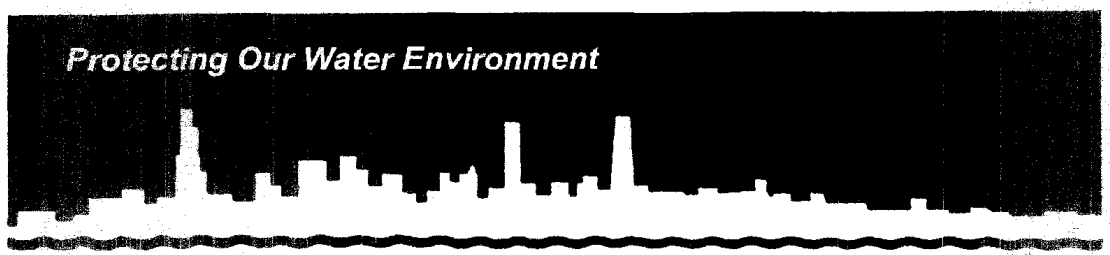

Metropolitan Water Reclamation District of Greater Chicago

RESEARCH AND DEVELOPMENT DEPARTMENT

REPORT NO. 04-3

AN INVESTIGATION OF SALINITY IN BIOSOLIDS GENERATED BY THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO

February 2004

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AN INVESTIGATION OF SALINITY IN BIOSOLIDS GENERATED BY THE METROPOLITAN WATER RECLAMATION DISTRICT OF GREATER CHICAGO

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DISCLAIMER

Mention of proprietary equipment and chemicals in this report does not constitute endorsement by the Metropolitan Water Reclamation District of Greater Chicago.

SUMMARY AND CONCLUSIONS

An investigation was conducted to determine the salinity level of the Metropolitan Water Reclamation District of Greater Chicago's (District's) biosolids, and to determine how the soluble salt concentration and chemical composition is affected by the District's standard biosolids processing trains. The following are the major conclusions of the investigation:

1. Salinity levels in digester draw biosolids from three District water reclamation plants (WRPs) decreased in the order John E. Egan (Egan) WRP > Stickney WRP > Calumet WRP.
2. Elevated salinity levels in digester draw biosolids from the Stickney, Calumet, and Egan WRPs that occur seasonally may be due to increased production of ammonia and alkalinity during anaerobic digestion resulting from higher volatile solids content of digester feed in February through April as compared to the volatile solids content of digester feed during summer and fall.

3. Road de-icing salts consisting of sodium chloride are only a minor component of biosolids salinity.
4. The soluble salt present in digester draw and centrifuge cake biosolids is composed primarily of ammonium bicarbonate.
5. Centrifuging of digester draw biosolids and lagoon dewatering of low solids processing train digester draw biosolids result in approximately the same reduction of soluble salt concentrations and have little effect on soluble salt chemistry.
6. Air-drying biosolids makes them aerobic, and it results in the oxidation of sulfides to soluble sulfate and the nitrification of ammonia to nitrate. As a result, biosolids pH drops during air-drying, and the predominant soluble salt component becomes ammonium sulfate.
7. Biosolids sodium content does not pose a significant limitation to the growth of most plant species, but it may restrict the use of some very sensitive tree species where heavy biosolids applications are made. This will need

to be researched before definitive conclusions can be drawn.

8. Although the high ammonia-N concentrations in biosolids are rapidly dissipated at the completion of air-drying, the concentrations may be high enough at the time of initial land application to inhibit germination and the establishment of turf and other ornamentals. Biosolids intended for use as a soil substitute or heavy soil conditioner should be managed very carefully if the concentration of unionized ammonia-N is greater than 2.0 mg/L on a saturation paste extract basis. The Biosolids Utilization and Soil Science Section can determine this within 24-hours of receiving a biosolids sample and can provide technical assistance for management of biosolids with high ammonia-N content.

9. Results of four annual salinity surveys indicated that biosolids salinity levels were high enough to limit the performance of turf and many ornamental species when biosolids are used as a topsoil substitute or soil amendment.

10. Biosolids can be subjected to a rapid screening procedure to determine the electrical conductivity and ammonia-N concentration prior to use. This will enable the District to identify biosolids that are best suited for use as a soil amendment or topsoil substitute. The rapid screening method has been developed, and it can be conducted in one day using an ion chromatograph. The Biosolids Utilization and Soil Science Section can determine biosolids EC within 24-hours of receiving a sample, and can provide technical assistance for management of biosolids with high EC.

11. Biosolids should be managed very carefully if they are intended for use as a soil substitute (no incorporation or mixing with soil) at unirrigated sites and have an EC_{corr} greater than 5.0 dS/m, or at irrigated sites and have an EC_{corr} greater than 8.0 dS/m.

12. Biosolids should be managed very carefully if they are intended for use as a soil conditioner (incorporation into soils to make 50/50 blend) at unirrigated sites and they have an EC_{corr}

greater than 9.0 dS/m, or at irrigated sites and they have an EC_{corr} greater than 12.0 dS/m.

13. There is little risk of boron toxicity from the heavy use of biosolids as a topsoil substitute or soil amendment. Some fruit and nut trees are sensitive enough to boron to be affected adversely by biosolids, but they are not typically grown in Illinois.

INTRODUCTION

The District has been utilizing biosolids for land reclamation for over 30 years. Much of the District's early experience with the use of biosolids for land reclamation is associated with the reclamation of land that was strip-mined for coal in Fulton County, Illinois. This reclamation project utilized biosolids as a soil conditioner to convert the marginal mine spoil soils into productive agricultural lands. The biosolids were utilized to gradually build up the soil organic matter, improve soil tilth, and provide crop nutrients. This was accomplished by making repeated annual applications of biosolids which were applied to the land at rates desirable for mine spoil reclamation (25 tons/acre annually or not to exceed 125 tons/acre in a five year period).

In the late 1980s and into the 1990s, the District undertook reclamation projects of a different nature. These projects utilized biosolids as a soil conditioner, or in some cases as a soil substitute, and the biosolids were applied at a one-time high application rate (typically 500 to 1,000 tons/acre applied in one year). This occurred at the coal refuse pile reclamation sites in Fulton County, Illinois, and in the Chicago Metropolitan Area at landfills and Controlled Sol-

ids Distribution Program sites. In this practice the biosolids were used to immediately convert marginal soils into very fertile, organic matter rich topsoils, or to substitute for topsoil by capping marginal soils or coal refuse material which were unsuitable for supporting vegetation.

When biosolids are applied to land in heavy, one-time applications of 500 to 1,000 tons/acre, which provides a 6 to 12 inch thick layer of biosolids, it is expected that the vegetation will be initially growing in a root zone consisting mainly of biosolids. There is very little dilution of the biosolids by incorporation and mixing with soil. The biosolids chemical property that most limits the establishment and performance of vegetation with this practice is biosolids salinity. The District's biosolids contain higher concentrations of essential plant nutrients and organic matter than surface soils, and they are also higher in soluble salts (Granato et al., 2000; Granato et al., 1998; Rodgers and Anderson, 1995).

Soil and biosolids salinity are determined by extracting with water (at various soil to water ratios) and measuring the electrical conductivity (EC) of the resulting extract. The electrical conductivity of the water extract increases in proportion to the amount of soluble salts that dissolve in the

water and ionize into charged cations and anions. When the amount of water used to extract the soil or biosolids is just enough to fill all of the pore space in the solids, the extraction ratio is referred to as the saturation paste extract, and the resulting measured electrical conductivity is designated as EC_e. The EC_e is considered to be the most accurate measure of the salinity in soil solution for plant roots.

The effect of soil salinity on plant performance is well documented (Maas, 1986; Mass and Hoffman, 1977). The United States Department of Agriculture's United States (U.S.) Salinity Laboratory, at the University of California in Riverside, California, has summarized the effects of salinity on the germination and growth of dozens of species of grasses, ornamentals and vegetables (Maas, 1986; Francois, 1982; Francois and Clark, 1979; Mass and Hoffman, 1977; Bernstein, et al., 1972). The U. S. Salinity Laboratory has defined four general ranges of soil extract EC_e which effect plants. These ranges are classified as sensitive, moderately sensitive, moderately tolerant, and tolerant to soil salinity, as shown in Table 1 (Mass and Hoffman, 1977). Soils having an EC_e between 6.0 and 10.0 dS/m limit the growth of even the salinity tolerant plants (Table 1), and soils with an EC_e

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TABLE 1

RANGES OF SATURATION PASTE EXTRACT EC (EC_e) EFFECTING THE GROWTH OF PLANTS THAT ARE SENSITIVE, MODERATELY SENSITIVE, MODERATELY TOLERANT, AND TOLERANT TO SOIL SALINITY

Plant Salinity Tolerance Classification	EC_e Range For Initiation of Growth Reduction
	-- dS/m --
Sensitive	0 to 1.2
Moderately Sensitive	1.2 to 3.0
Moderately Tolerant	3.0 to 6.0
Tolerant	6.0 to 10.0

greater than 10.0 dS/m are considered to be unsuitable for crops and most other plants (Mass and Hoffman, 1977).

The District's biosolids have had a measured EC_e in the 6.0 to 10.0 dS/m range and even higher (Granato et al., 2000; Granato et al., 1998; Rodgers and Anderson, 1995), and these biosolids have been demonstrated to inhibit the germination of some species of turf grass and native plants under unirrigated conditions (Granato et al., 1998).

Because biosolids salinity is high enough to pose a concern for the successful use of the District's biosolids as a topsoil substitute in some situations, a preliminary study was undertaken to document the levels and variability of biosolids salinity, to characterize the chemistry of the soluble salts, and to observe the changes in salinity that occur during biosolids processing through the standard District processing trains. This report summarizes the results of this preliminary study.

MATERIALS AND METHODS

The work described in this report was conducted between 1997 and 2001.

Annual Survey of Salinity in Biosolids from the District Solids Management Areas

Starting in 1998 samples were collected from biosolids that were stacked for final disposition at the District's solids management areas. This included the Calumet East and West drying areas, which process biosolids from the Calumet WRP; and the Harlem Avenue Solids Management Area (HASMA); Lawndale Avenue Solids Management Area (LASMA) including Vulcan and Marathon; Ridgeland Avenue Solids Management Area (RASMA); and the Stony Island Solids Management Area, which process biosolids from the Stickney WRP. Composite samples were taken from the surface 12 inches of at least two sides of each pile. Care was taken to exclude the thin surface layer of biosolids that had become completely air-dried.

Samples were stored refrigerated (5°C) in sealed plastic containers until they were analyzed. Each sample was analyzed for solids content, using a moisture balance, and was subjected to a 2:1 (volume water:biosolids fresh weight) extraction. The extract was analyzed for EC, ammonia-N, nitrate-N,

and pH. Some samples, selected at random, were also subjected to saturation paste extraction. This extract was analyzed for EC, ammonia-N, nitrate-N, and pH.

Investigation of Salinity in Digester Draw and Centrifuge Cake Biosolids from the Stickney, Calumet, and Egan WRPs

Digester draw and centrifuge cake biosolids from the Stickney, Calumet, and Egan WRPs were analyzed on a monthly basis. Digester draw samples were obtained from February 1998 through May 1999, and the centrifuge cake samples were obtained from February 1998 through March 1999. Monthly during these time periods, one weekly composite sample of both digester draw and centrifuge cake biosolids were split from the samples normally analyzed at the Stickney, Calumet, and Egan analytical laboratories. The samples were stored refrigerated (5°C) in sealed plastic containers until they were analyzed. Each sample was analyzed for solids content using a moisture balance.

Digester draw samples were beyond the saturation point and did not require any extraction. Therefore, they were centrifuged in the laboratory and the supernatant was utilized for analysis. Centrifuge cake biosolids samples from the three WRPs were subjected to saturation paste extraction prior to centrifuging.

Supernatant from the centrifuged extracts was analyzed for pH, EC, chloride, sulfate, total phosphorus, ammonia-N, nitrate-N, fluoride, alkalinity (expressed in this paper as bicarbonate), potassium, calcium, magnesium, sodium, zinc, cadmium, copper, chromium, nickel, lead, manganese, iron, aluminum, and boron.

Investigation of Changes in Biosolids Salinity During Air-Drying on Paved Beds

Drying cells at the Calumet East and West and LASMA solids management areas were selected for this investigation. Specifically, cells 4w and 2e were sampled at LASMA after being filled with low solids processing train biosolids (anaerobic digestion → lagoon dewatering and aging → air-drying) in August 1997. Cells 1 and 4 were sampled at the Calumet West drying site after being loaded with low solids processing train biosolids in September 1997, and cell 3 was sampled at the Calumet East drying site after being loaded with high solids processing train biosolids (anaerobic digestion → centrifuging → lagoon aging → air-drying). Biosolids drying was complete in May and June of 1998.

Composite samples of the biosolids were taken from across the selected cells immediately after completion of their initial loading and at completion of the drying cycle. The

samples were stored refrigerated (5°C) in sealed plastic containers until they could be analyzed. Each sample was analyzed for solids content using a moisture balance. Samples were subjected to saturation paste extraction and were subsequently centrifuged.

Supernatant from the centrifuged extracts was analyzed for pH, EC, chloride, sulfate, total phosphorus, ammonia-N, nitrate-N, fluoride, alkalinity (expressed in this paper as bicarbonate), potassium, calcium, magnesium, sodium, zinc, cadmium, copper, chromium, nickel, lead, manganese, iron, aluminum, and boron.

Biosolids Extraction Procedures

The saturation paste extraction was performed as prescribed by Rhoades (1996), except that the saturation paste was allowed to stand for only one hour instead of two prior to centrifuging to obtain extract. Sample size varied between 200 and 300 g fresh weight.

The 2:1 extraction was conducted by weighing 15.0 g (fresh weight) of biosolids into 50 mL conical plastic centrifuge tubes and adding 30 mL of ultra pure water. The mixture was shaken for 1 hour. Both the saturation paste and the 2:1 extracts were centrifuged to obtain a clear supernatant for analysis.

RESULTS AND DISCUSSION

The study of salinity in the District's biosolids consisted of the following activities:

1. Sampling the digester draw and centrifuge cake from the Stickney, Calumet, and Egan WRPs, and determining the salinity levels and salt composition over a 16-month period. This was done to determine the effects of centrifuging on salinity levels and salt composition, and to determine whether the salinity levels fluctuate seasonally.
2. Sampling biosolids on paved drying cells at the beginning and end of the drying cycle to determine changes in the salinity levels and salt composition during lagoon aging and the air-drying process.
3. Sampling biosolids stacked for final disposition at the District's eight solids management areas that produce an air-dried product (Calumet East and West, LASMA, Marathon, Vulcan, Stony Island, RASMA and HASMA) to determine pH, EC, nitrate-N, and ammonia-N levels, and to develop a rapid salinity screening procedure.

Investigation of Salinity in Digester Draw and Centrifuge Cake Biosolids From the Stickney, Egan, and Calumet WRPs

Salinity levels and salt chemistry were studied in samples of digester draw biosolids from February 1998 through May 1999, and in samples of centrifuge cake biosolids from February 1998 through March 1999 for the District's Stickney, Calumet, and Egan WRPs. No digester draw biosolids samples were analyzed for the Calumet WRP in March and May 1999.

DIGESTER DRAW BIOSOLIDS

A weekly composite digester draw biosolids sample was obtained once per month from the Stickney, Calumet, and Egan WRPs. The EC of the digester draw biosolids from each of the three WRPs showed a similar seasonal variation pattern (Figure 1).

The digester draw biosolids from the Egan WRP tended to have the highest EC while those from the Calumet WRP tended to have the lowest. The digester draw biosolids from the Egan WRP had maximum ECs in June 1998, 12.95 dS/m, and April 1999, 11.85 dS/m, and reached a minimum in November 1998, 7.14 dS/m, during the 16-month study period.

The Calumet WRP digester draw biosolids had maximum ECs in May 1998, 8.24 dS/m, and April 1999, 8.64 dS/m, and reached a minimum in October 1998, 5.39 dS/m, during the 16-month study period.

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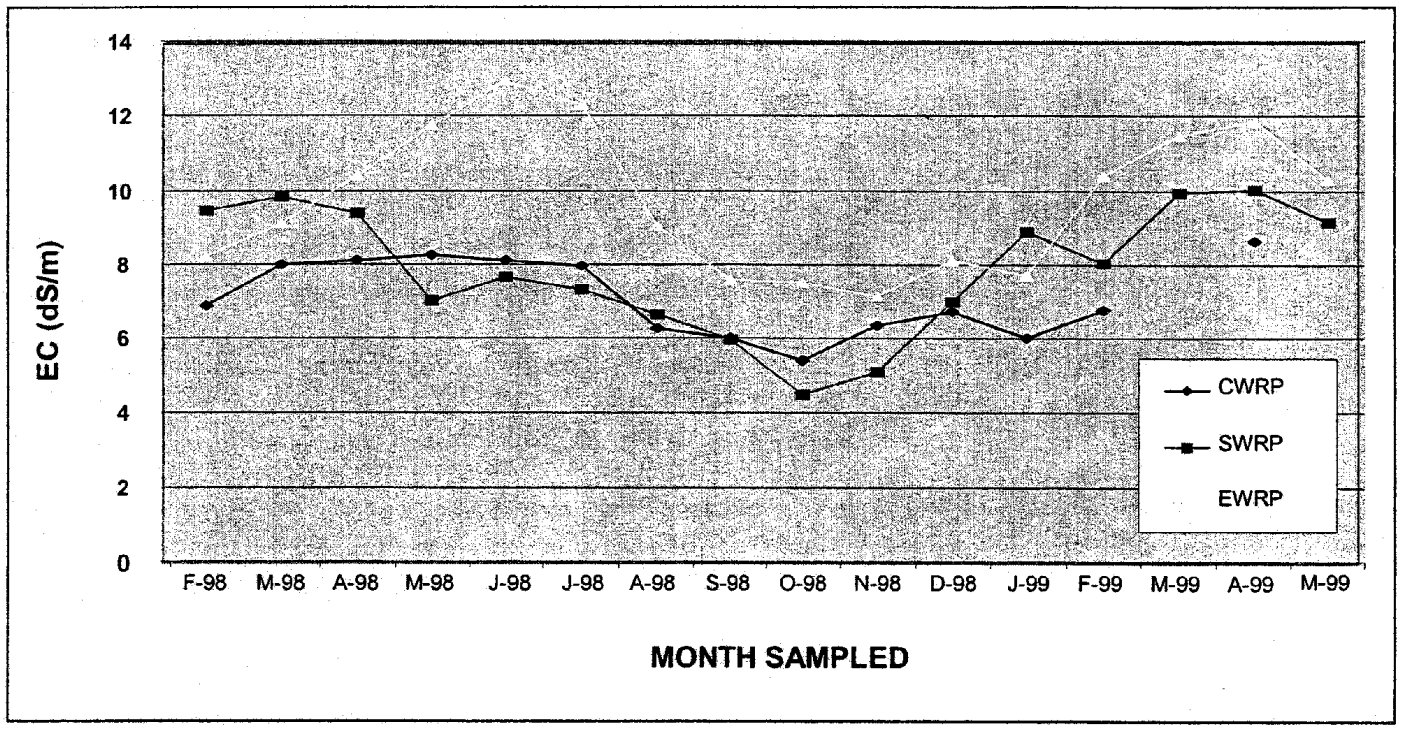
FIGURE 1

ELECTRICAL CONDUCTIVITY OF DIGESTER DRAW BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS



The digester draw biosolids from the Stickney WRP had maximum ECs in March 1998, 9.82 dS/m, and April 1999, 10.01 dS/m, and reached a minimum in October 1998, 4.50 dS/m, during the 16-month study period.

One obvious source of seasonal salinity is road deicing salt, which is composed primarily of sodium chloride (NaCl). We examined the chemistry of the soluble salts in the digester draw biosolids from the three WRPs. Ammonia-N, whose cationic form is NH_4^+, was by far the predominant cationic salt constituent (Figure 2), and bicarbonate, HCO_3^-, was by far the predominant anionic salt constituent (Figure 3). This is to be expected as both of these ions are produced during the anaerobic digestion process. While seasonal trends in the concentrations of sodium and chloride were observed in Figures 2 and 3, the concentrations of Na and Cl indicate that they are only minor constituents of the total soluble salt content of the biosolids. Table 2 summarizes the mean EC and the concentrations of soluble salts in the digester draw biosolids from the Stickney, Calumet, and Egan WRPs for the 16-month study.

This study did not examine salinity in wastewater influent at the Stickney, Calumet, and Egan WRPs. However, it is clear that sodium chloride concentration in influent does not have a large impact on salinity of digester draw biosolids

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FIGURE 2

MEAN CONCENTRATIONS OF SOLUBLE CATIONS IN DIGESTER DRAW BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS FROM FEBRUARY 1998 THROUGH MAY 1999

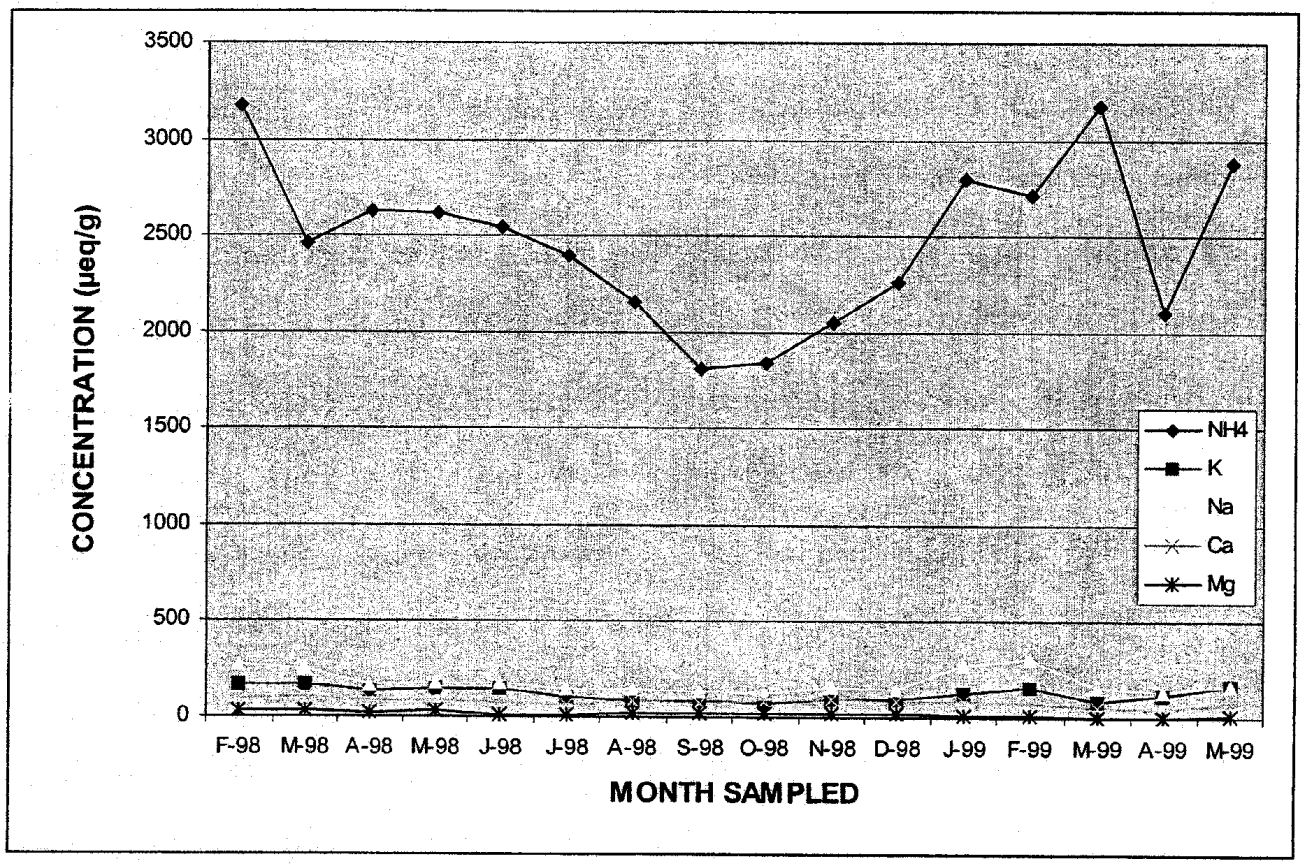

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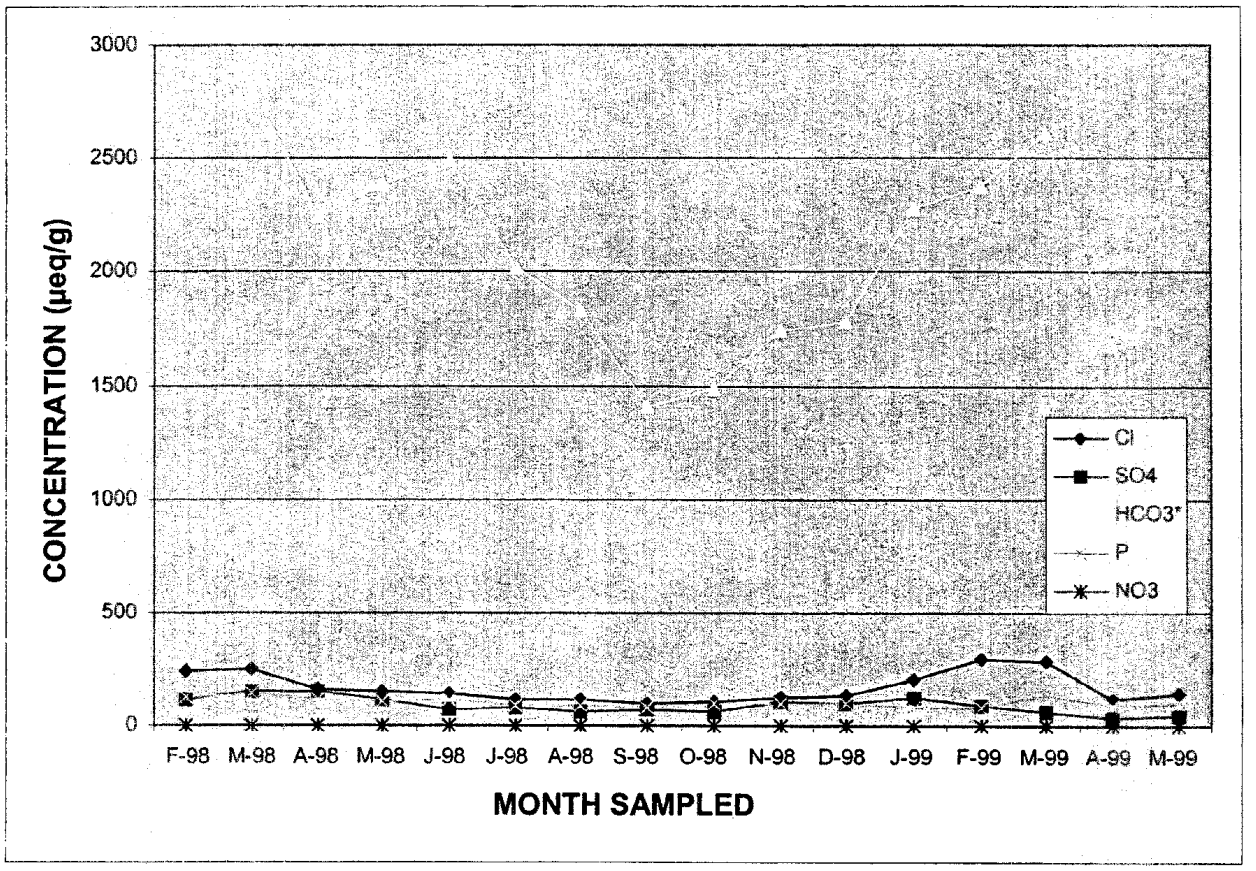
FIGURE 3

MEAN CONCENTRATIONS OF SOLUBLE ANIONS IN DIGESTER DRAW BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS FROM FEBRUARY 1998 THROUGH MAY 1999



*HCO3 is alkalinity expressed as bicarbonate.

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TABLE 2

MEAN EC AND CONCENTRATIONS OF SOLUBLE CATIONS AND ANIONS IN DIGESTER DRAW BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS FROM FEBRUARY 1998 THROUGH MAY 1999

		Mean Concentration		
Analyte*	Units	Stickney	Calumet	Egan
EC	dS/m	7.10	7.87	9.74
Cations	µeq/g	2193	2919	3700
Anions	µeq/g	1889	2553	3246
NH_3-N	µeq/g	1890	2173	3254
K	µeq/g	97	120	148
Na	µeq/g	136	242	183
Ca	µeq/g	55	90	96
Mg	µeq/g	12	21	18
Cl	µeq/g	134	195	169
SO_4	µeq/g	66	108	103
HCO_3	µeq/g	1615	1935	2827
P	µeq/g	71	91	142
NO_3-N	µeq/g	1.2	1.5	1.7

*HCO_3 = Alkalinity expressed as bicarbonate.

because the salinity predominantly consists of ammonia-N and alkalinity, the concentrations of which were found to fluctuate seasonally. Insofar as ammonia-N and alkalinity are products of anaerobic digestion, the seasonal fluctuations in ammonia-N and alkalinity concentrations in digester draw biosolids may be related to seasonal fluctuations that have been observed in the volatile solids content of digester feed at the three WRPs. Thorough evaluation of the connection between digester feed volatile solids content and digester draw salinity (ammonia-N and alkalinity concentration) was beyond the scope of this study.

CENTRIFUGE CAKE BIOSOLIDS

A weekly composite centrifuge cake sample was obtained from the Stickney, Calumet, and Egan WRPs each month during the study period. The EC_e in the centrifuge cake biosolids from the Stickney WRP followed a seasonal pattern similar to that observed for digester draw biosolids from that plant (Figures 1 and 4). This pattern was not observed for centrifuge cake biosolids from the Calumet and Egan WRPs. As with the digester draw biosolids, centrifuge cake biosolids from the Egan WRP tended to have the highest EC_e while those from the Calumet WRP tended to have the lowest (Figure 4).

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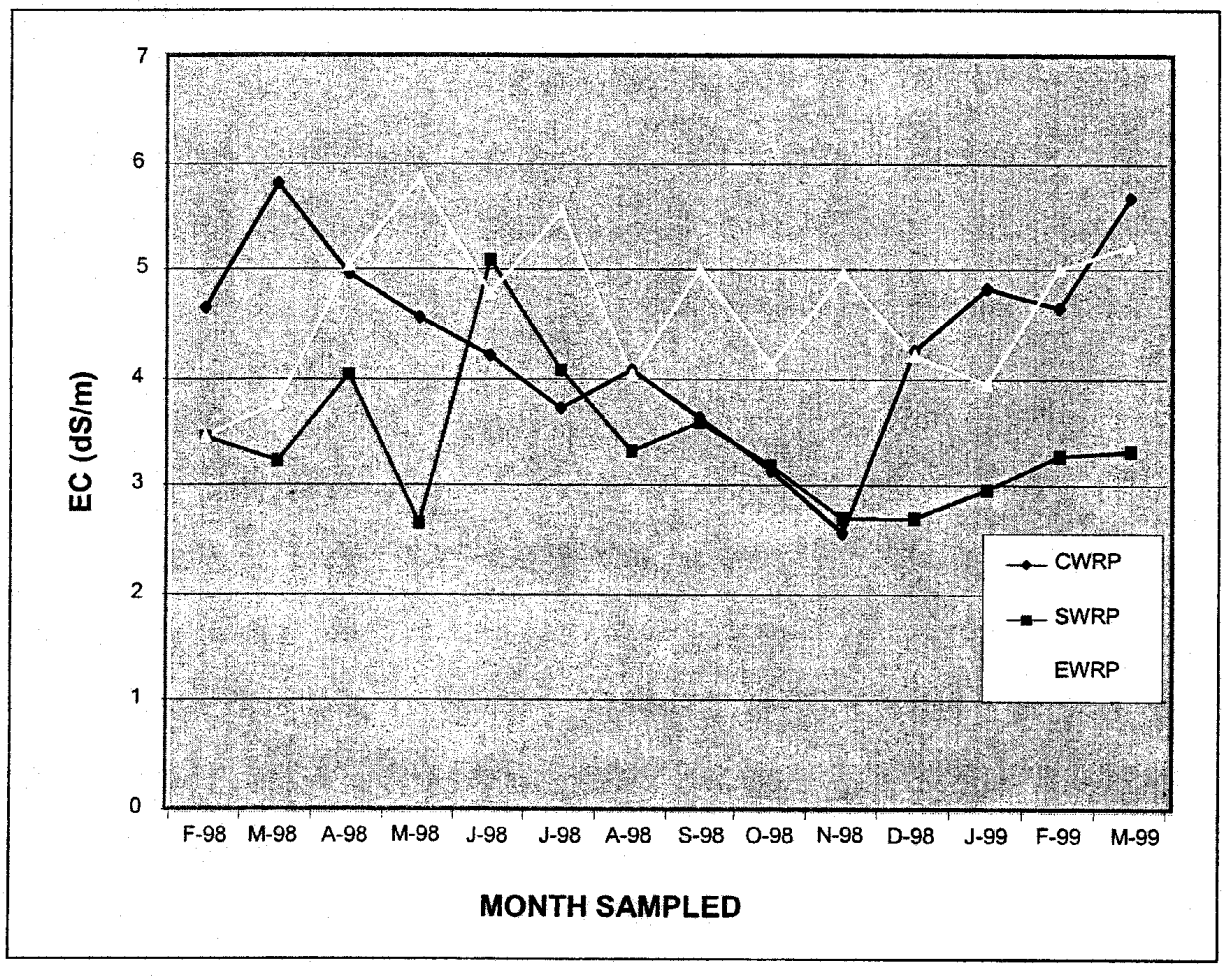
FIGURE 4

ELECTRICAL CONDUCTIVITY OF SATURATION PASTE EXTRACTS OF CENTRIFUGE CAKE BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS



The centrifuge cake biosolids from the Stickney WRP had maximum EC_es in March 1998, 5.80 dS/m, and March 1999, 5.66 dS/m, and reached a minimum in November 1998, 2.56 dS/m, during the 16-month study period.

We examined the chemistry of the soluble salts in the centrifuge cake biosolids from the three WRPs. The composition of the soluble salts was similar to that in digester draw biosolids. Ammonia-N was by far the predominant cationic salt constituent (Figure 5), and bicarbonate was still the predominant anionic salt constituent (Figure 6). Table 3 summarizes the mean EC_e and the concentrations of soluble salts in the saturation paste extracts of centrifuge cake biosolids from the Stickney, Calumet, and Egan WRPs from February 1998 through March 1999.

Investigation of Salinity in Lagoon-Aged and Air-Dried Biosolids from the Stickney and Calumet WRPs

The District's processing train that produces stabilized air-dried biosolids for use as a topsoil substitute in controlled solids distribution projects includes lagoon-aging and air-drying in paved cells. This investigation included the sampling of two Stickney and three Calumet WRP biosolids that were placed onto paved drying cells at the Calumet and LASMA solids management areas. The Egan WRP does not produce a

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FIGURE 5

MEAN SOLUBLE CATION CONCENTRATIONS IN SATURATION PASTE EXTRACTS OF CENTRIFUGE CAKE BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS

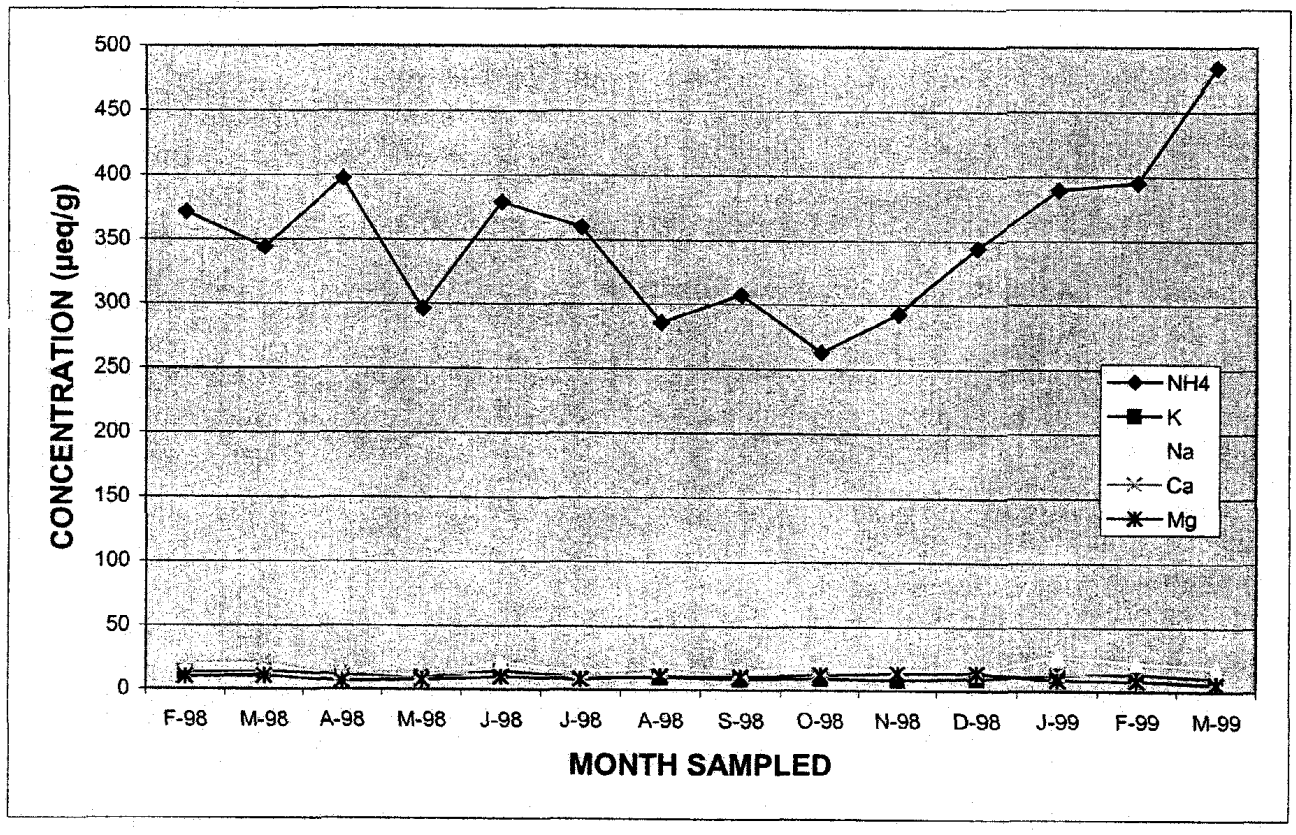

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FIGURE 6

MEAN SOLUBLE ANION CONCENTRATIONS IN SATURATION PASTE EXTRACTS OF CENTRIFUGE CAKE BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS

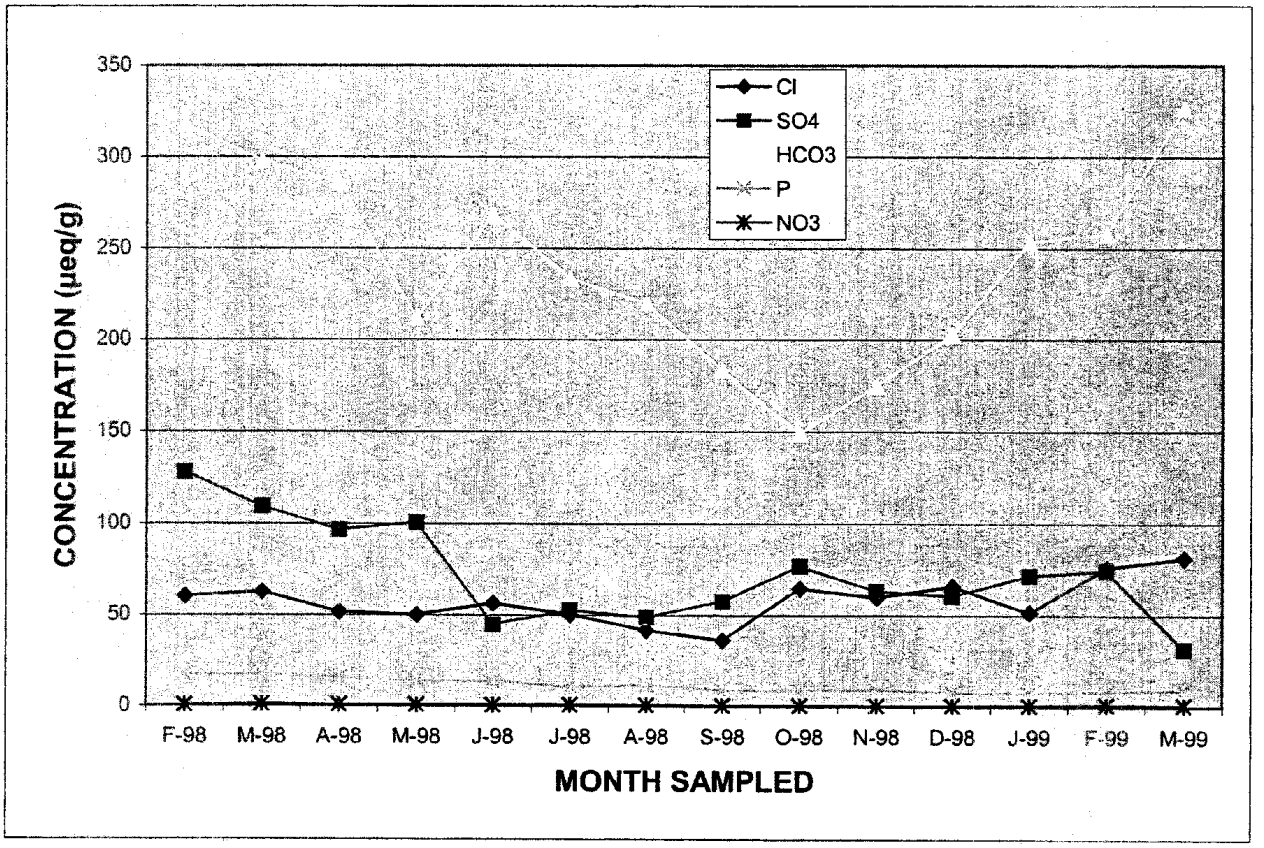

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TABLE 3

MEAN EC AND CONCENTRATIONS OF SOLUBLE CATIONS AND ANIONS IN SATURATION PASTE EXTRACTS OF CENTRIFUGE CAKE BIOSOLIDS FROM THE STICKNEY, CALUMET, AND EGAN WRPS FROM FEBRUARY 1998 THROUGH MARCH 1999

		Mean Concentration		
Analyte*	Units	Stickney	Calumet	Egan
EC	dS/m	4.33	3.41	4.64
Cations	µeq/g	390	349	482
Anions	µeq/g	367	394	419
NH_3-N	µeq/g	343	280	428
K	µeq/g	11	11	11
Na	µeq/g	17	21	15
Ca	µeq/g	9	19	13
Mg	µeq/g	8	12	9
Cl	µeq/g	29	63	81
SO_4	µeq/g	81	77	59
HCO_3	µeq/g	241	216	268
P	µeq/g	14	11	9
NO_3-N	µeq/g	0.6	0.6	0.6

*HCO_3 = alkalinity expressed as bicarbonate

lagoon-aged, air-dried biosolids product and was not included in this portion of the study. The biosolids placed on LASMA cells 2E and 4W (LASMA 2E and LASMA 4W) and the biosolids placed on Calumet West cells 1 and 4 (CAL 1W and CAL 4W) were generated by the low solids processing train (anaerobically digested biosolids placed directly into lagoons for dewatering and aging without prior centrifuging). The biosolids placed on Calumet East cell 3 (CAL 3E) were generated from the high solids processing train (anaerobically digested, centrifuged biosolids placed into lagoons for aging). The biosolids were sampled upon initial loading to the cells, which represented biosolids at the end of lagoon aging, and at the end of the drying cycle just prior to the biosolids being stacked for final disposition, which represents the District's final product.

BIOSOLIDS INITIALLY PLACED ON DRYING CELLS

The EC_e of the lagoon-aged biosolids at the beginning of air-drying (initial placement on the drying cells) were roughly within the range of 5.0 to 8.0 dS/m (Figure 7). The biosolids from LASMA 4W, LASMA 2E, CAL 4W, and CAL 1W all originated from the District's low solids processing train (anaerobic digestion and lagoon aging with no centrifuge

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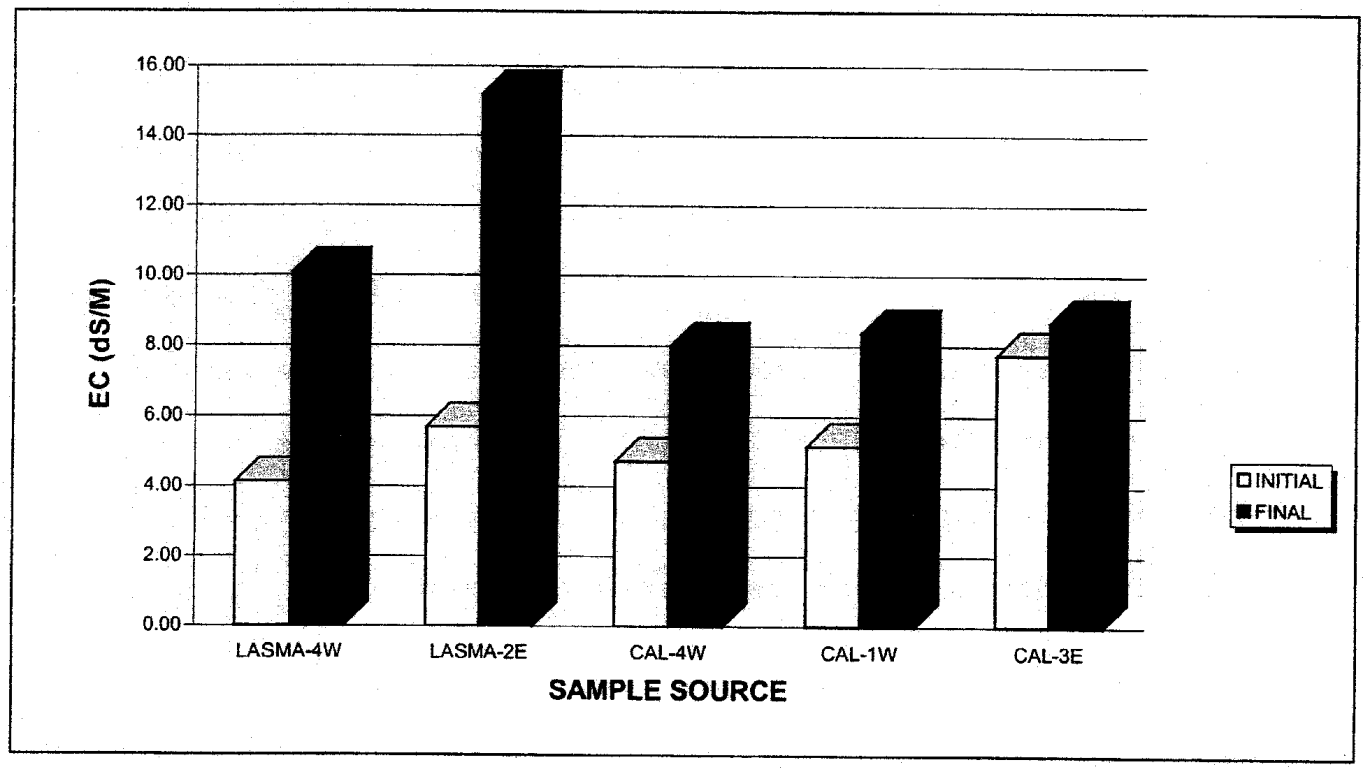
FIGURE 7

ELECTRICAL CONDUCTIVITY OF SATURATION PASTE EXTRACTS OF LAGOON-AGED STICKNEY AND CALUMET WRPs BIOSOLIDS AT THE BEGINNING (INITIAL) AND END (FINAL) OF AIR-DRYING ON PAVED CELLS



dewatering), and their initial EC_e ranged from approximately 5 to 6.5 dS/m. The initial EC_e of the biosolids from CAL 3E, originating from the high solids processing train (centrifuged and lagoon aged), was approximately 2.5 dS/m higher than the EC_e produced by the biosolids originating from the low solids processing train. It is not possible to determine whether this difference in EC_e for biosolids generated from the high solids and low solids processing trains is consistent or significant.

We examined the chemistry of the soluble salts in the lagoon-aged biosolids from the Stickney and Calumet WRPs at the time that they were initially placed on the drying cells. The composition of the soluble salts at this stage was similar to that in digester draw and centrifuge cake biosolids. Ammonia-N was the predominant cationic salt constituent (Figure 8). The biosolids from CAL 3E, generated from the high solids processing train, had the lowest soluble ammonia-N concentration, and it had much higher concentrations of soluble Ca and Mg than any of the other biosolids which were all generated by the low solids processing train. Bicarbonate was still the predominant soluble anionic salt constituent (Figure 9). However, for the biosolids originating from the

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FIGURE 8

MEAN SOLUBLE CATION CONCENTRATIONS IN SATURATION PASTE EXTRACTS OF LAGOON-AGED BIOSOLIDS FROM THE STICKNEY AND CALUMET WRPS AT INITIAL PLACEMENT ON DRYING CELLS

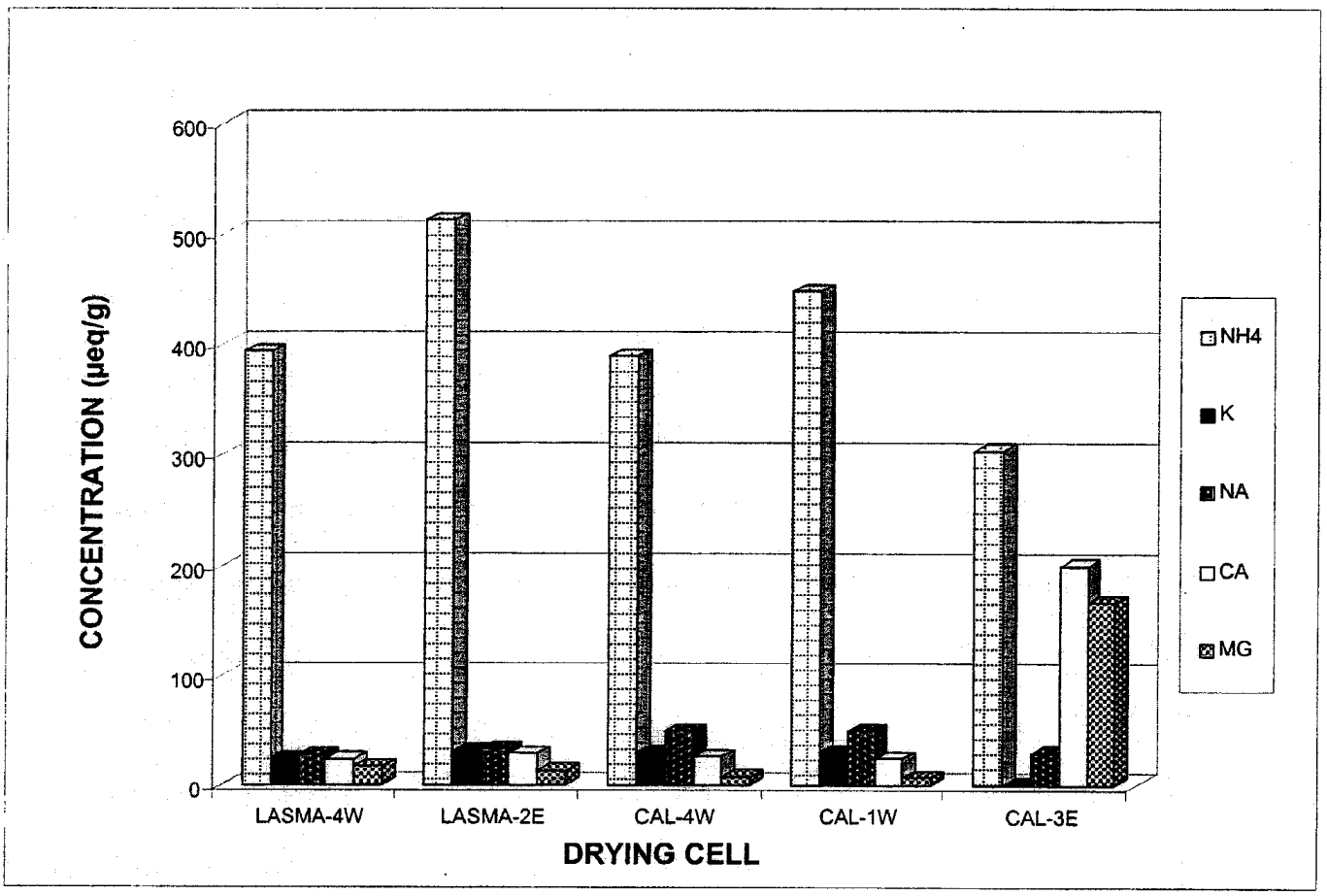

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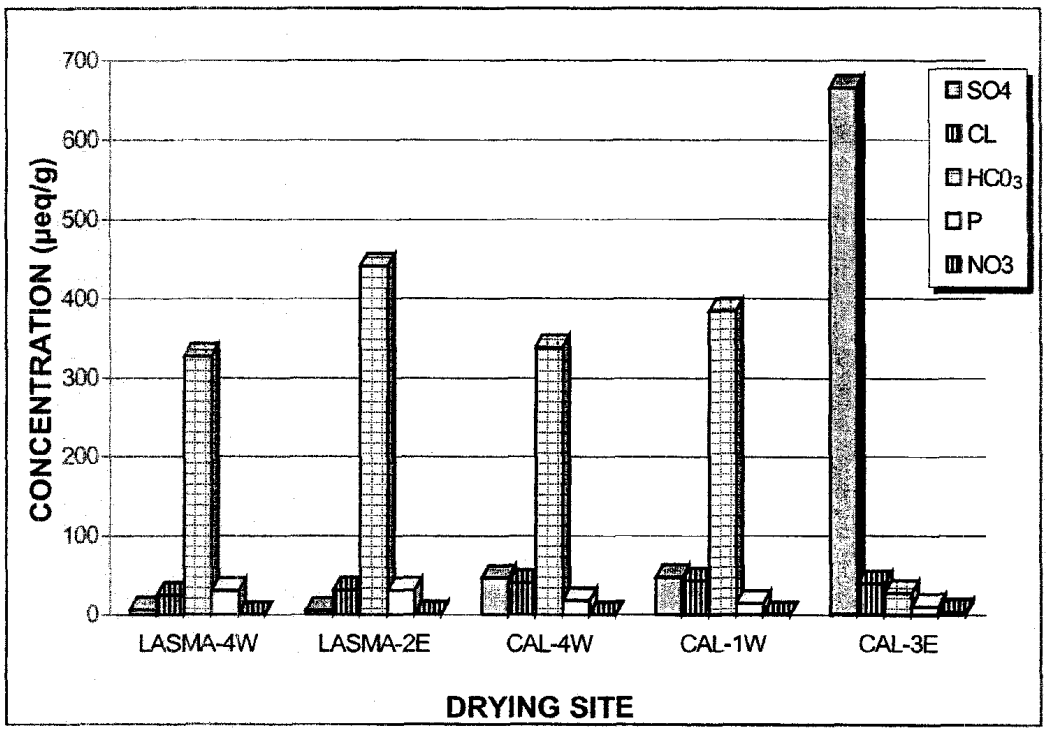
FIGURE 9

MEAN SOLUBLE ANION CONCENTRATIONS IN SATURATION PASTE EXTRACTS OF LAGOON-AGED BIOSOLIDS FROM THE STICKNEY AND CALUMET WRPS AT INITIAL PLACEMENT ON DRYING CELLS



high solids processing train, CAL 3E, bicarbonate was replaced by sulfate as the predominant soluble anionic species. Due to the small number of biosolids samples taken from both the low and high solids processing trains, no reliable inferences can be drawn as to differences in biosolids salinity that may result from aging digester draw and centrifuge cake in lagoons from comparisons of these data. However, in the present study it is clear that the salinity in the sample originating from the high solids processing train, Cal 3E, is different from that in the samples originating from the low solids processing train, LASMA 4W, LASMA 2E, Cal 1W, and Cal 4W.

Table 4 summarizes the overall mean EC_e, and the concentrations of soluble salts in the saturation paste extracts of lagoon-aged biosolids from the Stickney and Calumet WRPs at the time of initial placement on the drying cells. Since we could not verify whether the substantial difference in salt chemistry between Cal 3E and the other samples was due to differences in their processing history or due to sample handling, processing, or analysis, we excluded it from the table.

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TABLE 4

MEAN EC_e, pH, AND CONCENTRATIONS OF SOLIDS, SOLUBLE CATIONS, AND SOLUBLE ANIONS IN LAGOON-AGED BIOSOLIDS* FROM THE STICKNEY AND CALUMET WRPS AT THE INITIATION (INITIAL) AND COMPLETION (FINAL) OF AIR-DRYING

ANALYTE	UNITS	DRYING CELL INITIAL	DRYING CELL FINAL
Solids	%	11.2	71.2
H_2O:Solids**		9.0	1.94
pH		8.1	7.0
EC	dS/m	4.9	10.4
Cations	µeq/g	549	239
Anions	µeq/g	457	225
NH_3-N	µeq/g	436	104
K	µeq/g	29	9
Na	µeq/g	40	14
Ca	µeq/g	26	48
Mg	µeq/g	11	64
Cl	µeq/g	35	9
SO_4	µeq/g	26	172
HCO_3***	µeq/g	372	21
P	µeq/g	23	2
NO_3-N	µeq/g	0.4	21

* Biosolids were anaerobically digested and lagoon-aged originating from the low solids processing train and sampled from LASMA 4W and 2E and Cal 1W and 4W.

** H_2O:Solids is the solution to solids ratio of the saturation paste extract.

*** HCO_3 is alkalinity expressed as bicarbonate.

BIOSOLIDS AFTER COMPLETION OF DRYING

The EC_e of lagoon-aged biosolids at the end of air-drying were within the range 8.0 to 16.0 dS/m (Figure 7). The EC_e of the biosolids from LASMA 2E was approximately 6 dS/m higher than the EC_e produced by the other biosolids sampled. This high EC_e does not appear to be related to biosolids processing history since the other biosolids resulting from the low solids processing train had a much lower EC_e (Figure 7).

We examined the chemistry of the soluble salts in the lagoon-aged biosolids from the Stickney and Calumet WRPs at the completion of drying. The composition of the soluble salts had changed in comparison with the biosolids that were initially placed on the drying cells. Ammonia-N was still a major soluble cationic salt constituent, but its concentration was variable ranging from 29 μeq/g in biosolids from CAL 4W to 196 μeq/g in biosolids from LASMA 2E (Figure 10). The low soluble ammonia-N content of biosolids from CAL 4W and CAL 1W was due, at least in part, to the onset of nitrification in these biosolids. These biosolids also had the highest solids contents (78.5 and 74.6 percent for biosolids from CAL 4W and CAL 1W, respectively) and a greater volatilization of ammonia-N may also have occurred during the drying process. Calcium and magnesium were also major soluble cations in the air-dried

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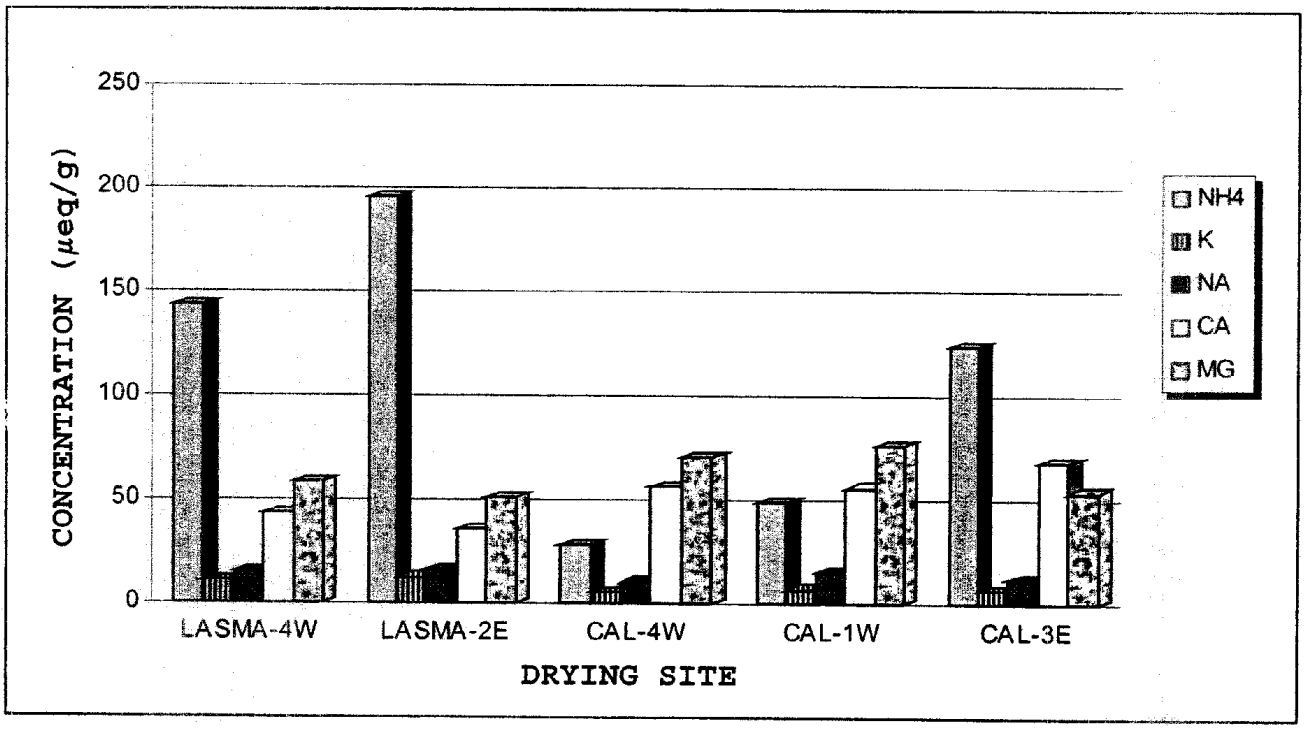
FIGURE 10

MEAN SOLUBLE CATION CONCENTRATIONS IN SATURATION PASTE EXTRACTS OF LAGOON-AGED BIOSOLIDS FROM THE STICKNEY AND CALUMET WRPS FOLLOWING COMPLETION OF DRYING



biosolids, but their concentrations were very similar in all of the biosolids sampled, ranging between 35 and 76 μeq/g (Figure 10).

During the air-drying process, bicarbonate was replaced by sulfate as the predominant soluble anionic salt constituent (Figure 11). This was observed to occur within the first three weeks after the beginning of the drying process (data not shown). Nitrification had occurred in the biosolids from CAL 4W and CAL 1W (Figure 11), which explains in part why they had the lowest soluble ammonia-N concentrations. Some of the soluble nitrate-N that formed may have been lost to leaching during rain events that may have occurred late in the drying cycle. Table 4 summarizes the overall mean EC_e and the concentrations of soluble salts in the saturation paste extracts of lagoon-aged biosolids from the low solids processing trains at the Stickney and Calumet WRPs at the completion of air-drying.

Changes in EC_e and Total Salinity through the Biosolids Processing Trains

The results of observations of salinity in biosolids at various stages of the processing trains, including at the completion of anaerobic digestion (digester draw), after centrifuging (centrifuge cake), after lagoon-aging (initial

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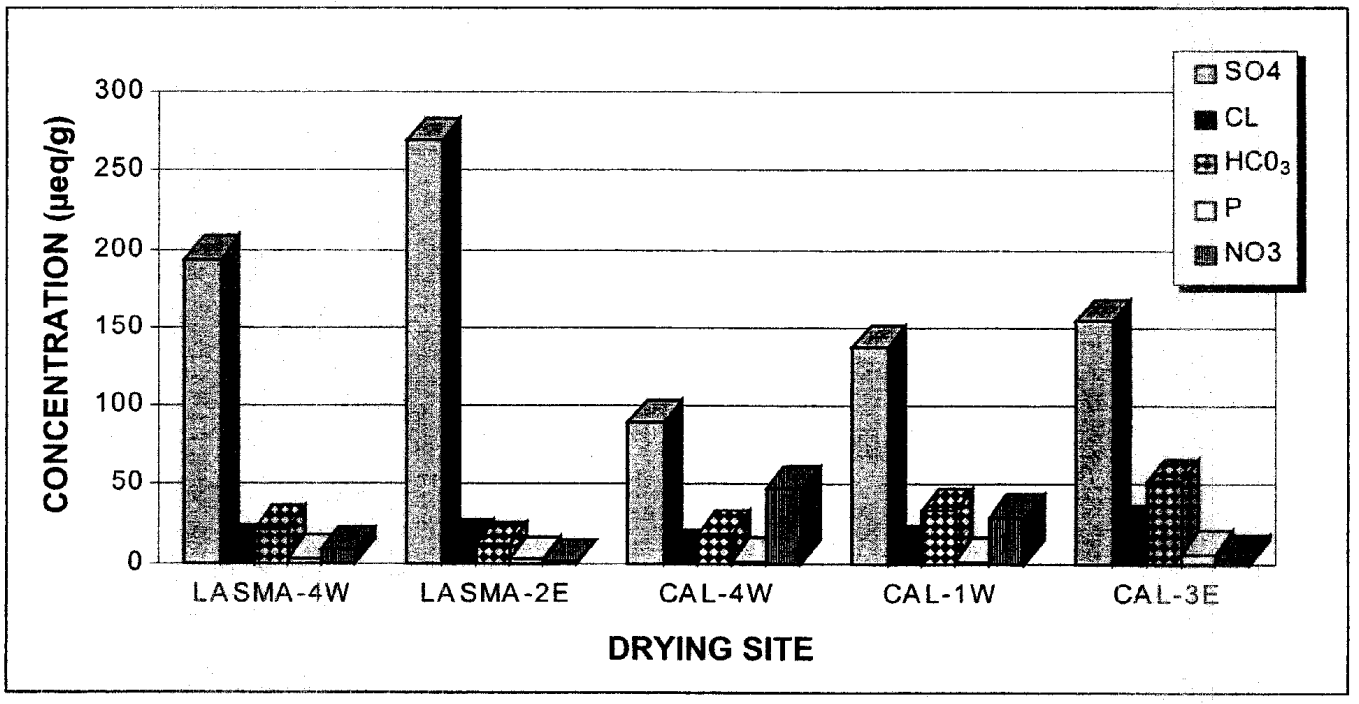
FIGURE 11

MEAN SOLUBLE ANION CONCENTRATIONS IN SATURATION PASTE EXTRACTS OF LAGOON-AGED BIOSOLIDS FROM THE STICKNEY AND CALUMET WRPS FOLLOWING COMPLETION OF DRYING



drying), and after completion of air-drying (final drying) have been determined. The mean EC of the Stickney and Calumet WRP digester draw biosolids and the saturation paste extracts of Stickney and Calumet WRP biosolids at the centrifuge cake, initial drying, and final drying stages of the processing train are summarized in Table 5.

The mean EC_e in biosolids from the Stickney and Calumet WRPs was 7.5 dS/m following anaerobic digestion. Anaerobically digested biosolids then enter either the low solids processing train or the high solids processing train. Biosolids entering the high solids processing train show a reduction in EC_e due to centrifuging, with the mean EC_e falling from 7.5 to 3.9 dS/m after centrifuging. Alternatively, biosolids that enter the low solids processing train are not centrifuged, but are placed directly into lagoons for dewatering and aging. Biosolids entering the low solids processing train show a reduction in EC_e due to lagoon aging, with the mean EC_e falling from 7.5 to 4.9 dS/m after lagoon aging.

We could not ascertain the effect of lagoon aging on biosolids salinity in the high solids processing train in this study because we were unable to collect enough samples of aged centrifuge cake at initiation of drying. The air-drying step

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TABLE 5

CHANGES IN STICKNEY AND CALUMET WRP BIOSOLIDS TOTAL SOLIDS CONTENT AND SATURATION PASTE EXTRACT SOLUTION:SOLIDS RATIO, EC, AND TOTAL SOLUBLE CATIONIC AND ANIONIC SALT CONSTITUENT CONCENTRATIONS AT VARIOUS STAGES OF THE DISTRICT'S BIOSOLIDS PROCESSING TRAINS

Stage of Processing	Solids Content	H20:Solids Ratio	Electrical Conductivity	Total Anions	Total Cations
	--%--		--- dS/m---	--- μeq/g ---	
Digester Draw	3.1	33.9	7.5	2221	2556
Centrifuge Cake	23.2	9.5	3.9	381	370
Initial Drying*					
Low Solids	11.2	9.0	4.9	457	549
High Solids	26.2	10.0	7.8	745	712
Final Drying**	71.2	1.9	10.4	225	239

* Initial drying was sampled immediately following loading onto paved drying cells. Low solids are digester draw biosolids that were lagoon-aged and loaded into Calumet 1W and 4W, and LASMA 2E and 4W for drying. High solids are centrifuge cake biosolids that were lagoon-aged and loaded into Calumet 3E.

** Includes biosolids from the low solids processing train only.

of the processing trains increased the mean EC_e in biosolids from the low solids processing train to 10.4 dS/m (Table 5). There are actually two factors that are influenced by the processing train which are responsible for these changes in EC_e. One factor is the effect of processing on the total soluble salt content of the biosolids, and the other factor is the effect of processing on the solution:solids ratio of the saturation paste extract.

Centrifuging reduced the EC_e from 7.5 to 3.9 dS/m, indicating that the biosolids had less soluble salts. The drop in EC_e due to centrifuging is a result of high amounts of soluble salts being removed with the centrate. In fact, the reduction in salt content was much greater than is reflected in the EC_e. The total anionic salt content was reduced from 2,221 to 381 μeq/g, and the total cationic salt content was reduced from 2,556 to 370 μeq/g (Table 5). The reason that the EC_e did not reflect this nearly six-fold decrease in soluble salt content is due to the fact that the solution:solids ratio decreased from 33.9 in digester draw to 9.5 in the saturation paste extract of centrifuge cake.

This study was not designed *per se* to determine changes in biosolids salinity during lagoon aging. However, since

four of the five lagoon-aged biosolids that were studied were generated in the District's low solids processing train, which does not include centrifuging, the effect of directly lagoon-aging digester draw biosolids can be deduced. The mean EC_e and mean total soluble salt content of lagoon-aged low solids processing train biosolids (designated initial drying in Table 5) are similar, but they are slightly higher than in centrifuge cake biosolids (centrifuged but not aged). This indicates that similar losses of salinity are achieved through centrifuging digester draw biosolids as through lagoon-aging them without centrifuging. Centrifuge cake biosolids had a mean EC_e of 3.9 dS/m, while lagoon-aged low solids processing train biosolids had a mean EC_e of 4.9 dS/m. The mean total cationic and anionic salt contents of the centrifuge cake biosolids were 370 and 381 μeq/g, while they were 549 and 457 μeq/g for lagoon-aged low solids processing train biosolids. However, like centrifuging, lagoon dewatering also produces a biosolids that has a lower saturation paste solution:solids ratio than digester draw biosolids so the magnitude of the reduction in soluble salt produced by this process is not totally reflected in the EC_e.

Air-drying of biosolids also reduces the salt content, but it increases the salinity perceived by roots of plants

that will be grown in the biosolids. The mean total cationic and anionic soluble salt concentrations were reduced from 549 and 457 μeq/g in low solids processing train biosolids initially placed on the drying cells to 239 and 225 μeq/g in final air-dried biosolids (Table 5). This is approximately a 50 percent reduction in total soluble salt content due to air-drying that is offset by a large decrease in the biosolids saturation paste solution:solids ratio that occurs during drying. The biosolids initially placed on the drying cells had a saturation paste solution:solids ratio of 9.0, and after air-drying the solution:solids ratio decreased to 1.9 (Table 5). As a result, the mean EC_e, which approximates the EC of soil solution encountered by roots growing in biosolids, increased from 4.9 dS/m in low solids processing train biosolids initially placed on the drying cells to 10.4 dS/m for biosolids after the completion of drying (Table 5).

Changes in Soluble Salt Composition through the Biosolids Processing Trains

The soluble anionic salt composition changes throughout the biosolids processing train. The mean soluble anionic salt composition of digester draw biosolids consists largely of bi-carbonate, which constitutes 83.9 percent of the total (Table 6). As would be expected of a product of anaerobic

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TABLE 6

CHANGES IN BIOSOLIDS SOLUBLE ANIONIC SALT CONSTITUENT CONCENTRATIONS AND pH AT VARIOUS STAGES OF THE DISTRICT'S BIOSOLIDS PROCESSING TRAIN

Stage of Processing	pH	Saturation Paste Extract Concentration					
		Total Anions	SO_4	HCO_3*	Cl	P	NO_3-N
		µeq/g	------- % Total Anions --------				
Digester Draw	7.9	2221	4.1	83.9	7.9	3.9	0.1
Centrifuge Cake	7.7	381	20.8	60.0	12.1	3.3	0.2
Initial Drying**	8.1	457	5.7	81.5	7.6	5.1	0.0
Final Drying**	7.0	225	171.9	21.0	9.2	1.7	21.0

* HCO_3 is alkalinity expressed as bicarbonate (HCO_3^-)

** Only biosolids from the low solids processing train were included (Cal 1W, Cal 2E, LASMA 2E, and LASMA 4W).

digestion, the mean sulfate and nitrate concentrations are very low in the digester draw biosolids, comprising 4.1 and 0.1 percent of the total anionic salt concentration, respectively (Table 6).

As biosolids move through the processing train, sulfate replaces bicarbonate as the predominant anionic soluble salt species (Table 6). Mean sulfate concentration in digester draw biosolids from Stickney and Calumet WRPs was 97 μeq/g, which constitutes 4.1 percent of the total soluble anions in digester draw biosolids. Lagoon dewatering and aging of anaerobically digested biosolids, without prior centrifuging, did not result in a replacement of bicarbonate with sulfate as the predominant anion. The mean sulfate concentration in lagoon-aged low solids processing train biosolids was 26 μeq/g, which constituted only 5.7 percent of the total anionic salt composition. However, centrifuging and air-drying resulted in greater changes in anionic salt composition. The mean sulfate concentration in centrifuge cake biosolids from the Stickney and Calumet WRPs was 79 μeq/g, which was 20.8 percent of the total soluble anionic salt content in centrifuge cake biosolids, while the mean sulfate concentration in air-dried biosolids was found to be 172 μeq/g, which was 76.5 percent of the total anionic salt content of these biosolids.

At the same time, bicarbonate concentration decreased from 83.9 percent of the total anionic salt concentration in digester draw biosolids to 9.3 percent of the total anionic salt concentration in air-dried biosolids (Table 6). This was largely due to nitrification during air-drying, which consumes alkalinity.

The air-drying of the biosolids also initiates the nitrification of ammonium. The soluble nitrate concentration remains very low through the processing train until significant air-drying occurs. The soluble nitrate-N concentration increased from 0.08 percent of the total soluble anionic salt concentration in lagoon aged biosolids at the onset of drying to 9.3 percent due to air-drying. The soluble nitrate concentration will increase to greater than this in the weeks following air-drying as the aerobic environment of the air-dried biosolids leads to nearly total nitrification of ammonium.

These processes, nitrification and oxidation of sulfide, produce acidity. The half reaction for nitrification is:

$$NH_4^+ + 2O_2 \rightarrow NO_3^- + 2H^+ + H_2O$$

The half reaction for sulfide oxidation is:

$$S^{-2} + 4H_2O \rightarrow SO_4^{-2} + 8H^+ + 8e^-$$

As the biosolids become aerobic, the acidity generated by these reactions leads to a reduction in alkalinity (which is expressed as bicarbonate) and a lowering of biosolids pH. Mean biosolids pH decreases from 7.9 to 7.0 through the processing train with the decrease almost entirely occurring due to air-drying (Table 6). After biosolids become aerobic following completion of air-drying, their pH can drop another pH unit to around 6.0 as nitrification accelerates and sulfide oxidation continues.

The relative mean concentrations of chloride and phosphate are decreased through the processing train. The mean concentrations of chloride and phosphate are 7.9 and 3.9 percent of the total anionic salt concentration in digester draw biosolids, and they decrease to 4.1 and 0.7 percent of the total anionic salt concentration in air-dried biosolids from the low solids processing train (Table 6).

The soluble cationic salt composition also changes throughout the biosolids processing train. The mean ammonium (NH_3-N) concentration in digester draw biosolids was 2,147 μeq/g, which represents 84.0 percent of the total soluble cationic salt concentration (Table 7). The mean soluble calcium and magnesium concentrations were very low in digester

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TABLE 7

CHANGES IN BIOSOLIDS SOLUBLE CATIONIC SALT CONSTITUENT CONCENTRATIONS AT VARIOUS STAGES OF THE DISTRICT'S BIOSOLIDS PROCESSING TRAINS

Stage of Processing	Saturation Paste Extract Concentration						
	pH	Total Cations	NH_3-N	K	Na	Ca	Mg
		µeq/g	----------	%	----------		
Digester Draw	7.9	2556	84.0	4.5	7.8	3.0	0.7
Centrifuge Cake	7.7	370	84.3	3.0	5.1	3.8	2.7
Initial Drying*	8.1	549	79.4	5.2	7.3	4.7	2.0
Final Drying*	7.0	239	43.5	3.7	5.9	20.0	26.7

* Only includes biosolids from the low solids processing train (Cal 1W, Cal 4W, LASMA 2E, and LASMA 4W).

draw biosolids, only 76.7 and 17.9 μeq/g, which represents 3.0 and 0.7 percent of the total cationic salt concentration. This would be expected at this phase of the processing train, which is characterized by high pH and alkalinity (Table 7).

As biosolids move through the processing train, calcium and magnesium replace ammonium as the predominant cationic soluble salt species (Table 7). Ammonium concentrations were 2,147 μeq/g, which constituted 84.0 percent of the total soluble cations in digester draw biosolids, 312 μeq/g, which constituted 84.3 percent of the total soluble cations in centrifuge cake biosolids, 436 μeq/g, which constituted 79.4 percent of the total soluble cations in lagoon-aged biosolids from the low solids processing train (designated initial drying in Table 7), and 104 μeq/g, which constituted 43.5 percent of the total cations in air-dried biosolids. This is due to the onset of nitrification, and to a lesser extent volatilization of ammonia.

At the same time, soluble calcium and magnesium concentrations increased from 76.7 and 17.9 μeq/g, which constituted 3.0 and 0.7 percent of the total soluble cationic salt concentration in digester draw biosolids, respectively, to 47.9 and 63.8 μeq/g, which constituted 20.0 and 26.7 percent of the total soluble cationic salt concentration,

respectively, in air-dried biosolids (Table 7). This is largely due to the solublization of Ca and Mg as nitrification and sulfide oxidation acidify the biosolids and reduce the alkalinity, especially during air-drying. Following completion of air-drying, the soluble ammonium concentration continues to decline due to nitrification, and as the biosolids pH continues to drop, as discussed above, greater amounts of calcium and magnesium solublize.

The relative mean concentrations of soluble sodium and potassium were nearly constant in the biosolids throughout the processing train. The mean concentrations of soluble sodium and potassium in digester draw biosolids were 199 and 115 μeq/g, which constituted 7.8 and 4.5 percent of the total soluble cationic salt concentration, respectively, and in air-dried biosolids their mean concentrations were 14.2 and 8.9 μeq/g, which constituted 5.9 and 3.7 percent of the total soluble cationic salt concentration, respectively (Table 7).

Potential for Ammonia Toxicity to Impact the Use of Biosolids as a Topsoil Substitute

Ammonium has been demonstrated to be the predominant soluble cationic salt species in District biosolids throughout the processing train. While the biosolids processing train reduces the amount of soluble ammonia-N present in the bio-

solids from 2,147 in digester draw biosolids to 118 μeq/g in air-dried biosolids (Table 7), there still remains enough soluble ammonia-N for the biosolids to sustain a significant concentration in soil solution. Ammonia toxicity to plants can occur via two separate mechanisms. First, excessive plant uptake of ammonia-N in the cationic ammonium form (NH_4^+-N) can result in phytotoxicity. This can occur if the plant is unable to assimilate the ammonium into organic forms quickly enough to prevent its build up in root and shoot tissues. Young plants, which have limited photosynthetic capacity, are particularly sensitive to this mode of ammonium phytotoxicity. Tissue concentrations of approximately 50 mmol/kg ammonia-N have been demonstrated to be phytotoxic to wheat (*Triticum aestivum*), which was grown in solution concentrations of 224 mg/L of ammonia-N (Gill and Reisenauer, 1993).

Second, ammonia-N in the neutral dissolved form, aqueous ammonia (NH_{3aq}-N), is thought to also be toxic to plants by disrupting the structure and function of root cells (Gill and Reisenauer, 1993). Solution concentrations of 2.1 to 2.8 mg/L NH_{3aq}-N were reported to produce phytotoxicity in wheat, cotton (*Gossypium hirsutum* L.) and sudangrass (*Sorghum bicolor* (L.) Moench; syn. *Sorghum sudanensis* L.). Concentrations of NH_{3aq}-N as low as 0.028 mmol/L were demonstrated to be phytotoxic to

tomato (*Lycopersicon esculentum* Mill.) (Gill and Reisenauer, 1993). The concentration of NH_{3aq}-N is dependent on the NH_4^+-N concentration and the pH of the soil solution according to the following equation:

$$[NH_{3aq}\text{-}N] = ([NH_4^+\text{-}N] \times 10^{-9.28}) / [H^+] \qquad \text{Eq. 1}$$

The mean, minimum, and maximum ammonia-N concentrations, expressed on a saturation paste extract basis, for biosolids from the salinity survey that has been conducted for the past four years are summarized in Table 8. Table 8 also summarizes the mean, minimum, and maximum pH for these same biosolids. As has been discussed previously, biosolids with high ammonia-N concentrations will also have a high pH since nitrification, which reduces biosolids ammonia-N concentrations, also acidifies the biosolids lowering their pH. Using Equation 1, the mean ammonia-N and mean pH for biosolids collected in 1998 through 2001 would produce solution concentrations of NH_{3aq}-N of 2.38 mg/L, 1.25 mg/L, 1.57 mg/L, and 2.77 mg/L respectively. These values are slightly higher than or very close to the concentration of 2.1 mg/L determined to be harmful to wheat, cotton, and sudangrass as discussed above. The maximum ammonia-N and pH for biosolids collected in 1998 through 2001

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TABLE 8

MINIMUM, MEAN, AND MAXIMUM AMMONIA-N CONCENTRATIONS AND pH IN SATURATION PASTE EXTRACTS OF BIOSOLIDS FROM THE SALINITY SURVEY CONDUCTED FROM 1998 THROUGH 2001

Year	Ammonia-N Concentration			pH		
	Min	Mean	Max	Min	Mean	Max
	---------mg/L --------					
1998	166	808	1975	5.97	6.75	7.66
1999	4.9	570	1616	5.68	6.62	7.67
2000	3.7	670	2975	5.84	6.65	8.76
2001	0.2	635	2093	5.70	6.92	8.20

would produce solution concentrations of NH_{3aq}-N of 48.5 mg/L, 39.7 mg/L, 72.6 mg/L, and 174 mg/L, respectively.

These values are quite high, and they would be expected to be phytotoxic to many plant species. These results indicate that when used as a soil conditioner or topsoil substitute, some batches of biosolids when applied in high amounts have the potential to produce ammonia phytotoxicity when initially placed on land. This potential problem can be avoided by screening the biosolids prior to distribution, and by requiring the ammonia-N concentration to be less than 400 mg/L (saturation paste basis) and the NH_{3aq}-N concentration to be less than 2.0 mg/L as computed using Equation 1 for biosolids released to sensitive project sites.

It should also be recognized that once the biosolids are dried and placed on land, nitrification occurs. This process rapidly reduces the ammonia-N concentration of the biosolids, and it also, in conjunction with sulfide oxidation, reduces biosolids pH which leads to the dramatic reduction in NH_{3aq}-N. Generally speaking, the risk of ammonia phytotoxicity becomes insignificant two to four weeks after the placement of biosolids on land for most biosolids, except for those with the highest ammonia-N concentrations and pH. Prescreening of biosolids prior to distribution, as described above, can immediately

reduce the risk of ammonia phytotoxicity from biosolids use to inconsequential levels.

Potential for Boron Toxicity to Impact the Use of Biosolids as a Topsoil Substitute

Boron is a plant micronutrient with a very narrow sufficiency range. Boron toxicities to plants are rare in natural soils, except in arid regions and are virtually non-existent in Illinois. Adriano (1986) reports that soluble boron concentrations in Illinois soils have been found to range between 0.2 and 1.22 mg/kg. Soluble boron concentrations in air-dried biosolids sampled at LASMA 4W, LASMA 2E, CAL 4W, CAL 3E, and CAL 1W were found to range from 1.26 to 1.91 mg/kg, which is only slightly higher than the range for Illinois soils reported above.

The concentrations of boron in saturation paste extracts from the biosolids sampled at LASMA 4W, LASMA 2E, CAL 4W, CAL 3E, and CAL 1W were found to range from 0.54 mg/L to 0.82 mg/L. Maas (1986) reported that boron concentrations in soil solution ranging from 0.3 to 1.0 mg/L will only have toxic effects on sensitive and very sensitive species (such as many beans, fruit, citrus, and nut trees), most of which are not routinely grown in Illinois. Mass (1986) also reported that moderately sensitive, moderately tolerant, and tolerant species

may be effected by 1.0 to 2.0 mg/L, 2.0 to 4.0 mg/L, and 6.0 to 8.0 mg/L of boron, respectively. Species such as Kentucky bluegrass (*Poa pratensis*), sweet clover (*Melilotus indica*), oats (*Avena sativa*), and corn (*Zea mays*) can tolerate soil solution boron concentrations of 2.0 to 4.0 mg/L. Maas (1986) reports that alfalfa can tolerate soil solution boron concentrations of up to 6.0 mg/L.

There does not appear to be any significant risk of boron toxicity due to use of biosolids as a topsoil substitute.

Potential for Sodicity to Impact the Use of Biosolids as a Topsoil Substitute

Biosolids contain higher concentrations of sodium than most naturally occurring soils in Illinois. Sodium can occur at high enough concentrations in some soils to impair plant growth. This impairment is usually due to interference with normal calcium and magnesium nutrition. Maas (1986) reports that sodium concentrations above 5 mmol/L, or 115 mg Na/L in irrigation waters can begin to cause effects in very sensitive woody ornamental species. We have observed sodium concentrations in the saturation paste extract of lagoon-aged, air-dried biosolids ranging from 106 to 209 mg/L with a mean sodium concentration of 157 mg/L. This indicates that very

sensitive woody species may be effected by the level of sodium in biosolids used as a topsoil substitute.

However, a standard index of sodium status of soils is the sodium adsorption ratio (SAR). This is computed as:

$$SAR = [Na^{+}]/\{([Ca^{+2}] + [Mg^{+2}])/2\}^{1/2} \qquad \text{Eq. 2}$$

This is an index of the exchangeable sodium concentration in soils relative to the exchangeable calcium and magnesium concentrations. Based on the concentrations of sodium, calcium, and magnesium in the saturation paste extracts of the lagoon-aged, air-dried biosolids, the SAR ranges from 1.3 to 2.5 with the mean SAR being 1.9. Soils are not classified as sodium affected until the SAR becomes greater than 12. It does not appear that soluble sodium concentrations in biosolids will limit their use as a topsoil substitute for supporting growth of all but the most sensitive woody species.

Results of Annual Salinity Surveys

From 1998 through 2001, biosolids were sampled at eight solids management areas immediately prior to final disposition. In 1998 a total of 59 samples were collected, while 41 samples were collected in 1999, 73 samples were collected in 2000, and 79 samples were collected in 2001. All of these

samples were extracted 2:1 (volume water:fresh weight of biosolids) and analyzed for EC, pH, NH_3-N, and NO_3-N. In addition, 8, 12, 12, and 9 samples were selected at random in 1998, 1999, 2000, and 2001, respectively, and they were subjected to saturation paste extraction and analyzed for EC, pH, NH_3-N, and NO_3-N.

The results of the saturation paste EC determinations on the selected samples from the annual surveys are presented in Table 9. Mean biosolids EC determined for 1999, 2000, and 2001 were 7.5, 6.8, and 7.0 dS m^{-1}, respectively (Table 9), and they indicate that biosolids used as a topsoil substitute can potentially limit the growth of all plant species, except those that are salt tolerant (Table 1). The mean biosolids EC determined for 1998, 10.4 dS m^{-1}, as well as the maximum EC determined for 1998, 1999, and 2001, which were 16.4, 10.2, and 15.9 dS m^{-1}, respectively, indicate that biosolids used as a topsoil substitute can potentially limit the growth of even salt tolerant plant species (Table 1). These findings formulated the basis for a further more in-depth study of biosolids salinity.

The first undertaking was to determine if a rapid screening procedure could be developed as a surrogate for the determination of EC utilizing the saturation paste method.

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TABLE 9

MEAN, MINIMUM, AND MAXIMUM ELECTRICAL CONDUCTIVITIES OF SATURATION PASTE EXTRACTS OF AIR-DRIED DISTRICT BIOSOLIDS FROM 1998 THROUGH 2001

Year	Biosolids Saturation Paste Extract Electrical Conductivity		
	Mean	Minimum	Maximum
	---------- dS/m ----------		
1998	10.4	7.9	16.4
1999	7.5	3.0	10.2
2000	6.8	3.0	9.6
2001	7.0	1.8	15.9

The saturation paste method requires a large sample size (approximately 250 g for biosolids), it is very time consuming and labor intensive (technicians have to stand over each individual sample gradually adding water and stirring until the saturation point is achieved), and it is not a precise endpoint with a constant solids to solution ratio.

Development of a Rapid Screening Procedure for Testing Biosolids Salinity

Since the rapid screening method would involve extracting biosolids with a standardized solution:solids ratio, which would not necessarily correspond with the ratio achieved at saturation, a preliminary investigation was conducted to determine the effect of solution:solids ratios on resulting extract EC. Two oven-dried biosolids samples, one originating from the Calumet WRP and one from the Stickney WRP, were extracted in triplicate using solution:solids ratios ranging from 20:1 to 0.8:1. As expected, for a given biosolids sample the extract EC increased as the solution:solids ratio decreased (Table 10). This is due to the soluble salts dissolving into smaller volumes of water when the solution:solids ratio is low, resulting in higher salt concentrations in the extract.

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TABLE 10

EFFECT OF SOLUTION TO SOLIDS RATIO ON BIOSOLIDS EXTRACT ELECTRICAL CONDUCTIVITY

H2O:Solids Ratio	Mean Extract EC*	
	Biosolids Sample 1	Biosolids Sample 2
	dS/m	
20:1	2.0	2.3
10:1	3.5	4.1
7.5:1	4.4	5.0
5:1	5.9	6.8
2.5:1	9.5	11.1
2:1	11.0	12.7
1:1	16.7	18.8
0.8:1	19.3	21.4

*Mean of three replicate analyses.

Because salt ions do not behave ideally in solution, the EC does not increase exactly in proportion with the decrease in solution:solids ratio. As solution:solids ratios decrease, the salt concentration in the extract increases as does the solution ionic strength. As this occurs, ion pairing, ionic atmosphere effects, and electrophoretic effects reduce the molar conductivity of dissolved ions.

However, over small changes in the solution:solids ratio these effects do not vary appreciably, and the EC that is measured at one solution:solids ratio can be converted to another solution:solids ratio basis with only a small error due to non-ideal behavior effects.

We selected a 2:1 (volume water:fresh mass biosolids) extract for our rapid screening procedure. Biosolids were not pre-dried prior to analysis because this adds a significant amount of time to the screening procedure, and it can also lead to volatilization of NH_3-N, a significant soluble salt constituent of biosolids. Instead, the moisture content was determined on a moisture balance using a small subsample of each biosolids sample.

From the saturation paste extracts that were conducted from 1998 through 2001, we determined that biosolids saturation pastes had a mean solution:solids ratio (volume water:dry

mass biosolids) of 2.33, and it ranged from 1.47 to 3.57. This range in solution:solids ratio for the saturation paste is due in part to the random variability and heterogeneity of biosolids samples because they vary in organic matter content and other properties affecting moisture retention.

For all biosolids samples that were extracted by both the saturation paste and 2:1 extract procedures, we corrected the EC obtained on the 2:1 extract to a 2.33:1 (volume water:dry mass biosolids) basis.

This was not a simple computation because the 2:1 extract is conducted using fresh biosolids that are not dried prior to analysis. Therefore, the actual ratio of solution:solids that existed in the 2:1 extract was dependent on the moisture content of the biosolids. The total amount of solution in the extract was computed by adding the amount of water in the fresh biosolids sample to the amount of extracting water that was used [30 mL + (15 g biosolids x biosolids moisture content)]. The amount of solids that were actually extracted was computed by multiplying the total fresh weight of biosolids by the biosolids total solids content (15 x biosolids total solids content). These two quantities were then divided to obtain the actual solution to solids ratio of the 2:1 extracts. The correction was then made by multiplying

the EC obtained by the 2:1 extract by the quotient of the solution to solids ratios of the 2:1 extract and the average for the saturation paste extract (2:33) as shown in the following formula:

$$EC_{corr} = EC_{2:1} \times \{[(30 + (15 \times \%H_2O))/(15 \times \%TS)]/2.33\} \quad \text{Eq. 3}$$

Where $EC_{2:1}$ is the EC determined on the 2:1 extract, EC_{corr} is the $EC_{2:1}$ corrected for biosolids moisture content and to the 2.33:1 saturated paste ratio, $\%H_2O$ is the biosolids moisture content expressed in decimal form, and %TS is the biosolids solids content expressed in decimal form.

We then compared the saturation paste EC with the corrected EC from the 2:1 extract (Figure 12). Regression analysis indicated that:

$$EC_{corr} = 1.05 \times EC_{Satpaste} \quad r^2 = 0.72 \quad \text{Eq. 4}$$

A regression slope of 1.00 with r^2 of 1.00 would indicate a perfect relationship between the corrected 2:1 extract EC (EC_{corr}) and the saturation paste extract EC ($EC_{satpaste}$). The actual regression slope indicates that the correction is very promising with the EC_{corr} overpredicting the $EC_{satpaste}$ by only 5 percent with r^2 of 0.72.

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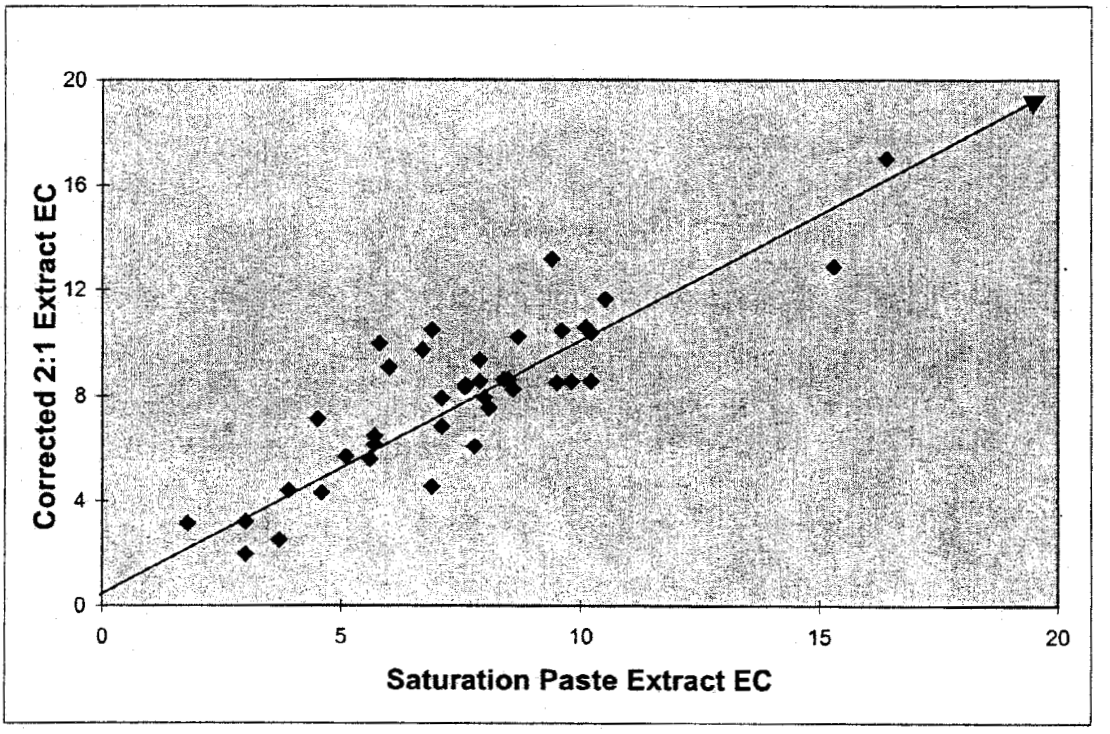
FIGURE 12

COMPARISON OF ELECTRICAL CONDUCTIVITY (dS/m) FROM CORRECTED 2:1 BIOSOLIDS EXTRACTS AND FROM SATURATION PASTE BIOSOLIDS EXTRACTS (ARROW REPRESENTS LINE DEFINED BY EQUIVALENT ECs)



We used Equation 3 to compute EC_{corr} for all of the survey samples taken from 1998 through 2001 that were originally analyzed with the 2:1 extract. The mean EC_{corr} for biosolids originating from the Calumet and Stickney WRPs are summarized in Table 11.

The accuracy of the rapid screening method can be optimized if the extract produces a final solution:solids ratio mimicking that of the saturation paste. Since biosolids are generally dried to solids contents between 60 and 80 percent, we recommend using 18.0 g of fresh biosolids and 24.0 mL of H_2O for the rapid screening extracts. This will produce a solution:solids ratio of 2.33:1 if the biosolids are at 70 percent solids content, the midpoint of the normal range for final product. This will produce just enough solution to determine the pH and EC, and the salt chemistry can be analyzed on small subsamples, less than 1 mL, of the extracts if necessary using ion chromatography.

It is beyond the scope of this report to set specific guidelines for EC requirements of biosolids used to support the growth of all turf, trees, and ornamentals that can be encountered at project sites in the Chicago metropolitan area.

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TABLE 11

MEAN ELECTRICAL CONDUCTIVITY CORRECTED TO A SATURATION PASTE BASIS FOR ALL SAMPLES COLLECTED IN BIOSOLIDS SALINITY SURVEY FROM 1998 THROUGH 2001

	Mean Biosolids Extract Electrical Conductivity	
Year	Calumet WRP	Stickney WRP
	---------- dS/m ----------	
1998	12.2 ± 3.26	14.0 ± 3.02
1999	8.8 ± 2.89	8.9 ± 2.73
2000	7.3 ± 3.46	10.2 ± 4.65
2001	8.6 ± 3.85	9.7 ± 4.36

*Values are means ± standard deviations.

However, insofar as the vast majority of sites are used to grow turf, we are making some recommendations for EC based quality control. For sites where trees or other ornamentals are grown, or where a mono-culture of sensitive turf grass is required (e.g. Kentucky bluegrass), the guidelines are not applicable and the Biosolids Utilization and Soil Science Section should be contacted for site specific recommendations.

As a guideline for interpreting the results of the rapid screening for EC the following should be used:

1. For biosolids used as a topsoil substitute (no mixing or incorporation into soil anticipated), the biosolids EC_{corr} should not be greater than 5.0 dS/m unless arrangements are made to utilize irrigation to establish seedlings and relieve salt stress during drought. Under irrigated management, biosolids EC_{corr} should not be greater than 8.0 dS/m.
2. For biosolids used as a topsoil substitute (mixing or incorporation into soil at a ratio of approximately 50/50 anticipated), the biosolids EC should not be greater than 9.0 dS/m at irrigated sites or 12.0 dS/m for biosolids going to sites that will be managed using irrigation.

LITERATURE CITED

Adriano, D. C., Trace Elements in the Terrestrial Environment, Springer-Verlag Publishers, New York, New York, 1986.

Granato, T. C., R. I. Pietz, O. Dennison, P. Tata, D. R. Zenz, and C. Lue-Hing, An Evaluation of the Suitability of Grass Species and Varieties for Germination and Growth in Biosolids, Research and Development Department Report 98-26, Metropolitan Water Reclamation District of Greater Chicago, 1998.

Granato, T. C., P. Tata, R. I. Pietz, C. Lue-Hing, and R. Lanyon, "Use of Biosolids as a Topsoil Substitute in Urban Markets - The MWRDGC Experience," In: Proceedings of the First Annual Innovative Uses of Biosolids Conference, Rosemont, IL, Water Environment Federation, June 2000.

Rodgers, C. S., and R. C. Anderson, "Plant Growth Inhibition by Soluble Salts in Sewage Sludge-Amended Mine Spoils," Journal of Environmental Quality 24:627-630, 1995.

Bernstein, L., L. E. Francois, and R. A. Clark, "Salt Tolerance of Ornamental Shrubs and Ground Covers," Journal of the American Society for Horticultural Science 97: 550-556, 1972.

Francois, L. E., and R. A. Clark, "Boron Tolerance of Twenty-five Ornamental Shrub Species," Journal of the American Society for Horticultural Science 104: 319-322, 1979.

Francois, L. E., "Salt Tolerance of Eight Ornamental Tree Species," Journal of the American Society for Horticultural Science 107: 66-68, 1982.

Maas, E. V., and G. J. Hoffman, "Crop Salt Tolerance - Current Assessment," Journal of the Irrigation and Drainage Division, ASCE, 103:115-134, 1977.

Mass, E. V., "Salt Tolerance of Plants," Applied Agricultural Research 1: 1-26, 1986.

Rhoades, J. D., "Salinity: Electrical conductivity and total dissolved solids," In Methods of Soil Analysis Part 3-Chemical methods, D. L. Sparks (ed), Soil Science Society of America, Madison, Wisconsin, 1996.

Gill, M. A., and H. M. Reisenauer, "Nature and characterization of ammonium effects on wheat and tomato," Agronomy Journal 85:874-879, 1993.