37.9a	0.00	0.02a
3	45.0	9.0	14.8a	0.51a	0.12a	5.6a	43.2a	40.6a	0.00	0.01a
4	90.0	9.0	15.7a	0.85a	0.05b	5.0a	39.3a	36.4a	0.00	0.01a
5	22.0	18.0	14.8a	0.52a	0.06b	5.1a	40.2a	40.8a	0.00	0.02a
6	45.0	18.0	18.0a	0.42a	0.06b	4.5a	42.9a	38.8a	0.00	0.01a
7	90.0	18.0	19.8a	0.59a	0.06b	5.8a	40.3a	45.8a	0.00	0.01a

*Values followed by the same notation within each column do not differ at the 0.05 level of significance, according to Duncan's Multiple Range Test.

periodically throughout the year by applying one bar tension on each lysimeter.

Although minor changes were noted in conductivity and chloride concentrations, changes in metal concentrations were not found. Treatment of fescue plots with 45.0 metric tons/ha sludge resulted in little change in conductivity measurements as compared to the control on July 1979 as shown in Table 11 for all depths at which samples were taken, except for the 30 cm depth. The 45.0 and 90.0 metric tons/ha sludge level resulted in an increase in conductivity at 61 and 92 cm depths.

Chloride concentrations were not altered greatly except with the application of 90.0 metric tons/ha sludge. Changes in the concentration of other elements were not found.

According to Table 12, the highest conductivity and chloride levels found in soil water collected December 1979 were associated with the lowest sludge treatment, while the lowest values were found with the highest treatment levels. Treatments 2, 3, and 5 caused a slight accumulation of Ni in the soil water solution sampled in December. The concentration of the chlorides and conductivity of the soil water samples collected in December (Table 12) when compared to the soil water collected in July 1979 (Table 11) showed a considerable increase in December. Titanium concentrations also increased somewhat when the fescue received treatments 5 and 6 at all depths.

Generally, Mn concentrations were found to be increased more by the lowest sludge treatment while the highest sludge treatment resulted in a somewhat lower concentration.

Table 11. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentration in soil water collected at four depths in fescue plots, July 1979.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric ton/ha		cm		$\mu\text{mhos/cm}$	mg/l						
1	0.0	0.0	30	6.6	111	0	0.01	0.12	0.16	0.14	0.01	21.3
	0.0	0.0	61	7.0	295	41	0.00	0.02	0.15	1.23	0.01	11.1
	0.0	0.0	92	7.2	198	7	0.02	0.01	0.24	0.27	0.01	25.8
	0.0	0.0	122	6.7	307	49	0.00	0.02	0.97	1.33	0.01	44.6
2	22.0	9.0	30	6.5	255	31	0.01	0.00	0.17	0.66	0.01	17.5
	22.0	9.0	61	6.7	305	51	0.00	0.00	0.19	1.10	0.01	17.3
	22.0	9.0	92	6.9	177	14	0.00	0.00	0.20	0.03	0.01	29.7
	22.0	9.0	122	6.1	205	19	0.00	0.00	0.18	0.02	0.01	59.5
3	45.0	9.0	30	8.0	751	129	0.00	0.00	0.16	0.02	0.01	30.9
	45.0	9.0	61	6.7	280	36	0.00	0.00	0.18	0.32	0.01	43.5
	45.0	9.0	92	6.0	295	60	0.00	0.03	0.18	0.67	0.01	15.9
	45.0	9.0	122	7.0	280	27	0.00	0.00	0.22	2.01	0.01	31.3
4	90.0	9.0	30	6.9	265	0	0.00	0.13	0.20	0.03	0.00	7.1
	90.0	9.0	61	7.0	480	118	0.00	0.00	0.20	1.14	0.00	44.2
	90.0	9.0	92	7.0	228	26	0.00	0.00	0.21	0.25	0.00	45.3
	90.0	9.0	122	6.4	380	88	0.00	0.01	0.20	0.64	0.00	45.3
5	22.0	18.0	30	6.3	325	54	*	0.01	0.20	0.48	0.01	13.9
	22.0	18.0	61	7.1	265	37	0.01	0.02	0.21	0.08	0.00	12.6
	22.0	18.0	92	7.4	196	10	0.04	0.02	0.21	1.56	0.01	45.1
	22.0	18.0	122	7.1	370	84	0.00	0.00	0.21	2.20	0.00	36.8
6	45.0	18.0	30	7.5	465	60	0.00	0.12	0.20	0.02	0.00	6.5
	45.0	18.0	61	7.1	405	78	0.00	0.05	0.19	0.25	0.00	13.1
	45.0	18.0	92	7.5	335	44	0.00	0.01	0.20	0.26	0.00	20.3
	45.0	18.0	122	7.5	545	22	0.00	0.01	0.22	0.03	0.01	68.1
7	90.0	18.0	30	7.9	60	228	0.00	0.03	0.23	0.08	0.01	5.4
	90.0	18.0	61	7.7	595	110	0.02	0.02	0.20	1.58	0.01	13.0
	90.0	18.0	92	6.4	650	168	0.01	0.00	0.20	2.62	0.01	23.3
	90.0	18.0	122	6.4	215	20	0.00	0.00	0.24	0.30	0.01	41.7

*Insufficient sample.

Table 12. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentration in soil water collected at four depths in fescue plots, December 1979.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric tons/ha		cm		$\mu\text{mhos/cm}$	mg/l						
1	0.0	0.0	30	6.8	375	110	0.00	0.03	0.19	0.09	0.01	10.2
	0.0	0.0	61	6.8	370	94	0.00	0.03	0.20	0.02	0.01	10.0
	0.0	0.0	92	6.8	1060	331	0.02	0.03	0.18	1.44	0.01	42.8
	0.0	0.0	122	7.2	520	120	0.01	0.02	0.21	1.98	0.02	60.8
2	22.0	9.0	30	8.1	2100	652	0.00	0.03	0.15	0.01	0.01	4.6
	22.0	9.0	61	6.3	3000	1091	0.03	0.04	0.18	7.32	0.01	21.3
	22.0	9.0	92	5.1	2300	766	0.01	0.03	0.22	9.08	0.01	55.1
	22.0	9.0	122	4.9	3400	1221	0.10	0.03	2.14	1.92	0.01	72.5
3	45.0	9.0	30	6.7	340	81	0.01	0.00	0.13	0.51	0.00	10.9
	45.0	9.0	61	6.8	280	60	0.01	0.00	0.12	0.44	0.00	11.6
	45.0	9.0	92	5.0	775	230	0.01	0.00	0.12	4.03	0.00	28.6
	45.0	9.0	122	5.0	910	234	0.05	0.01	0.12	3.92	0.01	35.5
4	90.0	9.0	30	6.8	186	38	0.01	0.01	0.06	0.02	0.00	5.8
	90.0	9.0	61	6.6	275	55	0.00	0.02	0.08	0.14	0.00	11.7
	90.0	9.0	92	7.1	220	35	0.01	0.00	0.10	0.01	0.00	30.7
	90.0	9.0	122	6.4	380	98	0.02	0.00	0.12	1.95	0.00	42.7
5	22.0	18.0	30	7.2	2400	743	0.03	0.09	0.21	3.24	0.00	21.6
	22.0	18.0	61	*	*	*	0.03	0.08	0.20	3.29	0.00	21.6
	22.0	18.0	92	*	*	*	0.04	0.09	0.23	15.2	0.00	36.2
	22.0	18.0	122	*	*	*	0.06	0.08	0.27	5.24	0.00	64.7
6	45.0	18.0	30	6.6	1090	302	0.02	0.07	0.24	1.13	0.00	11.4
	45.0	18.0	61	6.2	1120	342	0.02	0.07	0.23	1.88	0.00	17.0
	45.0	18.0	92	5.1	620	185	0.03	0.08	0.22	1.10	0.00	38.7
	45.0	18.0	122	7.5	870	211	0.01	0.08	0.22	2.04	0.00	68.3
7	90.0	18.0	30	8.1	1490	375	0.03	0.00	0.12	0.00	0.01	5.6
	90.0	18.0	61	7.4	475	125	0.02	0.00	0.14	2.38	0.01	45.3
	90.0	18.0	92	6.3	510	149	0.01	0.05	0.15	0.63	0.01	20.5
	90.0	18.0	122	5.3	580	170	0.01	0.03	0.18	1.29	0.01	16.3

*Insufficient sample.

Table 13 indicates that the soil water collected from the fescue plots treated with 90.0 metric tons/ha had the highest conductivity at the 61, 92, and 122 cm depths. The other treatments caused increases but they were not as pronounced. This trend also held true for chloride concentration.

Nickel, Na, and Mn concentrations in soil solution were highest on the 90.0 metric tons/ha sludge treatments at the 61, 92 and 122 cm depths.

Soil solution collected from the tubes in the corn plots in August 1978 (Table 14) showed that the highest conductivity was generally associated with the highest level of sludge treatment. Nickel concentrations were increased considerably in some instances but there was not a definite trend. Manganese was highest throughout the soil profile at the 90.0 ton rate of sludge, especially at the 122 cm depth.

The concentration of Na found in soil solution in February was increased due to the application of treatment 6 and 7. Conductivity and chlorides tended to increase at the 30 cm depth for all treatments except treatment 5 (Table 15). Sodium concentrations were higher compared to the control for treatments 6 and 7.

In December 1979 (Table 16), the conductivity and chloride values were higher for the low sludge rates while Mn was highest at the higher sludge rates.

The soil solution in April 1980 as shown in Table 17, indicates higher conductivity and chloride levels being associated with the treatments at higher lime levels. All other elements were not changed from the check to any extent.

Table 13. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentration in soil water collected at four depths in fescue plots, April 1980.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric tons/ha		cm		$\mu\text{mhos/cm}$	mg/l						
1	0.0	0.0	30	6.6	150	24	0.00	0.00	0.40	0.13	0.00	6.1
	0.0	0.0	61	6.0	220	35	0.00	0.00	0.40	0.22	0.00	11.4
	0.0	0.0	92	6.5	100	13	*	0.00	1.10	0.01	*	7.0
	0.0	0.0	122	6.3	330	79	0.00	0.00	0.50	1.57	0.00	39.8
2	22.0	9.0	30	6.7	225	*	0.00	0.00	0.60	0.04	0.01	9.3
	22.0	9.0	61	6.7	240	54	0.01	0.00	0.60	1.47	0.01	11.8
	22.0	9.0	92	4.9	525	155	0.02	0.00	0.60	2.49	0.00	26.9
	22.0	9.0	122	5.3	530	151	0.02	0.00	0.70	1.58	0.00	21.2
3	45.0	9.0	30	8.0	525	11	0.00	0.00	0.10	0.02	0.01	2.5
	45.0	9.0	61	5.8	380	91	0.01	0.00	0.20	2.15	0.00	13.0
	45.0	9.0	92	5.3	450	117	0.01	0.00	0.30	1.25	0.00	24.9
	45.0	9.0	122	6.5	360	76	0.00	0.00	0.20	0.84	0.00	47.6
4	90.0	9.0	30	7.0	230	18		0.00	1.00	0.14	0.00	1.5
	90.0	9.0	61	6.5	960	283	0.02	0.00	0.70	1.61	0.00	11.7
	90.0	9.0	92	5.0	1930	602	0.03	0.00	0.70	4.11	0.01	64.3
	90.0	9.0	122	5.7	2600	803	0.05	0.00	1.10	4.71	0.01	89.6
5	22.0	18.0	30	*	*	*	*	*	*	*	*	*
	22.0	18.0	61	6.8	550	138	0.01	0.00	0.70	0.13	0.00	15.0
	22.0	18.0	92	7.0	675	184	0.01	0.00	0.80	1.94	0.00	38.9
	22.0	18.0	122	6.8	690	190	0.01	0.00	1.00	2.85	0.00	75.5
6	45.0	18.0	30	7.7	490	76	0.00	0.00	0.20	0.22	0.00	6.9
	45.0	18.0	61	6.4	550	128	0.01	0.00	0.30	1.22	0.00	9.5
	45.0	18.0	92	5.1	470	126	0.02	0.00	0.20	0.71	0.00	45.5
	45.0	18.0	122	7.6	625	119	0.00	0.00	0.40	1.40	0.00	62.3
7	90.0	18.0	30	7.9	390	31	0.01	0.00	0.60	0.12	0.01	2.5
	90.0	18.0	61	7.3	1100	301	0.01	0.00	0.50	0.04	0.00	13.3
	90.0	18.0	92	7.0	1690	504	0.04	0.00	0.60	5.31	0.01	55.2
	90.0	18.0	122	4.9	1720	527	0.04	0.00	0.50	3.45	0.01	73.7

*Insufficient sample.

Table 14. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentrations in soil water collected at four depths in corn plots, August 1978.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric tons/ha		cm		µmhos/cm	mg/l						
1	0.0	0.0	30		*	150	*	*	*	0.07	*	*
	0.0	0.0	61		*	90	*	*	*	*	*	*
	0.0	0.0	92	6.5	1120	9	0.22	0.00	0.00	6.44	0.01	32.3
2	0.0	0.0	122	6.5	2500	2	0.00	0.00	0.00	0.10	0.02	10.7
	22.0	9.0	30	*	*	100	1.46					
	22.0	9.0	61	6.3	540	9	0.08	0.00	0.00	0.01	0.00	19.0
3	22.0	9.0	92	6.9	750	120	0.14	0.00	0.00	0.14	0.01	25.1
	22.0	9.0	122	7.2	650	7	0.10	0.00	0.00	0.14	0.00	56.5
	45.0	9.0	30	*	*	7	0.62			2.32	0.03	
4	45.0	9.0	61	6.8	2500	120	0.39	0.00	0.00	6.47	0.01	48.4
	45.0	9.0	92	6.0	800	120	0.18	0.00	0.00	1.81	0.01	54.4
	45.0	9.0	122	6.5	2000	58	0.62	0.00	0.00	3.41	0.02	51.1
5	90.0	9.0	30	7.0	5500	36	1.96	0.00	0.00	6.92	0.02	28.4
	90.0	9.0	61	6.5	300	140	0.05	0.00	0.00	1.04	0.00	12.3
	90.0	9.0	92	*	1250		3.16	0.00	0.00	8.11	0.04	
6	90.0	9.0	122	6.5	5100	24	0.80	0.00	0.00	34.6	0.02	42.6
	22.0	18.0	30	*	*	36	*	*	*	0.02	*	*
	22.0	18.0	61	7.5	3100	110	0.66	0.00	0.00	0.03	0.00	16.3
7	22.0	18.0	92	6.9	1790	63	0.45	0.00	0.00	14.1	0.01	20.5
	22.0	18.0	122	7.3	940	20	0.17	0.00	0.00	0.32	0.00	12.1
	45.0	18.0	30	6.7	2600	57	0.95	0.00	0.00	1.44	0.02	38.7
8	45.0	18.0	61	6.8	1660	55	0.37	0.00	0.00	3.31	0.01	99.3
	45.0	18.0	92	6.8	630	120	0.07	0.00	0.00	0.56	0.00	48.8
	45.0	18.0	122	6.9	1675	160	0.57	0.00	0.00	1.14	0.01	66.8
9	90.0	18.0	30	6.6	3600	120	0.49	0.00	0.00	2.39	0.01	31.8
	90.0	18.0	61	6.5	1320	68	0.25	0.00	0.00	4.77	0.01	44.8
	90.0	18.0	92	6.6	3200	80	0.87	0.00	0.00	4.91	0.02	128.0
10	90.0	18.0	122	6.5	2000	140	4.47	0.00	0.00	24.4	0.04	192.0

*Insufficient sample.

Table 15. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentration in soil water collected at four depths in corn plots, February 1979.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric tons/ha	cm			$\mu\text{mhos/cm}$	mg/l						
1	0.0	0.0	30	6.4	1220	145	0.08	0.00	0.11	7.9	0.00	6.9
	0.0	0.0	61	4.6	2300	175	0.17	0.00	0.12	76.0	0.01	17.2
	0.0	0.0	92	4.6	2500	145	0.19	0.00	0.12	35.0	0.01	20.1
	0.0	0.0	122	4.9	875	130	0.07	0.00	0.11	2.5	0.01	17.5
2	22.0	9.0	30	5.2	4300	885	0.12	0.00	0.15	52.0	0.01	10.1
	22.0	9.0	61	4.7	1340	205	0.07	0.00	0.14	9.5	0.00	15.8
	22.0	9.0	92	6.6	890	130	0.05	0.00	0.12	2.0	0.00	26.3
	22.0	9.0	122	6.5	990	130	0.06	0.00	0.12	1.7	0.00	24.3
3	45.0	9.0	30	6.7	2600	890	0.07	0.02	0.08	8.0	0.00	15.1
	45.0	9.0	61	6.1	2600	635	0.08	0.01	0.09	7.3	0.00	31.7
	45.0	9.0	92	4.4	1600	465	0.11	0.00	0.10	9.3	0.00	49.7
	45.0	9.0	122	6.8	1140	245	0.04	0.01	0.08	0.7	0.00	51.3
4	90.0	9.0	30	6.6	5100	1574	0.11	0.00	0.16	27.0	0.01	11.3
	90.0	9.0	61	5.0	1625	420	0.09	0.00	0.13	8.7	0.00	9.7
	90.0	9.0	92	5.1	3100	935	0.10	0.00	0.14	6.7	0.01	16.1
	90.0	9.0	122	6.2	2800	965	0.11	0.00	0.13	2.0	0.01	22.1
5	22.0	18.0	30	7.4	880	155	0.06	0.00	0.12	0.1	0.00	2.6
	22.0	18.0	61	6.9	1960	420	0.10	0.00	0.14	0.1	0.00	5.5
	22.0	18.0	92	5.1	1440	270	0.13	0.00	0.14	9.2	0.00	10.3
	22.0	18.0	122	7.7	810	105	0.07	0.00	0.12	0.1	0.00	6.4
6	45.0	18.0	30	7.2	3600	625	0.08	0.01	0.11	4.1	0.00	16.1
	45.0	18.0	61	4.7	2200	395	0.09	0.01	0.09	8.9	0.00	61.0
	45.0	18.0	92	4.2	4700	915	0.09	0.01	0.11	6.0	0.01	49.5
	45.0	18.0	122	6.7	3800	845	0.28	0.01	0.13	50.0	0.00	80.2
7	90.0	18.0	30	6.8	7700	2404	0.18	0.00	0.15	36.0	0.02	28.8
	90.0	18.0	61	4.8	4900	1524	0.14	0.02	0.13	8.4	0.01	30.1
	90.0	18.0	92	5.9	6400	1974	0.21	0.00	0.15	28.0	0.01	84.9
	90.0	18.0	122	6.8	2800	745	0.07	0.00	0.11	1.5	0.00	148.0

*Insufficient sample.

Table 16. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentrations in soil water collected at four depths in corn plots, December 1979.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric tons/ha		cm		$\mu\text{mhos/cm}$	mg/l						
1	0.0	0.0	30	*	*	*	*	*	*	*	*	*
	0.0	0.0	61	6.8	220	17	0.05	0.16	0.00	0.14	0.00	4.1
	0.0	0.0	92	6.7	450	34	0.05	0.04	0.00	0.20	0.06	26.1
	0.0	0.0	122	6.8	440	26	0.05	0.02	0.00	0.16	0.01	31.9
2	22.0	9.0	30	8.0	1610	280	0.05	0.09	0.00	0.23	0.02	5.5
	22.0	9.0	61	8.0	1740	296	0.05	0.07	0.00	0.03	0.00	6.3
	22.0	9.0	92	7.2	1690	510	0.05	0.06	0.00	0.05	0.00	13.7
	22.0	9.0	122	7.2	750	182	0.05	0.02	1.66	4.23	0.00	74.9
3	45.0	9.0	30	*	*	*	*	*	*	*	*	*
	45.0	9.0	61	*	*	*	*	*	*	*	*	*
	45.0	9.0	92	6.4	500	84	0.00	0.02	0.00	1.89	0.00	34.2
	45.0	9.0	122	7.2	725	95	0.00	0.04	0.00	0.39	0.00	20.6
4	90.0	9.0	30	7.6	1230	217	0.09	0.05	0.00	0.03	0.00	31.0
	90.0	9.0	61	7.6	980	113	0.02	0.04	0.00	1.62	0.00	14.8
	90.0	9.0	92	7.8	675	56	0.00	0.03	0.09	16.4	0.00	42.4
	90.0	9.0	122	7.5	640	65	0.00	0.14	0.00	4.63	0.00	39.3
5	22.0	18.0	30	*	*	*	*	*	*	*	*	*
	22.0	18.0	61	*	*	*	*	*	*	*	*	*
	22.0	18.0	92	7.6	1850	269	0.01	0.06	0.00	0.30	0.00	14.6
	22.0	18.0	122	7.3	1700	300	0.00	0.05	0.00	10.8	0.00	25.1
6	45.0	18.0	30	*	*	*	*	*	*	*	*	*
	45.0	18.0	61	7.6	2000	390	0.02	0.08	0.00	0.01	0.00	7.1
	45.0	18.0	92	7.2	2600	647	0.04	0.06	0.00	0.82	0.00	15.3
	45.0	18.0	122	7.6	1640	312	0.00	0.03	0.00	0.05	0.00	21.8
7	90.0	18.0	30	*	*	*	*	*	*	*	*	*
	90.0	18.0	61	*	*	*	*	*	*	*	*	*
	90.0	18.0	92	6.2	270	23	0.00	0.03	0.00	1.48	0.00	17.7
	90.0	18.0	122	6.4	280	12	0.00	0.03	0.00	6.66	0.00	23.8

*Insufficient sample.

Table 17. Conductivity, pH, chlorides, Ni, Ti, Fe, Mn, Cd, and Na concentration in soil water collected at four depths in corn plots, April 1980.

Treatment	Sludge	Lime	Depth	pH	Conductivity	Chlorides	Ni	Ti	Fe	Mn	Cd	Na
	metric	tons/ha	cm		$\mu\text{mhos/cm}$	mg/l						
-----ppm-----												
1	0.0	0.0	30	*	*	*	*	*	*	*	*	*
	0.0	0.0	61	6.7	140	2	0.00	0.00	0.00	0.05	0.00	1.9
	0.0	0.0	92	6.2	160	7	0.00	0.00	0.00	0.07	0.00	9.9
	0.0	0.0	122	5.2	320	14	0.02	0.00	0.00	2.41	0.00	20.4
2	22.0	9.0	30	7.8	440	42	0.00	0.00	0.00	0.05	0.01	5.0
	22.0	9.0	61	7.3	525	48	0.01	0.00	0.00	2.12	0.01	21.3
	22.0	9.0	91	5.9	375	32	0.02	0.00	0.00	0.58	0.00	19.1
	22.0	9.0	92	5.9	375	32	0.02	0.00	0.00	*	*	*
3	22.0	9.0	122	6.4	280	15	0.06	0.00	0.00	0.12	0.05	28.0
	45.0	9.0	30	*	*	*	*	*	*	*	*	*
	45.0	9.0	61	8.1	380	8	*	0.00	0.00	0.01	0.01	1.3
	45.0	9.0	92	7.5	630	62	*	0.00	0.00	0.02	0.01	6.2
4	45.0	9.0	122	7.3	530	90	0.01	0.00	0.00	2.11	0.01	83.7
	90.0	9.0	30	7.4	220	17	0.00	0.00	0.00	0.09	0.01	1.7
	90.0	9.0	61	7.1	425	43	0.00	0.00	0.00	0.14	0.01	3.3
	90.0	9.0	92	7.0	380	50	0.00	0.00	0.00	0.97	0.01	27.7
5	90.0	9.0	122	8.0	570	54	0.00	0.00	0.00	0.04	0.01	18.2
	22.0	18.0	30	7.6	800	70	0.00	0.00	0.00	0.05	0.01	26.3
	22.0	18.0	61	8.0	835	75	0.00	0.00	0.00	0.10	0.01	41.9
	22.0	18.0	92	7.4	575	32	0.00	0.00	0.00	0.98	0.01	58.1
6	22.0	18.0	122	7.7	580	45	0.01	0.00	0.00	2.21	0.00	48.9
	45.0	18.0	30	*	*	*	*	*	*	*	*	*
	45.0	18.0	61	7.9	470	11	0.00	0.00	0.00	0.02	0.01	1.4
	45.0	18.0	92	8.0	880	85	0.00	0.00	0.00	0.03	0.01	6.3
7	45.0	18.0	122	8.1	820	87	0.00	0.00	0.00	1.13	0.01	10.5
	90.0	18.0	30	7.9	380	*	0.00	0.00	0.00	0.01	0.01	1.4
	90.0	18.0	61	7.9	480	51	0.00	0.00	0.00	0.01	0.01	1.2
	90.0	18.0	92	7.9	625	92	0.00	0.00	0.00	0.02	0.02	2.5
	90.0	18.0	122	7.9	750	8	*	0.00	0.00	0.02	0.01	7.2

*Insufficient sample.

Soil Samples

Soil samples were collected from surface soils to determine the effect of the applied sludge-lime treatments on organic matter content, pH, and cation exchange capacity. As shown in Table 18, all treatments caused at least a 0.5 pH unit increase in pH of the soil collected from the corn plots. The cation exchange capacity and organic matter were not affected by any treatment. The organic matter content of the soil was found to be somewhat higher than would be expected. This may be explained by cropping history since the area where the plots were located had previously been under continuous sod. Fescue plots (Table 19) gave the same general trend as the corn plots as a result of the treatments.

Analysis of the soil samples extracted with DTPA showed occasional elevated values over those samples from the check plot with the exception of Cd which varied little over all treatments in the corn plots (Table 20). The same trend appeared in the fescue plots (Table 21) with the exception of Mn which showed a tendency to increase as a result of the sludge applications.

Table 18. Organic matter, pH, and cation exchange capacity of soils collected from corn plots as influenced by applications of sludge and lime.

Treatment	Sludge	Lime	Organic Matter	pH	Cation Exchange Capacity
	--metric tons/ha--		%		meq/100g
1	0.0	0.0	2.7	5.4	10.8
2	22.0	9.0	2.6	6.3	11.2
3	45.0	9.0	2.8	6.2	10.5
4	90.0	9.0	2.8	6.0	10.7
5	22.0	18.0	2.5	5.9	10.1
6	45.0	18.0	2.2	6.1	10.7
7	90.0	18.0	2.8	6.1	10.9

Table 19. Organic matter, pH, and cation exchange capacity of soils collected from fescue plots as influenced by applications of sludge and lime.

Treatment	Sludge	Lime	Organic Matter	pH	Cation Exchange Capacity
	--metric tons/ha--		%		meq/100g
1	0.0	0.0	3.3	6.4	10.6
2	22.0	9.0	3.4	6.9	10.7
3	45.0	9.0	3.5	6.9	10.8
4	90.0	9.0	3.7	7.0	11.1
5	22.0	18.0	3.4	7.2	10.5
6	45.0	18.0	3.7	6.8	10.8
7	90.0	18.0	3.7	7.1	10.6

Table 20. Nickel, Zn, Fe, Cd, Cu, and Mn concentration extracted with DTPA from soil collected at 30, 61, 92, and 122 cm depths in corn plots.

Treatment	Sludge	Lime	Depth	Ni	Zn	Fe	Cd	Cu	Mn
	metric tons/ha		cm	-----ppm-----					
1	0.0	0.0	30	0.05	0.7	7.49	0.02	0.40	2.89
	0.0	0.0	61	0.06	0.7	4.28	0.03	0.32	3.11
	0.0	0.0	92	0.07	0.7	7.90	0.05	0.35	8.16
	0.0	0.0	122	0.09	1.0	6.91	0.03	0.31	6.13
2	22.0	9.0	30	0.06	1.3	5.51	0.03	0.37	6.40
	22.0	9.0	61	0.08	1.1	8.60	0.04	0.84	1.50
	22.0	9.0	92	0.19	1.1	8.54	0.02	0.76	0.92
	22.0	9.0	122	0.20	1.2	8.19	0.04	0.25	2.43
3	45.0	9.0	30	0.05	0.7	9.54	0.03	0.50	0.78
	45.0	9.0	61	0.06	0.7	7.20	0.03	0.35	5.74
	45.0	9.0	92	0.31	1.9	7.96	0.02	0.68	1.15
	45.0	9.0	122	0.36	2.1	9.83	0.03	0.57	0.69
4	90.0	9.0	30	0.06	0.2	6.91	0.04	0.44	4.39
	90.0	9.0	61	0.07	0.6	6.74	0.05	0.41	9.86
	90.0	9.0	92	0.13	0.9	7.26	0.03	0.55	0.65
	90.0	9.0	122	0.25	0.4	5.92	0.03	0.35	0.32
5	22.0	18.0	30	0.61	0.4	6.43	0.04	0.52	9.30
	22.0	18.0	61	0.04	0.4	1.46	0.04	0.48	8.62
	22.0	18.0	92	0.09	0.5	3.82	0.04	0.62	1.35
	22.0	18.0	122	0.12	0.4	3.82	0.03	0.56	2.43
6	45.0	18.0	30	0.05	0.6	8.78	0.05	0.29	0.64
	45.0	18.0	61	0.08	1.1	6.97	0.03	0.44	2.58
	45.0	18.0	92	0.20	1.4	6.62	0.04	0.24	1.03
	45.0	18.0	122	0.19	1.4	6.03	0.04	0.25	0.91
7	90.0	18.0	30	0.00	0.2	4.17	0.03	0.22	9.37
	90.0	18.0	61	0.00	1.2	3.82	0.04	0.88	4.93
	90.0	18.0	92	0.52	1.7	2.98	0.04	0.51	0.91
	90.0	18.0	122	0.59	2.2	7.42	0.04	0.70	1.20

Table 21. Nickel, Zn, Fe, Cd, Cu, and Mn concentration extracted with DTPA from soil collected at 30, 61, 92, and 122 cm depths in fescue.

Treatment	Sludge	Lime	Depth	Ni	Zn	Fe	Cd	Cu	Mn
	metric tons/ha		cm	-----ppm-----					
1	0.0	0.0	30	0.21	1.1	6.87	0.03	0.28	0.54
	0.0	0.0	61	0.29	1.1	6.92	0.03	0.25	0.22
	0.0	0.0	92	0.18	1.1	6.07	0.04	0.10	0.74
	0.0	0.0	122	0.22	1.1	6.69	0.05	0.11	0.67
2	22.0	9.0	30	0.25	0.9	9.78	0.04	0.22	1.78
	22.0	9.0	61	0.18	0.6	7.20	0.04	0.11	6.33
	22.0	9.0	92	0.30	2.0	3.73	0.04	0.24	6.98
	22.0	9.0	122	0.17	1.4	5.18	0.05	0.10	2.77
3	45.0	9.0	30	0.38	2.2	8.77	0.03	0.32	1.64
	45.0	9.0	61	0.32	1.9	2.43	0.05	0.41	4.08
	45.0	9.0	92	0.11	0.6	4.23	0.02	0.10	1.45
	45.0	9.0	122	0.23	1.1	8.65	0.05	0.12	1.07
4	90.0	9.0	30	0.24	1.2	7.06	0.04	0.28	2.17
	90.0	9.0	61	0.12	0.6	4.23	0.05	0.09	2.96
	90.0	9.0	92	0.15	1.4	7.01	0.03	0.09	0.84
	90.0	9.0	122	0.19	1.0	6.05	0.02	0.09	0.63
5	22.0	18.0	30	0.44	1.6	7.16	0.02	0.47	9.01
	22.0	18.0	61	0.29	1.5	5.83	0.05	0.24	7.24
	22.0	18.0	92	0.10	0.6	4.61	0.02	0.10	1.67
	22.0	18.0	122	0.17	1.1	6.41	0.00	0.13	5.58
6	45.0	18.0	30	0.26	0.8	8.94	0.03	0.15	5.06
	45.0	18.0	61	0.09	0.2	2.27	0.05	0.04	0.60
	45.0	18.0	92	0.13	0.6	4.59	0.05	0.09	0.65
	45.0	18.0	122	0.26	1.1	8.46	0.04	0.16	0.67
7	90.0	18.0	30	0.37	1.4	3.02	0.04	0.18	5.58
	90.0	18.0	61	0.18	0.6	6.04	0.05	0.08	3.23
	90.0	18.0	92	0.19	0.7	5.93	0.05	0.10	1.41
	90.0	18.0	122	0.26	1.6	7.46	0.03	0.20	1.50

CHAPTER V

SUMMARY AND CONCLUSIONS

Research was conducted at an E. I. DuPont de Nemours and Co., Inc., plant site near New Johnsonville, Tennessee, to observe the effects of applying 0, 22, 45, and 90 metric tons/ha of an industrial sludge to corn and fescue. Each sludge rate was applied with 9 and 18 metric tons/ha lime except for the 0 rate. The sludge and lime was applied together using a flail-type spreader. Treatment applications were made to the fescue plots after the establishment of a sod, while the applications were applied to the corn plots a few weeks prior to planting.

Fescue samples were collected in June, July, and August 1979, and analyzed for selected elements and to obtain yield measurements. Similarly, corn ear-leaves were sampled at silking, while the fodder and grain samples were collected near maturity for analysis. Yield data was obtained from fodder weights.

Evaluation of the quality of water in the soil profile was performed by placing ceramic cup lysimeters in each treatment across one replication at depths of 30, 61, 92 and 122 cm. These samples were analyzed for pH, conductivity, concentration of chlorides, and Ni, Ti, Fe, Mn, Cd and Na.

Soil samples were taken at depths, corresponding to those at which the lysimeters were placed for heavy metal analysis. Surface samples

were also taken for the purpose of determining the effect the treatments had on organic matter, pH, and cation exchange capacity.

As a result of this study, the following conclusions were made:

1. Corn and fescue yields were not significantly affected by sludge and lime applications as high as 90 metric tons/ha and 18 metric tons/ha respectively.
2. Elemental concentrations in plant tissue at all stages of growth did not reach levels that would be considered toxic to plants and/or result in harmful effects on animal or human health.
3. Manganese concentrations were found to be significantly increased in fescue, corn ear-leaf and fodder as a result of applying 90 metric tons/ha sludge.
4. Cadmium concentrations tended to decrease with crop maturity in both corn and fescue.
5. Chloride concentration in soil water samples increased during the winter months as a result of increased rainfall and leaching. leaching.
6. Nickel concentration in soil water tended to increase due to the 90 metric ton/ha rate of sludge.
7. The pH of the soil water samples was not altered by any rate of the lime and sludge.
8. Soil samples indicated that Ni, Fe and Mn concentrations tended to increase somewhat as a result of the sludge-lime applications.

It must be emphasized that the results presented in this investigation show only initial effects of the sludge amendments. Should these applications be repeated over an extended period of time, additional monitoring of the site and the vegetation growing thereon would seem to be essential. The acid nature of this material seems to be the limiting factor in land application. Additional research is needed to gain a more complete evaluation.

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APPENDICES

APPENDIX A

PROCEDURE FOR SOIL EXTRACTION

BY THE DTPA METHOD

1. Allow samples to air dry at room temperature, grind to pass a 2.0 mm sieve and store.
2. Weigh a 10.00 gram sample and place in 125 ml Erlenmeyer flask.
3. Add 20 ml of a mixture of 0.005 M DTPA, 0.01 M CaCl_2 and 0.1 M TEA adjusted to pH 7.30.
4. Shake for 2 hours on a rotary shaker.
5. Filter the solution through Whatman No. 42 filter paper.
6. The filtrate is ready for analysis.

APPENDIX B

WALKLEY-BLACK METHOD FOR DETERMINING

ORGANIC MATTER

1. Grind soil to pass a 40 mesh screen and store.
2. Weigh 1.00 gram of soil and place in 500 ml Erlenmeyer flask.
3. Add 10 ml of $1N$ $K_2Cr_2O_7$ and 20 ml of concentrated H_2SO_4 .
4. Swirl gently and allow to set for 30 minutes on an asbestos pad.
5. Add 200 ml of distilled H_2O and 10 to 12 drops of Orthophenanthroline indicator.
6. Titrate with $0.5 N$ $FeSO_4$ and note color change from green to brick red.
7. If more than 8 ml of the available 10 ml of $K_2Cr_2O_7$ is reduced, repeat the determination with less soil.

APPENDIX C

AMMONIUM ACETATE, pH 7, METHOD FOR DETERMINING CATION EXCHANGE CAPACITY

1. Grind soil to pass a 2.0 mm sieve and store.
2. Weigh 25 grams of soil and place in 250 Erlenmeyer flask.
3. Add 50 ml NH_4OAc , stopper, shake, and allow to set overnight.
4. Transfer contents to a Buchner funnel fitted with Whatman No. 42 filter paper.
5. Filter using gentle suction and leach 200 ml NH_4OAc over a period of no less than 1 hour.
6. Transfer leachate to 250 ml volumetric flask and retain for analysis.

APPENDIX D

DETERMINATION OF CHLORIDES BY THE SILVER NITRATE METHOD

1. Use a 100 ml sample or a suitable portion diluted to 100 ml.
2. Titrate samples and standards in the pH range 7 to 10 directly.
Adjust samples not in this range with 1 N H_2SO_4 or 1 N NaOH.
3. Add 1.0 ml K_2CrO_4 indicator solution.
4. Titrate with standard silver nitrate titrant to end point.
5. Establish the reagent blank value by the titration method.

VITA

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