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Salt Content Determination for Bentonite Mine Spoil: Saturation Extracts Versus 1:5 Extracts

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Abstract—The reliability of estimating salt content in saturated extracts from 1:5 (1spoil:5water) extract levels for bentonite mine spoil was examined by regression analyses. Nine chemical variables were examined that included pH, EC, Ca^{++}, Mg^{++}, Na^{+}, K^{+}, HCO_3^{-}, SO_4^{-} , and Cl^{-}. Ion concentrations from 1:5 extracts were estimated with high predictability for Ca^{++}, Mg^{++}, Na^{+}, $SO_4^{=}$, HCO_3^{-}, and EC conductivity. Overall, calculated ion concentrations from 1:5 extracts show significant relationships with saturation extracts, except for Cl^{-} and K^{+}. The significant relationships we found suggest that 1:5 extracts can be used to reliably estimate salinity of bentonite mine spoils with less effort and expense.

Keywords: bentonite, mine spoil, salt content, salinity, saturation extracts, 1:5 extracts, ion concentrations

Introduction

Bentonite mine spoil is an atypical plant substrate with chemical and physical characteristics that complicate revegetation efforts. Spoil is typically saline-sodic (Bohn and others 1979) with an electrical conductivity (EC) that ranges from 9 to 49 (dS/m), a sodium adsorption ratio (SAR) of 20-56 (Hemmer and others 1977; Bjugstad and others 1981; Uresk and Yamamoto 1986) and a high cation exchange capacity (CEC) (53-59 cmol/kg) (Voorhees and Uresk 1990). High percentages of expansive clays dominate and saturation percentages are high and variable (255-331) (Voorhees and Uresk 1990).

Salinity estimates from 1:5 (1spoil:5water) extracts were used because of difficulties encountered in preparation of saturated pastes for bentonite substrate with high and variable saturation percentages (Uresk and Yamamoto 1986; Voorhees 1986; Voorhees 1990; Voorhees and others 1987; Voorhees and others 1991; Voorhees and Uresk 1990). While a 1:5 extract provides a fair indication of the prevalence of certain salts, it does not represent the true chemical environment experienced by a plant. The 1:5 extract has no direct relationship with the range of moisture in the field as contrasted with the saturation extract. Concentrations of some salts increase with increasing moisture content while concentrations of other salts decline. This phenomenon results from differences in solubilities among salts, changes in activity coefficients, diverse ion effects, cation exchange reactions, negative adsorption of ions, hydrolysis, increased solution of silicate minerals, alkaline earth carbonates, and low solubility of gypsum (Richards 1954). Sulfate or carbonate salts in significant quantities can cause large deviations in estimates from actual measured quantities in saturation extracts. Concentration of sulfate ions in bentonite mine spoil is high, averaging over 2000 $\mu g\ g^{-1}$ (Voorhees and Uresk 1990).

Chemical and physical characteristics of bentonite spoils for reclamation and revegetation of spoils are very difficult to obtain and are expensive. Our purpose was to develop simple regression models for an easier, yet reliable, method of obtaining chemical characteristics from bentonite spoils using 1:5 extracts vs the more expensive and time consuming saturation extracts for chemical analyses. Information on the relationships between concentrations of various salts in 1:5 extracts and saturation extracts prepared for the same sample would be useful to interpret the chemistry of bentonite spoil for a larger area with less time and expense. The 1:5 extract may provide a more accurate insight on changes in the plant environment with changes in moisture content that can be applied over a larger area with increased sample collection for analyses.

The objective of this study was to test the reliability of estimating salt content from 1:5 extracts as compared with saturation extracts of bentonite mine spoil.

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Materials and Methods

Sixteen spoil samples were collected at random from 0 to 20 cm depth from bentonite mined sites near Upton, Wyoming (Uresk and Yamamoto 1986; Uresk and Yamamoto 1994). Each of these samples was dried and mixed prior to assay for pH, electrical conductivity (EC), and soluble Ca^{++}, Mg^{++}, Na^{+}, K^{+}, HCO_3^-, $SO_4^=$, and Cl^- in 1:5 (1 spoil:5water) extract. A subsample of each sample was also analyzed for these same constituents using saturation extracts. The saturation percentage was also measured. Soluble ion content of each saturation extract was estimated from the soluble ion content of the 1:5 extract using the following formula: Ion concentration = 1:5 extract x 500/saturation percentage. All samples were analyzed at Colorado State University Soil Testing Laboratory.

The pH was measured using a combination electrode on the saturation paste and 1:5 spoil:water extract. EC was measured on 1:5 extracts by a conductivity cell. EC was measured in the soil saturation paste extracts after the paste was vacuum filtered. Levels of Ca^{++}, Mg^{++}, K^{+}, and Na^{+} were measured in saturated and 1:5 extracts using inductively coupled plasma atomic emission spectrometry (Jones 1977). Sulfate content of spoil was measured using a turbidimetric method (Rhoades 1982). Bicarbonate and Cl were determined by titration and ion-chromatography procedures, respectively (Franson 1989).

All estimated values for 1:5 extracts were compared with measured values from saturated extracts by examination of scatterplots and regression analysis. Measured EC of the saturation extracts were compared with estimated EC from 1:5 extracts (500/saturation percentage x electrical conductivity 1:5 extract). Prediction equations developed from regression analyses were determined for significant variables (SPSS 1999). Calibration intervals (95 percent prediction limit) for individual points were determined for each of the ions and electrical conductivity. An example to estimate the calibration intervals for an extract point is presented in a figure (Graybill 1976). Regression relationships between measured saturation and estimated values were accepted as statistically significant at $p \leq 0.05$.

Results and Discussion

The concentration of all ions except Cl^- was greater in saturation extracts as compared to 1:5 extracts (table 1). Regression analysis showed that levels of Cl^- in 1:5 extracts were related to levels of $SO_4^=$ in these 1:5 extracts ($r^2 = 0.59$; $p = 0.0005$). However, levels of Cl^- in saturation extracts were not predictable from levels of Cl^-, $SO_4^=$ or HCO_3^- in either 1:5 or saturation extracts. Chloride levels in plant tissue decrease when $SO_4^=$ in the substrate increases (Eaton 1966), presumably because of decreased solubility of Cl^- in the presence of the more soluble $SO_4^=$. These ionic relationships when high or low, have important implications for plant growth and demonstrate the complexity of solute and cation exchange, especially on a substrate as complex and unique as bentonite.

Table 1—Means (SD) for selected chemical variables and ion concentrations in saturation and 1:5 extracts and for estimations of saturation extract measurements from 1:5 extract measurements (Est) of bentonite mine spoil samples taken from a site near Upton, Wyoming.

Variables	Units	Saturation	1:5	Estimate
pH	-	5.8(0.5)	6.1(0.5)	-
EC*	S/m	0.71(0.19)	0.42(0.59)	-
Ca	meq/l	6.7(3.6)	4.4(3.6)	12.1(12.3)
Mg	meq/l	4.1(1.9)	1.9(0.6)	4.8(2.2)
Na	meq/l	64.3(12.0)	21.3(2.1)	52.5(12.9)
K	meq/l	0.27(0.04)	0.24(0.04)	0.58(0.16)
HCO_3	meq/l	0.53(0.20)	0.28(0.19)	0.64(0.34)
SO_4	meq/l	96(27)	44(10)	112(35)
Cl	meq/l	0.68(1.11)	3.3(5.7)	7.9(13.5)

*EC = electrical conductivity

Linear regression coefficients between measured chemical variables in saturation extracts with variables in 1:5 extracts (table 2) showed significant correlations for EC, Ca^{++}, Mg^{++}, Na^{+}, K^{+}, HCO_3^-, and $SO_4^=$, ($p \leq 0.05$). However, r^2 values were considered significant ($r^2 \geq 0.50$) only for EC, Ca^{++}, Mg^{++}, Na^{+}, $SO_4^=$ and HCO_3^-. Therefore, estimation of saturation extract values from 1:5 extract measurements on bentonite mine spoil should be limited to these ions. Overall predictability from 1:5 extracts was good for these variables (r^2 ranged from 0.72 to 0.89) (figure 1). Thus these equations would best be used to predict a single estimate from bentonite mine spoil with the 1:5 extracts using the estimated values.

An example of a calibration estimate to predict at the 95 percent limit for single point estimate is presented in figure 2 for Na. A calibration estimate (figure 2) of saturated Na from the 1:5 extract is obtained by drawing a horizontal line from the estimated value through the regression model and prediction limits. A line is then drawn down to the saturated Na axis for an estimated 60 meq/l and for both predication limits. The calibration-saturated

Table 2—Linear regression coefficients for the relationship between chemical variables measured in saturation extracts and estimates of similar chemical variables from 1:5 extracts for bentonite mine spoil taken from a site near Upton, Wyoming.

Variables	r^2	p	SE[1]	SE[2]
EC*	0.89	≤0.001	0.024	0.158
Ca	0.77	≤0.001	0.584	4.660
Mg	0.85	≤0.001	0.212	1.804
Na	0.89	≤0.001	1.127	10.124
HCO_3	0.70	≤0.001	0.048	0.252
SO_4	0.85	≤0.001	3.520	25.993
K	0.37	0.012	-	-
Cl	≤0.01	0.996	-	-

* EC = electrical conductivity
[1] Standard error of the estimate
[2] Calibration standard error

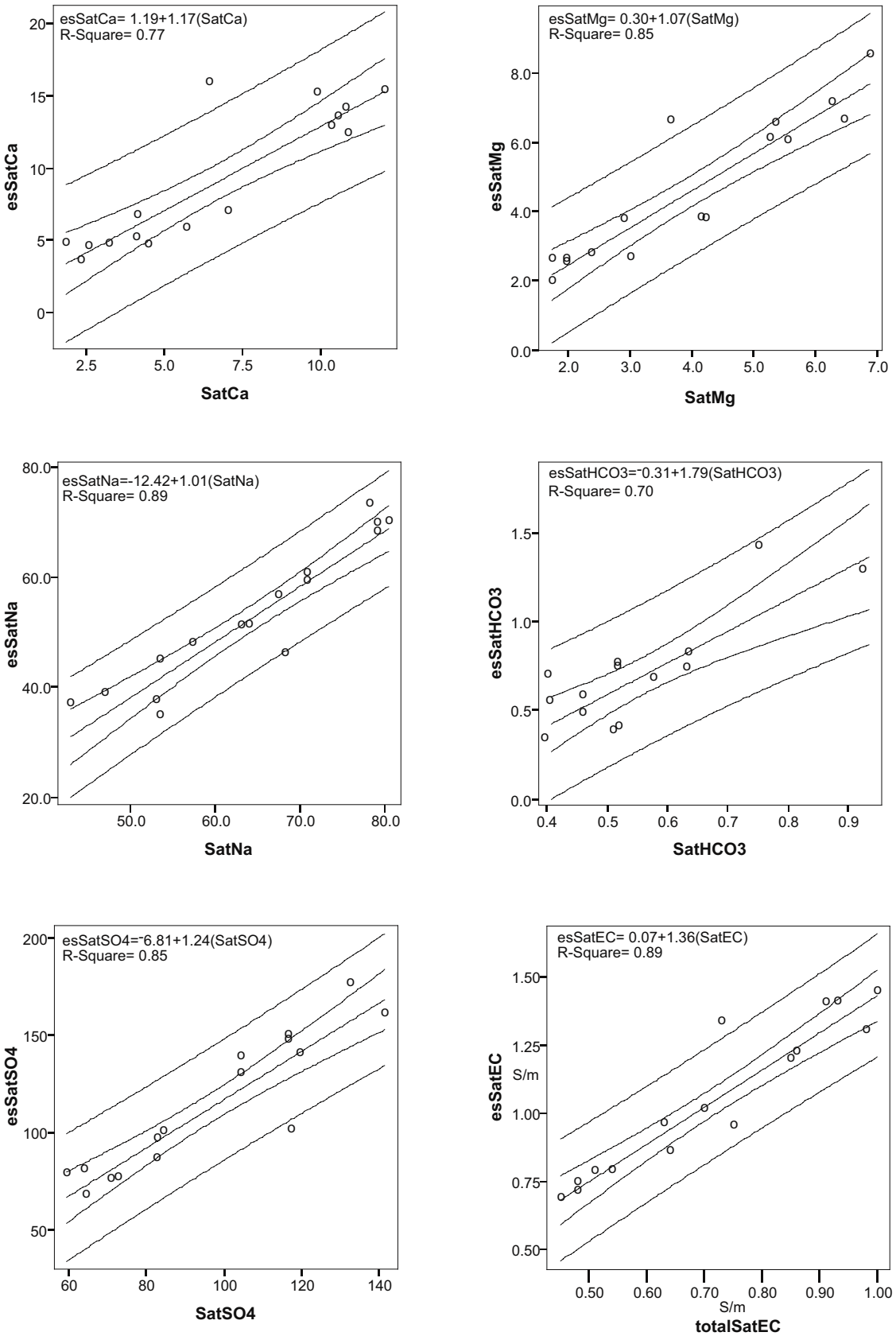

Figure 1—Scatterplots of measured (saturation extract) versus estimated concentrations of saturation extracts (1spoil:5water) in meq/l for Ca^{++}, Mg^{++}, Na^{+}, K^{+}, Cl^{-}, HCO_3^{-}, and SO_4^{-} ions; and estimated total ions S/m (totalSatEC) versus electrical conductivity esSatEC. Scatterplots are 95 percent prediction intervals for the mean and single observations.

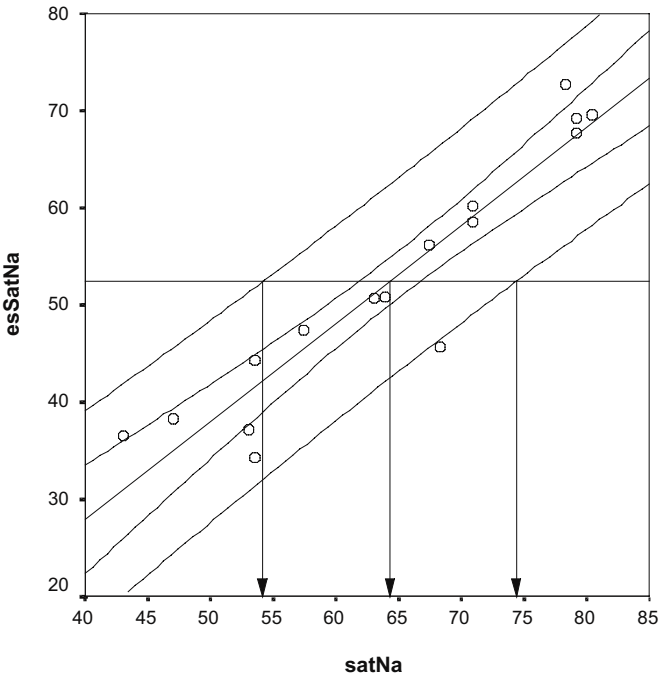

Figure 2—Calibration example for Na^+ based on regression between estimated 1:5 extracts ($esSatNa^+$) and measured (saturation, $SatNa^+$) with associated 95 percent prediction limits for a single observation.

estimate is 60 with limits of 49 and 71. Reliability of ±11 units for Na is considered sufficiently precise for management purposes considering a single point (figure 2). Many points can be estimated and pooled for a mean to represent the area of concern for management. Sodium is one of the most important ions that affects the chemical and physical environment of plants on bentonite mine spoil, so this finding should be useful for revegetation efforts.

The predictive equations are reliably accurate for the variables presented in figure 1. Since the reliability for all ions tested is considered sufficient for a single point, the response of spoil to treatments designed to modify ion concentrations can be monitored using 1:5 extracts with less effort and lower costs.

Ions with high correlations (Ca^{++}, Mg^{++}, Na^+, HCO_3^-, $SO_4^=$) made up 96 percent of the ions in saturation extracts and more than 95 percent of the ions in the 1:5 extract (figure 1). Estimates of total salts in saturation extracts from measurements of salts in 1:5 extracts are highly correlated ($r^2 = 0.89$) with the electrical conductivity of saturation extracts ($p \leq 0.001$). Where sulfate or carbonate salts with relatively low solubility are present in appreciable quantities, the apparent amount of soluble salt depends on the soil:water extraction ratio. These results show that soil salinity (EC) measurements of 1:5 extracts where $CO_3^=$ and $SO_4^=$ salts are present will provide an estimate similar to saturation extracts.

Poor correlations between measured and calculated parameters for Cl^- ions were attributed to diverse ions effects, cation exchange reactions, negative adsorption of ions, hydrolysis, and increased solution of silicate minerals, alkaline earth carbonates, and gypsum. The predictive value of equations for those ions with low correlations between 1:5 extract concentrations and saturation extract concentrations (i.e., Cl^-, K^+) would likely be improved by including other predictive variables in the equation, such as the concentrations of other ions. This was not considered to be within the scope of this study and would require a larger sample size for multiple regression.

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