EVALUATION OF 1:5 SOIL TO WATER EXTRACT ELECTRICAL CONDUCTIVITY METHODS AND COMPARISON TO ELECTRICAL CONDUCTIVITY OF SATURATED PASTE EXTRACT

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Evaluation of 1:5 Soil to Water Extract Electrical Conductivity Methods and Comparison to
Electrical Conductivity of Saturated Paste Extract

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ABSTRACT

He, Yangbo, M.S., Department of Soil Science, College of Agriculture, Food Systems, and Natural Resources, North Dakota State University, June 2011. Evaluation of 1:5 Soil to Water Extract Electrical Conductivity Methods and Comparison to Electrical Conductivity of Saturated Paste Extract. Major Professor: Dr. Thomas M. DeSutter.

Conducting a 1:5 soil:water extract to measure electrical conductivity (EC) is an approach to assess salinity and is the preferred method used in Australia. However, the influence of salinity on plant growth is predominantly based on saturated paste extract electrical conductivity (EC_e) and EC_e is recommended as a general method for estimating soil salinity internationally, so it is necessary to convert $EC_{1:5}$ to EC_e. The objectives of this research were to 1) compare methods of agitation (shaking plus centrifuging (shaking/centrifuging), shaking, and stirring) for determining $EC_{1:5}$; 2) determine optimal times for equilibration for each method across a range of salinity levels determined from saturated paste extracts (EC_e) (objectives 1 and 2 are for paper 1); and 3) develop predictive models to convert $EC_{1:5}$ data to EC_e based on four different 1:5 extraction methods listed above and a USDA-NRCS equilibration technique (objective 3 is for paper 2). The soils evaluated for the two studies were from north central North Dakota, USA, where 20 soil samples having EC_e values ranging from 0.96 to 21 dS m^{-1}were used for the first study (objectives 1 and 2), and 100 samples having EC_e values ranging from 0.30 to 17.9 dS m^{-1}were used in the second study (objective 3). In the first study, for each method, nine equilibrium times were used up to 48 hrs. In the second study, a uniform agitation time (8 hrs) was applied to the first three agitation methods, and 1 hr was also used for the USDA-NRCS method. For the first study, significant relationships ($p \leq 0.05$) existed between values of $EC_{1:5}$ and agitation time across the three methods. Agitation methods were significantly different ($p \leq 0.05$) from each other for 65% of the soils and shaking/centrifuging was significantly different ($p \leq 0.05$) from stirring for all soils. In addition, for 75% of the soils, shaking/centrifuging was significantly different ($p \leq 0.05$) from shaking. Based on these results, methods were analyzed separately for optimal equilibration times. The agitation times required for the three methods to reach 95 and 98% of equilibration were a function of the level of soil salinity. For soils with EC_e values less than 4 dS m^{-1}, over 24 hrs was needed to obtain both 95 and 98% of equilibration for the three methods. However, less than 3 and 8 hrs were needed to reach 95 and 98% equilibration, respectively, across methods for soils having EC_e values greater than 4 dS m^{-1}. These results indicate that establishing a standard method is necessary to help reduce variation across $EC_{1:5}$ measurements. In the second study, the value of EC_e was highly correlated with $EC_{1:5}$ ($p < 0.0001$) across four agitation methods in non-transformed, $\log_{10}$-transformed, and dilution ratio models through regression analysis. The values of coefficient of determination (r^2) were greatly improved and average about 0.87 using $\log_{10}$-transformation compared to other two models (r^2 values of about 0.68 for the non-transformed models and 0.69 for the dilution ratio models). Since agitation methods were determined to be highly correlated with each other, any regression model determined under the four agitation methods were applicable for the estimation of EC_e from another method. The results from this research indicate that comparing data across studies should be done with caution because both agitation method and time can influence results. Also, estimation of EC_e from $EC_{1:5}$ can be done with confidence, but models may not be transferrable across different soil orders or across various salt types.

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PREFACE

This thesis was written as a series of two manuscripts that will be submitted for publication in appropriate scientific journals. The 'Abstract' provides a general understanding of the importance of this study, and how both research studies are related to the main issue: evaluation of $EC_{1:5}$ soil to water electrical conductivity methods and comparison to electrical conductivity of saturated paste extract. The 'Literature Review' summarizes previous research and concepts that are related to soil electrical conductivity. The two research studies are then presented, and each article contains a specific abstract, introduction, materials and methods, results, discussion, conclusions, and references sections. Finally, the general conclusions from both research studies in the thesis are presented.

GENERAL INTRODUCTION

Salinization of soil is the accumulation of water soluble salts in the soil solum or regolith to a level that affects agricultural production, economic welfare, and environmental health (Rengasamy, 2006). Saline soils are characterized by the following standards: 1) electrical conductivity of saturated paste extract (EC_e) > 4 dS m^{-1}, 2) exchangeable sodium percentage (ESP) < 15, and 3) a pH < 8.5 (USDA, 1954). The estimated global area covered by salt-affected soils is about 930 million ha (2303 million acres) (Szabolcs, 1979; Szabolcs, 1989). The salts in soil may result from soil weathering processes, capillary transport of salts from shallow groundwater, ocean spray, or human irrigation (Chhabra, 1996). Salt accumulation decreases osmotic water potential in soil and adversely affects the water availability for crops and may be accumulated toxic levels for plants (Katerji et al., 2003). Accumulated salts (especially sodium salts) would affect soil properties by swelling and dispersion of clays in soil resulting in clogging of soil pores, which can then lower soil's permeability and decrease its infiltration (Shainberg and Singer, 1990). Due to the lower osmotic potentials in saline soils, plants exhibit water stress symptoms such as wilting, stunting with cupped leaves, and chlorosis (Ogle et al., 2004; USDA, 1954).

Management ranging from tile drainage, leaching, no-tillage or shallow tillage for seedbed preparation to salt tolerant crops selection are common methods used to control saline soils (Franzen, 2007). Management strategies are based on soil type, salt type, plant variety and plant growth stage, and water availabilities. In order to determine the severity of saline soils and the influence on crop production, soil salinity levels have to be measured.

Accurately measuring soil salinity using a reliable method will allow one to determine whether a soil is saline versus non-saline level of salinity (Abrol et al., 1988).

Electrical conductivity (EC) of a soil extract is one of the most widely used parameters for describing soil salinity (USDA, 1954). Soil salinity is conventionally defined and measured internationally using saturated paste extracts (EC_e) (USDA, 1954), where this extract attempts to simulate the environment of naturally occurring soil moisture. Results from these extractions are thought to be the best indicator of plant response to salinity. However, difficulty exists in obtaining saturation extract, which prohibit it from routine use (Longenecker and Lyerly, 1964).

Soil to water suspensions of different ratios such as 1:1, 1:2, 1:2.5, 1:5 and 1:10 have been used to measure soil EC. The 1:5 ratio has the same benefits (simple and rapid) as the 1:1, 1:2 and 1:2.5 ratios, and it also has wide acceptance in Australia, China and Central Asia (Committee of Saline Soils of Soil Science Society of China, 1989; Rayment and Lyons, 2011; Shirokova et al., 2000). Techniques for determining $EC_{1:5}$ vary between studies from different countries and to date, there is no internationally recognized standard for determining $EC_{1:5}$, which would allow for greater confidence when comparing data across methods.

In general, predictions of EC_e from $EC_{1:5}$ have been very good with coefficient of determination (r^2) values greater than 0.91 (Khorsandi and Yazdi, 2007) and these predictions have been accomplished in different regions (Slavich and Petterson, 1993; Sumner and Naidu, 1998). Parameters considered to influence the relationship between EC_e and $EC_{1:5}$ include soil texture, types of salts present in the soil, and water content of the saturated paste soil (Al-Mustafa and Al-Omran, 1990; Chi and Wang, 2010; Khorsandi and

Yazdi, 2007; Sonmez et al., 2008). Due to the absence of an internationally recognized 1:5 sample preparation method, the relationships between EC_e and $EC_{1:5}$ that have already been established have varied between studies. No research was found when predicting EC_e from $EC_{1:5}$ that took into account agitation methods and agitation time, and to date, no research has been conducted on soils from the Northern Great Plains. Therefore, the purpose of this thesis is to evaluate different agitation methods and agitation times in making 1:5 soil to water extracts and to develop and test the relationship between values of $EC_{1:5}$ of 1:5 soil to water extract and EC_e of saturated paste extract under different agitation methods and the same equilibration time

LITERATURE REVIEW

Saline Soils and Salinity Problems

A saline soil is defined as one containing sufficient soluble salts to adversely affect the growth of most plants (Soil Science Society of America, 2001). Saline soils are characterized by the following standards: 1) electrical conductivity of saturated paste extract (EC_e) > 4 dS m^{-1}, 2) exchangeable sodium percentage (ESP) < 15 and, 3) a pH < 8.5 (USDA, 1954). Soil salinization is a widespread limitation to agricultural production especially in arid and semi-arid areas throughout the world and is a major impediment to sustainable agriculture worldwide (Qadir and Oster, 2002). The estimated global area covered by salt affected soils is about 930 million ha (2303 million acres) (Sumner and Naidu, 1998; Szabolcs, 1989) (Table 1).

Table 1. Global distribution of salt affected soils (Szabolcs, 1989).

Continent	Areas in millions of ha		
	Saline†	Sodic (Alkali)‡	Total
North America	6.2	9.6	15.8
Central America	2.0	—	2.0
South America	69.4	59.6	129.0
Africa	53.5	27.0	80.5
South Asia	83.3	1.8	85.1
North & Central Asia	91.6	120.1	211.7
Southeast Asia	20.0	—	20.0
Australasia	17.4	340.0	357.4
Europe	7.8	22.9	30.7
Total	351.5	581.0	932.2

† Saline is defined as presence of soluble salts in soil or water which may result in reduced plant production.

‡ Sodic is defined as presence of high proportion of sodium ions relative to other cations in a soil or in a water, in the United States and throughout much of the rest of the world, the ESP value of greater than 15 was the criteria for separating sodic soils, while in Australasia, the ESP value was lowered to 6.

Weathering of geologic materials is an important process for soil formation, but also helps introduce salts into the plant root zone. Salt accumulation in the plant root zone commonly occurs as water transports salts upward from shallow groundwater through capillary forces. Upward water movement is due to the soil water potential difference and is affected by soil texture, for example, large pores in coarse-textured soils offer much less resistance to water flow than fine-textured soils, but water will travel further up towards the surface in fine-textured soils (Chhabra, 1996; Hillel, 1998). As evapotranspiration occurs, dissolved salts precipitate and accumulate near or at the surface of the soil (Rengasamy, 2006; Seelig, 2000). Under irrigated agriculture, secondary salinization may occur if dissolved salts from irrigation water accumulate in the soil profile due to poor drainage, subsequently, the salts may migrate to the soil surface (Chhabra, 1996). Human fertilization and application of waste materials may also introduce salts to agricultural fields (Chhabra, 1996). Areas with sloping stratified geologic materials are prone to saline seeps, when the lateral flows of water along the impermeable layer discharges and forms saline areas (Seelig, 1978). In North Dakota, saline seeps are more commonly located in the south and west of the Missouri River and also in the Missouri Coteau (Seelig, 2000).

Dissolved salts, both cations and anions, have an affinity for water molecules and this attraction forms a hydration shell around the ions, and thus lowers the osmotic potential of soil water (Corwin, and Lesch, 2003; Hillel, 1998). Sometimes, the osmotic water potential is low enough in the soil to cause plant water in cells to move into the soil from plant roots (Lauchli and Epstein, 1990). Due to the reduction of the osmotic potential, plants exhibit browning and brittle leaf tips and margins, and may wilt and become chlorotic (Ogle et al., 2004; USDA, 1954). Salinity may also inhibit plant nutrient balance. For example, sodium ions have been shown to influence the calcium nutrition, and inadequate concentrations of calcium may adversely affect plant membrane function and growth (Cramer et al., 1988). If excessive amounts of salts enter the plant, they may accumulate to toxic levels in older leaves (Munns, 2002). Both decreased water potential and accumulated toxicity may reduce plant yield (Maas and Hoffman, 1975).

Excessive salts may also alter soil properties such as swelling, porosity, permeability, and water retention. In some cases, salts accumulate and form a white salt crust on the soil surface, and other times, no visible salt accumulation is observed, which is a factor of moisture content (Franzen, 2007). The negatively charged clay particles in soil attract cations, with which they form an electrostatic double layer (Hillel, 1998). When divalent cations within the clay interlayer are replaced by monovalent cations, the relatively weak force between clay layers and interlayer cations permits water to enter into the interlayer region, forcing layers apart, and causes swelling of clay (Schaetzl and Anderson, 2005). High sodium levels combined with low EC soil water can lower a soil's permeability due to collapsing of aggregates and clogging of large interaggregate pores (Shainberg and Singer, 1990). Dispersed clay particles not only seal the open soil surface, but may move with percolating water and migrate into the soil profile, which can be seen by the presence of argillic horizon deeper in the soil profile (Hillel, 1998). The hydraulic conductivity of soil is affected by salt composition and concentration, and the nature (e.g., whether primarily kaolinite or montmorillonite) and quantity of clay present (Hillel, 1998; Shainberg and Singer, 1990). Reduced hydraulic conductivity for saline soils may control water movement through the profile, or a nearly impermeable soil layer can be developed in sodic soils that can cause a perched water table (Robbins and Wiegand, 1990).

A variety of methods have been identified to mitigate soluble salt accumulation in soils. Leaching soluble salts using low EC water ($EC \leq 2.3$ dS m^{-1}) is primarily used in irrigation cropping systems with the goal of reducing salinity below the minimum plant tolerance level (USDA, 1954), and the amount of water required for leaching has been estimated by Watson and Knowles (1999). Tile drainage is an effective way to remove salts through tile lines into drainage canals or natural water ways from saline areas (USDA, 1954; USDA, 2005). To prevent the upward movement of salts into the root zone through capillary rise, the USDA (2005) recommends that the groundwater table must be lowered to a depth greater than 1.2 m. Salt tolerant crops such as sugarbeet (*Beta vulgaris*) and barley (*Hordeum vulgare*) have also been used in saline areas, and plants such as alfalfa (*Medicago sativa*), and wheatgrass (*Triticum aestivum*) are used to help reduce the upward movement of salts via capillary rise (Franzen, 2007; Kruse et al., 1990; Ogle and John, 2010).

Electrical Conductivity

Electrical conductivity of a soil extract is the most widely used approach for describing soil salinity (USDA, 1954). Electrical conductivity is the reciprocal of the electrical resistance measurement of a material across a specified volume (Rhoades et al., 1999; USDA, 1954), and is more suitable for salinity assessment than electrical resistance because conductivity increases with salt content, thus simplifying the interpretation of readings (USDA, 1954). The EC of a soil suspension is used to estimate the concentration of soluble salts in the soil, which consist predominately of cations Na^+, Ca^{2+}, and Mg^{2+} and the anions Cl^-, SO_4^{2-} and HCO_3^- (Rayment and Higginson, 1992).

Since conductance (C) is reciprocal of resistance (R) (ohms), C is expressed in reciprocal ohm of mhos (USDA, 1954), where the resistance of a soil solution is related to cross-sectional area (A) (cm^2) (inversely proportional) and its length (L) (cm) (directly proportional). Specific resistance (Rs) is the resistance of a cube of sample (1cm edge), but practical conductivity cells are not in this dimension and in theory, the cell constant (K) is determined by the cell electrode space distance (L) and cross-sectional area (A) ($K = L/A$), where the unit is cm^{-1}. The actual conductivity reading is obtained by multiplying the conductance (C) by cell constant (K). When K is applied, the measured conductance is specific conductance which is referred to as electrical conductivity (FAO, 1999; USDA, 1954). In the international system of units (SI), the reciprocal of ohm is the siemen (S), and in this system, EC is reported in siemens per meter ($S\ m^{-1}$) or decisiemens per meter ($dS\ m^{-1}$) (the later unit was used because it has the same numerical value as the old commonly used unit of $mmho\ cm^{-1}$) (FAO, 1999; USDA, 1954). Other specific conductivity units that are present in soil science literature and in other science disciplines include $\mu mho\ cm^{-1}$, $\mu S\ cm^{-1}$or $mmho\ cm^{-1}$.

The total amount of electrical current that can be conducted through an aqueous solution is related to the species, concentration, and the specific nature of dissolved ions (valence of ions) (Reluy et al., 2004; Tolgyessy, 1993). Formulas that approximate EC to the concentration of ion species in water have been developed. For example, Eq. [1] shows the relationship between EC and dissolved ions (Tolgyessy, 1993),

$$\sum (C_i f_i) = EC \qquad [1]$$

where, EC is the electrical conductivity ($\mu S\ cm^{-1}$); C_i is the concentration of ionic species *i* in the solution ($mg\ L^{-1}$); and f_i is the conductivity factor for ionic species *i*, where the conductivity factor reflects the cations and anions carrying electrical current to a different degree. The conductivity factors of ions commonly found in water is shown in Table 2. For example, f_i for Ca^{2+} is 2.60 $\mu S\ cm^{-1}$ per $mg\ L^{-1}$, for SO_4^{2-} is 1.54 $\mu S\ cm^{-1}$ per $mg\ L^{-1}$. In reality, the calcium concentration at saturation is about 600 $mg\ L^{-1}$ without other ions besides calcium and sulfate, thus the contribution of gypsum in soil would be about 2.484 dS m^{-1} ((2.60+1.54) µS cm^{-1} per mg L^{-1} × 600 mg L^{-1}) (Hem, 1985).

Table 2. Conductivity factors of ions commonly found in water (Greenberg et al., 1980).

Ion	Conductivity factor (25°C)
	µS cm^{-1} per mg L^{-1}
Ca^{2+}	2.60
Mg^{2+}	3.82
K^+	1.84
Na^+	2.13
HCO_3^-	0.72
CO_3^-	2.82
Cl^-	2.14
NO_3^-	1.15
SO_4^{2-}	1.54

1dS m^{-1} = 1000 µS cm^{-1}

Total dissolved solids (TDS) can be used to assess soil salinity, however, plants respond to salt concentration in the soil solution rather than total salt content of soil (Rhoades et al., 1989), whereas, EC is commonly used as an expression of the total dissolved solute concentration of an aqueous sample (FAO, 1999). The relationship for EC and TDS is TDS (mg L^{-1}) ≈ 640 × EC (dS m^{-1}) (for less saline soils with EC between 0.1 and 5.0 dS m^{-1}), or TDS (mg L^{-1}) ≈ 800 × EC (dS m^{-1}) (for EC > 5.0 dS m^{-1}) (FAO, 1999; Sparks, 2003). Total cation concentration (TCC) is also an important parameter used to describe salinity and is broadly related to EC by TCC (mmol$_c$ L^{-1}) ≈ 10 EC (dS m^{-1}) and has been used for saturated pastes and 1:5 extracts (USDA, 1954). Soil solution ionic strength (*I*) is related to EC. Ionic strength is a measure of the total concentration of ions in solution and is important for the determination of the activities of ions in solution (Bhuiyan et al., 2009; Snoeyink and Jenkins, 1980),

$$I = 1/2 \sum C_i Z_i^2 \qquad [2]$$

where, I is ionic strength (mol L^{-1}); C_i is concentration of the i^{th} ion in solution (mol L^{-1}); and Z_i is the charge of the i^{th} ion. Gillman and Bell (1978) measured I of a range of soils from North Queensland and provided a relationship of $I_{0.1} = 0.0446*EC_{1:5} - 0.000173$, where $I_{0.1}$ was soil ionic strength at 0.1 bar, and $EC_{1:5}$ had unit of dS m^{-1} at 25°C.

The solubility of salts will influence the concentration of salts in the soil water, and will thus influence EC measurements (Rayment and Lyons, 2011). Some of the dissolved salts are non-ionic and some ions combine to form ion-pairs, which are less charged or neutral and thus contribute less to EC than fully dissociated ions (FAO, 1999). An ion-pair is a pair of oppositely charged ions held together by coulomb attractions without forming covalent bond. Experimentally, an ion-pair behaves as one unit in determining conductivity. For example, about 28.3% of the soluble Ca and SO_4 in a 10 mM $CaSO_4$ solution are paired as neutral $CaSO_4^0$ (Adams, 1974). The neutral ion-pair species and reduced charge ion-pairs will influence the single ion activity coefficients in solution and then further affect the ionic strength-EC relationship (Griffin and Jurinak, 1973; Sparks, 2003).

EC Measurement in Different Countries

Although determination of soil EC is common worldwide, various methods are used to quantify this parameter. In the United States and Spain soil salinity is most commonly assessed using saturated pastes (Table 3). However, dilution methods are also frequently used with the 1:5 being the predominant method used in Australia, China, and Central Asian countries. In China, the 1:5 ratio is the most popular method used to determine soil salinity and is related to the main salts in solution that contribute to EC of soil extracts (Committee of Saline Soils of Soil Science Society of China, 1989). In Central Asia, the classifications of soil salinity are based on laboratory measurements of the total dissolved (toxic) salts or the chloride ion concentration in the 1:5 soil water extracts (Shirokova et al., 2000). Even though each of the methods listed in Table 3 are acceptable, the conversion of one method to another is not straight forward and data collected from different methods can be difficult to compare.

Advantages and Limitations for Alternative EC Measurement Methods

Among the many EC methods, the conductivity of saturated extract (EC_e) is recommended for appraising soil salinity (USDA, 1954). This measurement has been proven to be very consistent and has become the "world standard" for classifying soil salinity (USDA, 1954). Popularity of the saturation extract method lies in the fact that saturation percentage (SP) is directly related to the field moisture range where the moisture content of field soil fluctuates between lower permanent wilting percentage and the upper wet end of saturation. The concentration of soluble salts in a saturation extract is about one fourth of that at the lower end of field moisture range (wilting point), and two times that in the upper field moisture range (field capacity) (USDA, 1954). The effect of salinity on crop yield has been characterized by EC_e and yield is not significantly decreased until a threshold EC_e is exceeded (Maas and Hoffman, 1975; Tanji and Kielen, 2002). The model describing this relationship is

$$Y = 100 - B(EC_e - A) \qquad [3]$$

This linear plateau model is a simplified form described by Maas and Hoffman (1975) where Y is the calculated crop yield in percent (100% is maximum); B is the percent yield decrease per unit salinity increase above the threshold; EC_e is the average root zone salinity (dS m^{-1}; saturated extract); and A is the threshold EC_e in dS m^{-1} (Tanji and Kielen, 2002).

Table 3. Popular measurements of EC in different countries.

Country or organization	Popular methods	Processes	References
FAO[†]	EC_a[‡]	Measure using EM; Four-electrode sensors; TDR	FAO (1999)
	EC_e[§]	Add distilled water to air-dred soil (200–400 g), stir and allowing mixture to stand several hr, soil paste should glisten as it reflects light, flow slightly when the container is tipped, slide freely and cleanly off a spatula, and consolidate easily (saturation), extract mixture by suction using a funnel and filter paper, measure EC_e with conductivity meter	FAO (1999)
	EC_w[¶]	Collect a sample of soil water using an in-situ extractor and measure its EC; the second is to measure EC_w directly in the soil using in-situ, imbibition-type salinity sensors	FAO (1999)
Australia	$EC_{1:5}$[#]	Mechanically shaking the required amount of soil sample and DI water at 25 °C in a closed system for 1 hr, followed by 20–30 min for the soil to settle, measure the supernatant by conductivity cell	Rayment and Higginson (1992)
USA	EC_e	Add DI water to air-dried soil while stirring with a spatula. After mixing, the mixture is allowed to stand for one hr or more, and then criteria of saturation should be checked. At saturation the soil paste glistens, flows slightly when the container is tipped, and slides freely off the spatula. Soil paste is extracted with a funnel, filter paper and vacuum pump, and EC determined	USDA (1954)
	$EC_{1:1}$	Place the required amount of soil and distilled water (1:1) in a bottle, and agitate in a mechanical shaker for 15 min. Allow content to stand for at least one hr, agitate again for 5 min, filter, and then measure EC using a conductivity meter. Or if the content was shaken by hand (the solution was shaken for 30 s at least 4 times at 30 min intervals before filtration), measure EC as above	USDA (1954)
	$EC_{1:5}$	Similar as 1:1 ratio extract, but soil/water is 1:5	USDA (1954)
	$EC_{1:5}$[††]	Soil and required amount of water were added to polyethylene bottle, the soil water suspension was maintained at room temperature for 23 hr, then shaken on a reciprocating shaker for 1 hr, and then filtered followed by measurement of filtered solutes by EC meter	Soil Survey Staff (2011)
China	$EC_{1:5}$	Procedure is similar to that in U.S. Salinity Laboratory Staff (1954)	Committee of Saline Soils of Soil Science Society of China (1989)
Central Asia	$EC_{1:5}$	USSR classifications of soil salinity used in Central Asia is based on measuring total dissolved solids and sum of soluble salts in 1:5 soil water extract. No detailed procedure mentioned	Shirokova et al. (2000)
Spain	EC_e	Saturated pastes were prepared by adding DI water ($EC_{25} \approx 1$ µS cm^{-1}) to 400 g of soil according to the method described by Rhoades (1946), stir until nearly saturated, then soil water mixtures were allowed to equilibrate for 4 to 5 hr at room temperature, filter with highly retentive paper, vacuum to collect filtrate, and determine EC of filtrate	Visconti (2010); Rhoades (1996)
	$EC_{1:5}$	Add 60 mL of DI water to 12 g of soil in 200 mL screw lid containers. Lids contained six 9-mm holes for gas exchange. The suspensions were shaken in a reciprocal shaker for 24 hr, poured into a 60 mL centrifuge tube and centrifuged at 1400 *g* for 10 min. The solutions were decanted , filtered and EC determined	Visconti (2010)

[†] FAO, Food and Agriculture Organization.

[‡] EC_a, electrical conductivity of bulk soil.

[§] EC_e, electrical conductivity of saturated soil paste extract.

[¶] EC_w, electrical conductivity of soil solution.

[#] $EC_{1:1}$, $EC_{1:5}$, electrical conductivity of 1:1 and 1:5 soil to water extract.

[††] Procedure not developed for determining EC.

Disadvantages still exist in obtaining a saturation extract, which prohibits this method from routine use. Disadvantages include the time needed for equilibration (≈ 24 hr) and the skill requirement needed to assess the correct moisture content of the paste, which is also related to the reproducibility of this method (Longenecker and Lyerly, 1964). For example, for very fine-textured soils (clay soils), the amount of water that must be added to reach saturation can vary by 10% or more and is influenced by the rate of adding water and the amount of mixing, where the more rapid the rate of adding water, the lower the SP may be (USDA, 1954).

Due to the difficulties encountered in preparing saturation extracts, soil to water suspensions of different ratios such as 1:1, 1:2, 1:2.5, 1:5 and 1:10 have been used to measure soil EC. These dilutions are popular because more information can be gathered about soil salinity with less labor compared to saturation extract (Jurinak and Suarez, 1996). Also, these dilutions have advantages of simplicity, reduced time, and monetary investment compared to saturation extract (Franzen, 2007; Sonmez et al., 2008). Khorsandi and Yazdi (2007) estimated EC_e by EC of 1:2 and 1:5 extraction ratios and found that the relative time required for preparation and analysis of one sample in their laboratory was 4.9 min for either of the two dilution ratio methods compared to 36 min for saturated paste methods.

The higher dilution ratios can also be used for soil salinity assessment and good relationships have been developed between $EC_{(1:n)}$ to EC_e,

$$EC_e = K \times EC_{1:1(1:2.5,\ 1:5)} \qquad [4]$$

where, EC_e is the EC of saturated paste extract (dS m^{-1}), $EC_{1:1(1:2.5,\ 1:5)}$ is the dilution ratio EC (dS m^{-1}), and K is an empirical factor (Shirokova et al., 2000). Applying the relationships allows one to use the convenience of the dilution method to assess plant response by converting to EC_e.

The 1:1 ratio is a common commercial laboratory measurement in addition to saturated paste (USDA, 1954). The 1:5 ratio has the same benefits as the 1:1, 1:2 and 1:2.5 ratios, and has wide acceptance in Australia because other parameters such as pH, water soluble Cl^- and NO_3^- can be determined from the same extract (Rayment and Lyons, 2011). In addition, sparingly soluble salts will contribute to EC at a greater extent at 1:5 than at more concentrated ratios such as 1:1, 1:2, 1:2.5, and saturation extract due to the additional water dilution (Rayment and Higginson, 1992).

Specific $EC_{1:5}$ Measurement Procedures

The $EC_{1:5}$ is commonly used to measure soil salinity in irrigated areas around the world. However, due to the diversity of soil properties, and the absence of a uniform international sample analysis, the standard measurement of $EC_{1:5}$ has not been established (Zhang et al., 2009) and thus $EC_{1:5}$ determinations procedures vary between studies from different countries (Table 3). Also, various preparation methods range from mechanically shaking, shaking plus centrifuging, to hand stirring, different agitation times to mix the soil and water vary, and different settling methods and times prior to EC measurement have also been used to make 1:5 extracts in different studies worldwide (Table 4). No comparison has been done between the different techniques in preparing 1:5 extract and EC analysis between different studies. The factors listed above make the soil salinity analysis work by 1:5 extract electrical conductivity not uniform in somewhat, and make the data transfer from one study to another difficult.

Table 4. Specific agitation methods, equilibration times, and times prior to $EC_{1:5}$ measurement from different studies.

Method	Agitation time	Settling method and time prior to EC measurement	References
Mechanically shake 15 min, stand at least 1 hr, agitate again for 5 min, filter and take EC	20 min	Filtration	USDA (1954); Chi and Wang (2010)
Mechanically shake for 1 hr, settle for 20–30 min, take EC	1 hr	Naturally settling 20–30 min	Loveday (1974); Rayment and Higginson (1992)
Mechanically shake for 24 hr, centrifuge for 10 min, take EC	24 hr	Centrifugation 10 min	Visconti et al. (2010)
Mechanically shake for 1 hr, or shake by hand for 1 min at least 4 times at 30 min intervals, filter, take EC	1 hr	Filtration	Rhoades (1982); Marion et al. (1991); Khorsandi and Yazdi (2007)
Mechanically shake for 1 hr, suspensions were centrifuged, supernatant is filtered, then take EC	1 hr	Centrifugation and filtration, no specific time	Nortario del Pino et al. (2008) and Marion et al. (1991)
Mechanically shake for 12 hr, then filtered with 0.45 μm filter paper, take EC	12 hr	Filtration	Hurraβ and Schaumann (2006)
Stir over a period of 1 hr, then filtered for extract, take EC	1 hr	Filtration	Al-Mustafa and Al-Omran (1990)
Shake for 1 min of soil and rain water slurry(soil is placed into graduated bottle 100 mL mark and rain water is added to 600 mL mark), settle 1 min, take EC, determine by pre-made table	1 min	Naturally settling 1 min	Henschke and Herrmann (2007)
Shake greater than 30 min, settle 15 min prior to take EC	> 30 min	Naturally settling 15 min	Walker (2008)

Estimating EC_e from $EC_{1:5}$

Significant relationships exist between EC_e and $EC_{1:5}$ for soils conducted from Uttar Pradesh with divergent saline-sodic tracts, for soils collected from Riverine Plain having the properties of flood plain soil types, and for soils located in Songnen Plain, Northeast China, with the parent minerals rich in smectite clay and sodium-aluminum silicate minerals (Agarwal et al., 1961; Chi and Wang, 2010; Slavich and Petterson, 1993). In general, predictions of EC_e from $EC_{1:5}$ have been very good with coefficient of determination (r^2) values greater than 0.91 in different regions (Table 5). However, because soils of different regions may vary considerably in properties as well as in their composition of soluble salts, the regression equations used to describe EC_e to $EC_{1:5}$ relationships also can vary (Table 5).

Table 5. Regression equations for relating EC_e and $EC_{1:5}$ in different regions.

Category	Regression equation	r^2	Reference
Soil texture			
Sandy soil	$EC_e = 8.22\ EC_{1:5} - 0.33$	0.98	Sonmez et al. (2008)
Loamy soil	$EC_e = 7.58\ EC_{1:5} + 0.06$	0.99	
Clay soil	$EC_e = 7.36\ EC_{1:5} - 0.24$	0.99	
Water content			
$\theta = 0.20–0.45$	$EC_e = 11.74EC_{1:5} - 6.15$	0.94	Chi and Wang (2010)
$\theta = 0.45–0.63$	$EC_e = 11.04EC_{1:5} - 2.41$	0.96	
$\theta = 0.20–0.63$	$EC_e = 11.68EC_{1:5} - 5.77$	0.94	
Gypsum			
No gypsum	$EC_e = 7.9432EC_{1:5} + 0.2792$	0.91	Khorsandi and Yazdi (2007)
Gypsum	$EC_e = 9.1447EC_{1:5} - 15.723$	0.94	
No specific standard			
—	$EC_e = (2.46 + 3.03/\ \theta_{SP}^{\dagger})\ EC_{1:5}$	0.99	Slavich and Petterson (1993)
—	$EC_e = 6.4EC_{1:5}$	—	Landon (1991)
—	$EC_e = 9.572EC_{1:5} - 1.012$	0.90	Al-Mustafa and Al-Omran (1990)
—	$EC_e = [(500 + 6ADMC^{\ddagger})\ /SP^{\S}]^{b\,\P}EC_{1:5}$	—	Shaw (1994)

† θ_{SP}, the water content of the saturated paste.
‡ ADMC, air dry moisture content.
§ SP, saturation percentage.
¶ b, coefficient, $0 < b < 1$.

Several major factors used to establish the relationship between EC_e and $EC_{1:5}$ have included soil texture, soil water content, salt type and amount, and dilution (Al-Mustafa and Al-Omran, 1990; Chi and Wang, 2010; Khorsandi and Yazdi, 2007; Slavich and Petterson, 1993; Sonmez et al., 2008). Soil texture has been shown to influence the relationship between EC_e and $EC_{1:5}$, and conversion factors estimating EC_e from $EC_{1:5}$ tend to increase as soil texture classes change from fine-textured to medium-textured (Al-Mustafa and Al-Omran, 1990; Sonmez et al., 2008). Slopes for regression equations between EC_e and $EC_{1:5}$ varied with saturated paste water content (θ_{SP}). The θ_{SP} is strongly related to clay content of soil so that it can be used for a broad characterization of soil texture groups (Slavich and Petterson, 1993). Therefore, the regression equations reported in the study of Slavich and Petterson (1993) estimating EC_e from $EC_{1:5}$ are classified in a range of θ_{SP} 0.20–0.45, and greater than 0.45, and has some similarity to the influence of soil texture on the relationship between ECe and $EC_{1:5}$ (Chi and Wang, 2010). Unfortunately, the value of $EC_{1:5}$ is not a simple dilution of the saturated paste extract and the conversion factor between EC_e and $EC_{1:5}$ is not 5 (Slavich and Petterson, 1993). The 1:5 extracts dissolve larger amounts of salts and exhibit a stronger effect of mineral dissolution compared to saturated pastes (Chi and Wang, 2010; Landon, 1991; Slavich and Petterson, 1993).

The relationships between EC_e and $EC_{1:5}$ are also dependent on the types of salts, and exhibit different degrees of variations within high and low salinity ranges. When soil samples contain gypsum the measurement of EC deviates and introduces errors in the interpretation of data, because calcium and sulfate concentrations remain near-constant or increase with dilution from SP to 1:5 ratio, while the concentration of other ions decreases with dilution (Robbins and Wiegand, 1990). Khorsandi and Yazdi (2007) observed that dividing their experimental results into soils with and without gypsum greatly improved the accuracy of their EC_e and $EC_{1:5}$ models. Agarwal et al. (1961) demonstrated that relationship between EC_e and $EC_{1:5}$ values could be improved if soils are classified on salinity status. For the high-salinity soil SO_4-Cl and CO_3-HCO_3 groups (EC_e values ranging from 12 to 300 dS m^{-1}), the ratio of $EC_e/EC_{1:5}$ was between 9.8 and 13 and 6.6 to 14.4, respectively, and fairly uniform, while the ratio was lower and ranged from 1.7 to 8.3 for low-salinity CO_3-HCO_3 group soils having EC_e values between 0.5 and 10 dS m^{-1}.

Although several equations predicting EC_e from $EC_{1:5}$ extractions have been proposed (Landon, 1991; Shaw, 1994; Slavich and Petterson, 1993), these relationships are empirical models for particular sets of data and are geographically limited in their applications. Also, Sonmez et al. (2008) stated that the estimated EC_e results from $EC_{1:5}$ using regression models are not as precise as results measured from saturated paste extracts. Predictive relationships between EC_e and $EC_{1:5}$ have been developed using various 1:5 extraction methods and times and thus uncertainty exists when applying these models to data derived from different extraction methods from which the models were developed. To date, no research has been conducted on soils from the Northern Great Plains with the intent of establishing relationships between EC_e and $EC_{1:5}$. So, we intend to test not only the equilibration time but the agitation methods effect on the $EC_{1:5}$ values and EC_e and $EC_{1:5}$ relationships in the soils from Northern Great Plains.

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PAPER 1. EVALUATION OF 1:5 SOIL TO WATER EXTRACT ELECTRICAL CONDUCTIVITY METHODS

Abstract

Conducting a 1:5 soil:water extract to measure electrical conductivity (EC) is an approach to assess salinity and has been the preferred method in Australia but not common in the United States. The objectives of this research were to 1) compare methods of agitation for determining $EC_{1:5}$ and 2) determine optimal times for equilibration for each method across a range of salinity levels determined from saturated paste extracts (EC_e). Soils evaluated for this study were from north central North Dakota (USA) and had EC_e values ranging from 0.96 to 21.2 dS m^{-1}. For each method, nine agitation times were used, up to 48 hrs. The three agitation methods were shaking plus centrifuging (shaking/centrifuging), shaking, and stirring. Agitation methods were significantly different ($p \leq 0.05$) from each other for 65% of soils and shaking/centrifuging were significantly different ($p \leq 0.05$) from stirring for all soils. In addition, 75% of the shaking/centrifuging soils were significantly different ($p \leq 0.05$) from shaking. Based on these results, methods were analyzed separately for optimal equilibration times. The agitation times required for the three methods to reach 95 and 98% of equilibration were a function of the level of soil salinity. For soils with EC_e values below 4 dS m^{-1}, over 24 hrs was needed to obtain both 95 and 98% of equilibration for the three methods. However, less than 3 and 8 hrs were needed to reach 95 and 98% equilibration, respectively, across methods for soils having EC_e values greater than 4 dS m^{-1}. These results indicate that establishing a standard method is necessary to help reduce variations across $EC_{1:5}$ measurements.

Introduction

Electrical conductivity (EC) of a soil extract is the most widely used parameter for describing soil salinity (USDA, 1954). Electrical conductivity estimates the concentration of ions in the soil, and consists predominately of cations Na^+, Ca^{2+}, and Mg^{2+} and the anions Cl^-, SO_4^{2-} and HCO_3^- (Rayment and Higginson, 1992). The standard laboratory method for determining the EC of a soil is by using a saturated paste extract (EC_e) (Rhoades et al., 1989; USDA, 1954). Due to the difficulties encountered in determining the appropriate water saturation point when preparing a saturated paste extract (Longenecker and Lyerly, 1964), soil to water ratios of 1:1, 1:2, 1:2.5, 1:5, and 1:10 have been used to determine the EC values of soils (Hogg and Henry, 1984; Slavich and Petterson, 1993; Sonmez et al., 2008). The 1:5 ratio is the preferred method for determining soil EC in Australia and China (Rayment and Lyons, 2011). The 1:5 ratio has the advantage of simplicity, reduced time, and cost compared to saturation extract (Franzen, 2007). The 1:5 ratio also dissolves larger amount of solutes than the saturation paste extract, especially for sparingly soluble salts (Reitemeier, 1946).

Both mechanical shaking and stirring methods have been used to prepare 1:5 extracts. For example, the standard 1:5 method used in Australia is by mechanically shaking the required amount of soil sample (20 g) and deionized water (DI) (100 mL) for 1 hr, followed by 20–30 min of undisturbed settling before measurement of EC (Rayment and Higginson, 1992). Other 1:5 procedures include agitation methods of shaking, and stirring, and agitation times between 20 min and 24 hrs (Chi and Wang, 2010; USDA, 1954; Visconti et al., 2010) followed by undisturbed settling, centrifuging, or filtration prior to EC measurement (Al-Mustafa and Al-Omran,1990; Chi and Wang, 2010; Hurraβ and

Schaumann, 2006; Khorsandi and Yazdi, 2007; Loveday, 1974; Marion et al., 1991; Notario del Pino et al., 2008; Rayment and Higginson, 1992; Rhoades, 1982; USDA, 1954; Visconti et al., 2010).

Although many $EC_{1:5}$ methods have been reported, influences on EC by different agitation methods and times for equilibration are likely to occur. Progress towards establishing a standard method for $EC_{1:5}$ is needed so that variations between different laboratory values can be minimized. The objectives of this research were to (1) compare three methods of preparation and extraction (shaking plus centrifuging, shaking and stirring) for determining $EC_{1:5}$ and (2) determine optimal times for equilibration for each agitation method across a range of soil EC_e.

Materials and Methods

Soil samples used in this study (n = 20) were collected from the 0 to 30 cm and 0 to 90 cm depths from soils in Benson and Ramsey counties in North Dakota, USA, (approximately 48°15′84″–48°33′48″ N, 98°59′76″–99°59′76″ W). All samples were Mollisols but had different suborder classifications and the five textural control sections ranged from fine (the particle size range is from 0.002 to 0.05 mm) to sandy over loamy (> 45% of sand, <50% of silt, and < 20% of clay) (Table 6). Each sample was air-dried, ground to pass through a 2 mm sieve, and stored in plastic bags until analysis. Saturated pastes were made following the methods outlined by UDSA (1954) and electrical conductivity of saturated paste extract (EC_e) was determined on each extract, which were done independently previously for a larger study looking at the soil salinity conditions in Devil's Lake Area prior to this study (Table 6).

Table 6. Taxonomic information and EC$_e$ of soils used in this study.

ID #	Series in map unit	Family	EC$_e$ dS m^{-1}
1	Hamerly-Barnes	Hamerly; Fine-loamy, mixed, superactive, frigid Aeric Calciaquolls Barnes; Fine-loamy, mixed, superactive, frigid Calcic Hapludolls	0.96
2	Hamerly-Barnes		1.24
3	Hecla	Hecla; Sandy, mixed, frigid Oxyaquic Hapludolls	2.05
4	Hamerly-Wyard	Wyard; Fine-loamy, mixed, superactive, frigid Typic Endoaquolls	2.91
5	Towner	Towner; Sandy over loamy, mixed, superactive, frigid Calcic Hapludolls	3.14
6	Hamerly-Wyard		4.66
7	Bearden-Lindaas	Bearden; Fine-silty, mixed, superactive, frigid Aeric Calciaquolls Lindaas; Fine, smectitic, frigid Typic Argiaquolls	5.33
8	Hamerly-Tonka	Tonka; Fine, smectitic, frigid Argiaquic Argialbolls	6.91
9	Overly	Overly; Fine-silty, mixed, superactive, frigid Pachic Hapludolls	7.06
10	Hamerly-Cresbard	Cresbard; Fine, smectitic, frigid Glossic Natrudolls	7.12
11	Hamerly-Tonka		9.02
12	Bearden	Bearden; Fine-silty, mixed, superactive, frigid Aeric Calciaquolls	9.23
13	Hamerly-Cresbard		11.3
14	Bearden		13.1
15	Cresbard-Barnes		13.8
16	Bearden		16.8
17	Cresbard-Svea	Svea; Fine-loamy, mixed, superactive, frigid Pachic Hapludolls	16.8
18	Cresbard-Barnes		17.1
19	Cresbard-Svea		19.6
20	Bearden		21.2

Soil suspensions were prepared using 35 mL of ultra-pure water and 7 g of soil. Treatments included three different agitation methods, nine agitation time levels, and four replications. Agitation methods included shaking plus centrifuging (shaking/centrifuging), shaking, and stirring. Soil suspensions were agitated in a mechanical shaker (132 rev min^{-1}) for the shaking and shaking/centrifuging methods. For the stirring method, subsequently, the sample was stirred using a glass rod for 10 sec initially and at the end of each of the times listed in the following. The detailed procedure can also be checked in Appendix A.

Suspensions were agitated for 5, 15, 35, 75, 175, 355, 715, 1435, and 2875 min (48 hrs) following the agitation time used in the studies of Chi and Wang (2010), Hurraβ and Schaumann (2006), Rhoades (1982), USDA (1954) etc. For the two shaking methods, after each agitation time level, the soil solutions assigned on each time were removed from the shaker and were allowed to settle for 5 min or were centrifuged for 5 min at a relative centrifuge force (RCF) of 4870 × g for the shaking and shaking/centrifuging methods, respectively. After the prescribed settling time EC was determined using a conductivity probe (Sension 378; Hach Co., Loveland, CO, USA). For the stirring method, samples dedicated to the specific agitation time were also allowed to settle for 5 min after the stirring interval, and the procedures about how to stir each of the solutions can be checked in Appendix A. All samples for each agitation time and method were used for only one EC measurement during this study.

For each agitation time and method, a 1413 $\mu S\ cm^{-1}$ standard solution (KCl) (Rayment and Higginson, 1992) and one blank (ultra-pure water only) were analyzed following the respective settling method and time criteria. Although temperature may affect EC readings, the EC meter used for this study was not influenced by possible temperature differences between the agitation methods (Briese, 2010). The EC meter was calibrated by NaCl solution (1000 $\mu S\ cm^{-1}$) (Cat. No. 2243-16, Ricca Chemical Company, Arlington, Texas) prior to each agitation time measurement.

Statistical analyses

Analysis of variance (ANOVA) and least significant difference (LSD) at $p \leq 0.05$ were performed using the PROC ANOVA procedure in SAS 9.2 (SAS Inc., Cary, NC) for agitation methods and equilibration time comparison. Regression analyses were used to determine relationship between $EC_{1:5}$ values and agitation time.

Equilibration time analyses

Times for equilibration were empirically determined from the relationships between $EC_{1:5}$ values and agitation times for the specific agitation methods. The maximum $EC_{1:5}$ value was assumed to be y_o obtained from the regression equation, from which then 95 and 98% of the maximum $EC_{1:5}$ was determined. Both 95 and 98% of equilibration were determined due to acceptable limits of error in methodology and to reduce the estimated time for equilibration. From the 95 and 98% $EC_{1:5}$ value, x (time) was determined from the equation of $y = y_0 + a/x + b/x^2$.

Results

Agitation methods

The data from each agitation method and time were fit using the regression model

$$y = y_0 + a/x + b/x^2 \qquad [5]$$

where, y is the modeled value of $EC_{1:5}$ (dS m^{-1}), y_0 is the extrapolated value of y at very large value of $EC_{1:5}$, x is agitation time (min), and a and b are coefficients. Coefficients of determination (r^2) values obtained from Eq. [5] ranged from 0.759 to 0.999, 0.683 to 0.996, and 0.474 to 0.997 for shaking plus centrifuging (shaking/centrifuging), shaking, and stirring, respectively, for all the 20 soils in our study (Table 7). Typical results for soils having EC_e values less than and greater than 4 dS m^{-1} were shown in Figure 1. For soils having an EC_e of less than 4 dS m^{-1}, the first three reading time points (10, 20, and 40 min) were removed and the remaining $EC_{1:5}$ values and the remaining corresponding agitation time of soils 1 through 5 were fit to Eq. [5].

Table 7. Regression equation ($y = y_0 + a/x^{\#} + b/x^2$) parameters for 20 soils (Table 6) across three agitation methods: shaking plus centrifuging (1), shaking (2), and stirring (3).

Soil ID	Treatment	Model parameter values			r^2
		y_0	a	b	
1[†]	1Shaking plus centrifuging[‡]	0.243	-21.04	1101	0.922
	2Shaking[§]	0.231	-20.47	1164	0.827
	3Stirring[¶]	0.197	-12.41	652.3	0.903
2[†]	1	0.407	-34.58	1823	0.869
	2	0.430	-42.73	2271	0.881
	3	0.337	-19.96	889.1	0.977
3[†]	1	0.279	-13.59	651.6	0.963
	2	0.275	-18.98	1009	0.936
	3	0.268	-28.55	1857	0.912
4[†]	1	0.531	-13.13	386.7	0.996
	2	0.533	-25.21	1382	0.957
	3	0.446	-0.523	-260.6	0.854
5[†]	1	0.407	-11.46	591.8	0.759
	2	0.413	-17.69	1026	0.683
	3	0.368	-4.232	197.1	0.474
6	1	2.434	-38.04	218.1	0.976
	2	2.225	-24.91	106.2	0.959
	3	1.443	-21.09	138.7	0.894
7	1	1.149	-1.986	0.891	0.907
	2	1.164	-1.684	-0.218	0.897
	3	1.069	-0.230	-12.43	0.904
8	1	2.237	-2.531	-8.240	0.840
	2	2.117	-4.222	-30.85	0.949
	3	1.957	-9.242	53.42	0.782
9	1	3.476	-28.46	137.2	0.996
	2	3.292	-16.77	-9.814	0.995
	3	2.575	-26.11	146.8	0.919
10	1	1.482	-8.989	60.85	0.932
	2	1.401	-3.977	-1.375	0.967
	3	1.295	-4.515	28.21	0.854
11	1	3.632	-27.68	127.1	0.995
	2	3.451	-24.85	102.5	0.996
	3	2.860	-19.27	96.95	0.962
12	1	3.946	-17.80	45.09	0.999
	2	3.709	-7.792	-7.634	0.992
	3	3.662	-18.96	11.05	0.997
13	1	3.329	-34.08	215.6	0.938
	2	3.061	-27.64	158.9	0.923
	3	2.439	-19.53	120.3	0.949
14	1	4.181	-11.39	52.79	0.904
	2	3.888	-0.869	-33.94	0.940
	3	3.692	-56.01	408.7	0.950
15	1	2.987	-7.007	15.31	0.875
	2	2.987	-7.007	15.31	0.875
	3	2.875	-12.02	56.08	0.951
16	1	4.841	-20.48	90.03	0.981
	2	4.465	-1.420	-154.3	0.994
	3	4.334	-29.10	172.4	0.987
17	1	4.081	-13.76	70.39	0.828
	2	1.164	-1.684	-0.218	0.928
	3	3.811	-13.91	87.30	0.879

Table 7. (Continued)

Soil ID	Treatment	Model parameter values			r^2
		y_0	a	b	
18	1	2.530	-0.529	-14.47	0.939
	2	2.543	-0.359	-50.88	0.967
	3	2.511	-3.722	2.842	0.972
19	1	4.717	-17.05	55.03	0.994
	2	4.491	-14.63	65.89	0.972
	3	4.018	-13.51	-26.05	0.993
20	1	5.883	-17.46	96.37	0.934
	2	5.569	-5.620	12.75	0.906
	3	5.506	-28.32	181.9	0.981

† Model parameters were determined after exclusion of the 10, 20, and 40 min time points.
‡ Within the treatment column, 1 represents the shaking plus centrifuging agitation method.
§ Within the treatment column, 2 represent the shaking agitation method.
¶ Within the treatment column, 3 represent the stirring agitation method.
# Unit of x in the equation is minutes.

Two distinct patterns were observed in the study that were separated by a soil EC_e value of 4 dS m^{-1}. For soils with EC_e values greater than 4 dS m^{-1}, data fit the model very well with r^2 values greater than 0.782 for all methods (Table 7 and Fig. 1A). However, for soils with EC_e values less than 4 dS m^{-1}, the r^2 values ranged from 0.474 to 0.996 when using the model across all the three agitation methods (Table 7 and Fig. 1B). Removal of the 10, 20, and 40 min time points, and corresponding $EC_{1:5}$ values at 10, 20, and 40 min time point for soils 1 through 5 (EC_e less than 4 dS m^{-1}) increased average r^2 from 0.833 to 0.902, 0.772 to 0.857, and 0.773 to 0.824 for shaking/centrifuging, shaking, and stirring, respectively, across the first five soils thus reducing model error. Although a quadratic model distributed the relationship better for soil with EC_e values less than 4 dS m^{-1}, the regression models of inverse second order (Eq. 5) was chosen for agitation methods and equilibration time comparisons across the overall 20 soils.

Differences between the three agitation methods (Fig. 1) were apparent. The mean value of $EC_{1:5}$ ranged from 0.18 to 5.61 dS m^{-1}, 0.17 to 5.45 dS m^{-1}, and 0.16 to 5.11 dS m^{-1} for shaking/centrifuging, shaking, and stirring, respectively (Table 8). Between the three agitation methods, 65% of soils were significantly different ($p \leq 0.05$) from each other. The shaking/centrifuging method was significantly different from stirring 100% of the time, and the shaking/centrifuging was significantly different from shaking 75% of the time. Of the shaking/centrifuging soils that were not significantly different ($p > 0.05$) from shaking, three of these soils had EC_e values less than 4 dS m^{-1} (soils 2, 3 and 4) (Table 8).

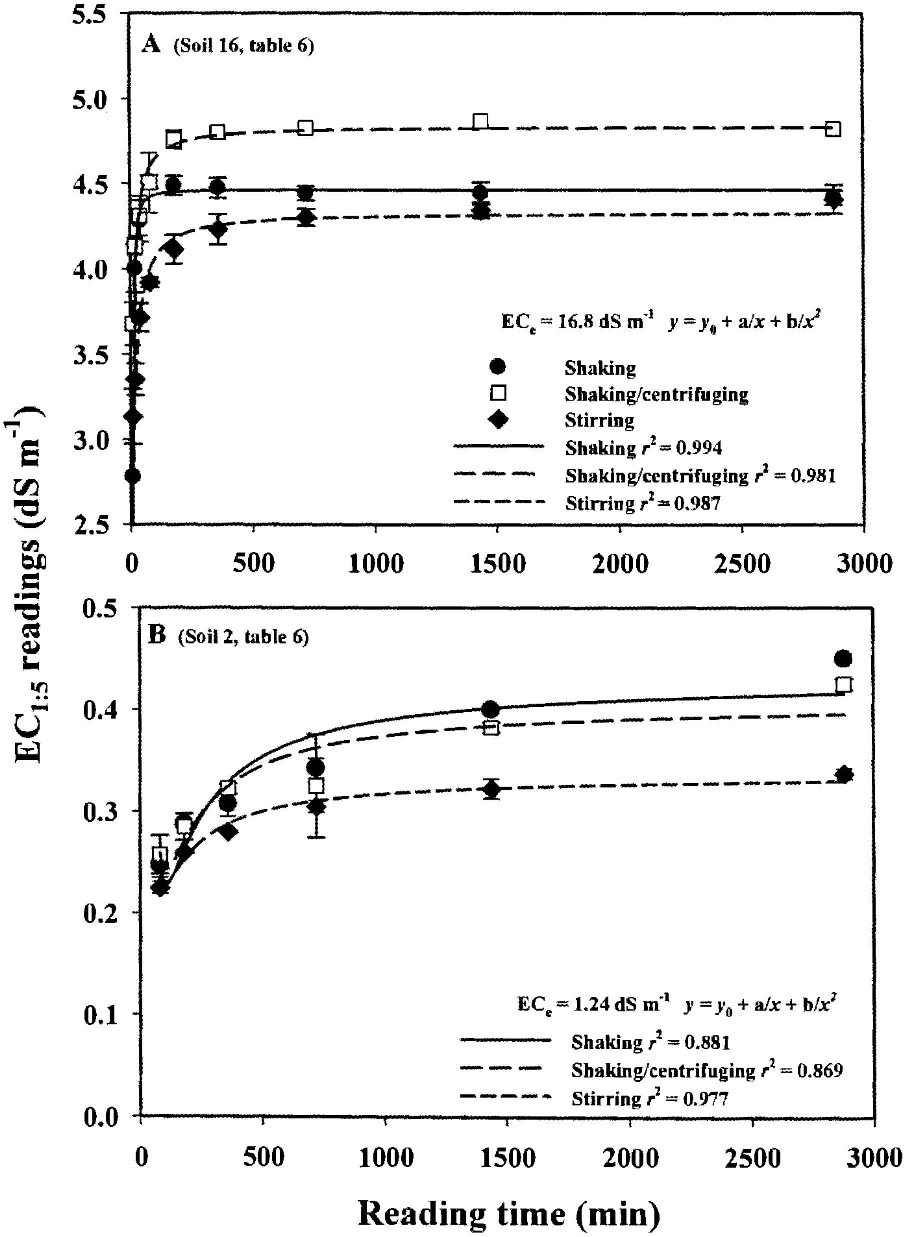

Figure 1. Relationships between $EC_{1:5}$ and agitation time for three agitation methods for two soils. "A" and "B" show representative examples of soils having EC_e values greater than and less than 4 dS m^{-1}, respectively. The vertical bar indicates standard deviation.

Table 8. Mean $EC_{1:5}$ and least significant difference values of soil samples used to compare the three agitation methods.

Soil	Agitation method			LSD[†]
	Shaking plus centrifuging	Shaking	Stirring	
	min			
1	0.18a[‡]	0.17b	0.16c	0.00
2	0.28a	0.28a	0.25b	0.01
3	0.22a	0.21ab	0.20b	0.01
4	0.45a	0.44a	0.42b	0.01
5	0.37a	0.35b	0.34c	0.01
6	1.92a	1.83b	1.18c	0.05
7	1.11b	1.13a	1.05c	0.02
8	2.17a	1.98b	1.83c	0.07
9	3.05a	2.91b	2.22c	0.05
10	1.36a	1.30b	1.23c	0.03
11	3.16a	3.01b	2.55c	0.05
12	3.58a	3.50b	3.21c	0.06
13	2.90a	2.69b	2.19c	0.06
14	4.01a	3.96a	3.06b	0.23
15	2.86a	2.85a	2.69b	0.05
16	4.52a	4.21b	3.95c	0.05
17	3.88a	3.67b	3.63b	0.08
18	2.49a	2.42b	2.42b	0.05
19	4.34a	4.20b	3.59c	0.06
20	5.61a	5.45b	5.11c	0.06

† LSD, least significant difference.

‡ Within rows, means followed by the same letter are not significantly different according to LSD (0.05).

Equilibration time

Due to the significant differences observed between the three agitation methods (Table 8), time for equilibration was determined individually for each of these methods. In general, time for equilibration of the 1:5 extract increased as salinity, (expressed by EC_e), decreased (Fig. 2). Times required for soils to reach 95 and 98% of equilibration were a function of the various levels of salinity in the soils across respective agitation methods (Fig. 2).

Estimated agitation time required for soils to reach 95 and 98% equilibration varied within the 20 soils within each method (Table 9). Equilibration time for soils having an

EC_e of less than 4 dS m^{-1} generally were significantly different ($p \leq 0.05$) than soils having EC_e values greater than 4 dS m^{-1}. This trend was displayed more often for shaking and shaking plus centrifuging (shaking/centrifuging) compared to the stirring method. For soils having EC_e values less than 4 dS m^{-1}, equilibration time was greater than 233 and 515 min (3.88 and 8.59 hrs) for 95 and 98% equilibration across the three methods, respectively.

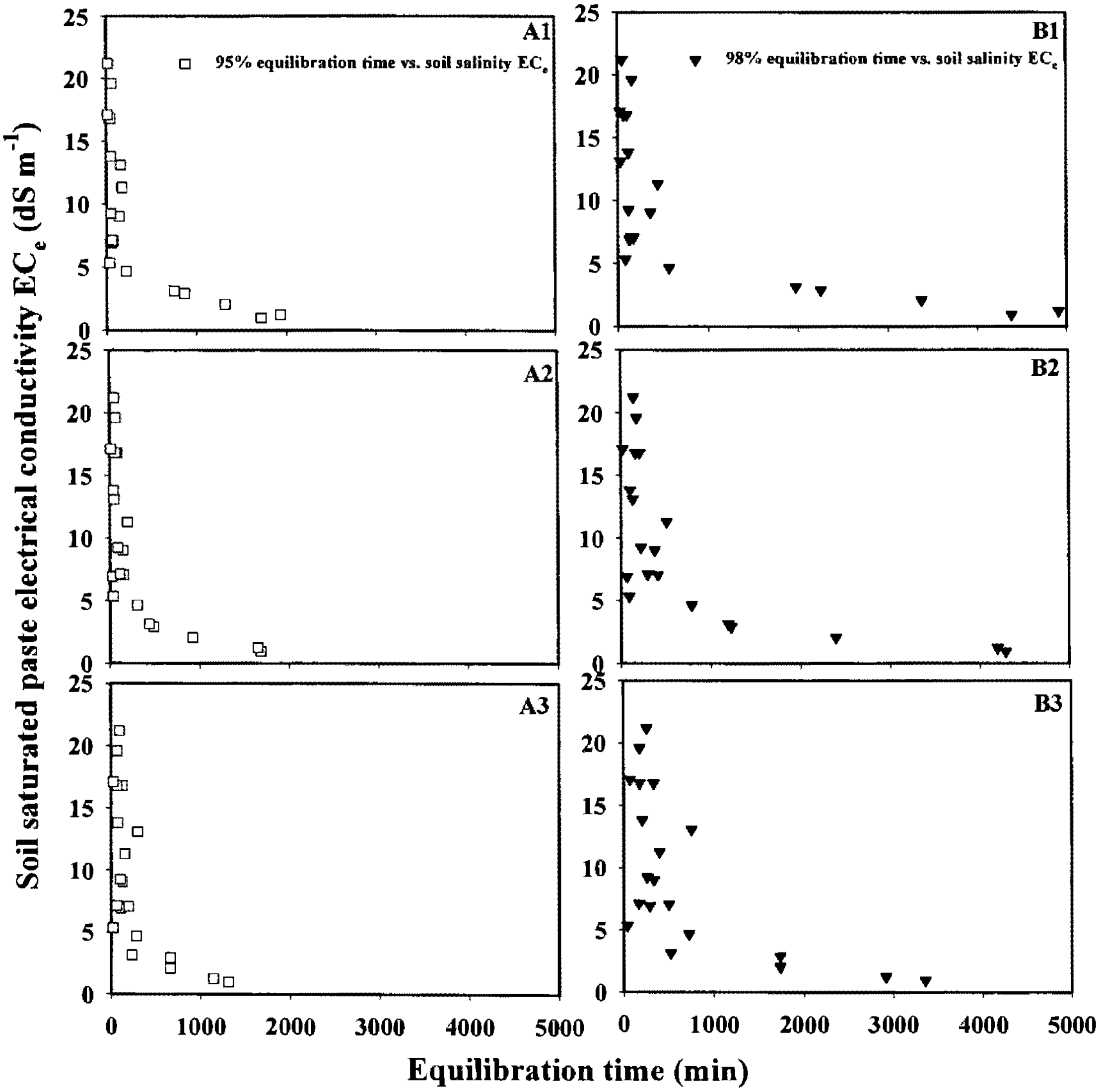

Figure 2. Time for 95% of equilibration in 1:5 soil:water extract for 20 soils across three different agitation methods: A1 (shaking), A2 (shaking plus centrifuging), and A3 (stirring), and 98% of equilibration across B1 (shaking), B2 (shaking plus centrifuging), and B3 (stirring).

Table 9. Estimated time required to reach 95 and 98% of equilibration for all the soils across three agitation methods: shaking plus centrifuging (shaking/centrifuging), shaking, and stirring.

Soil ID	Soil EC_e	Time for 98% of max $EC_{1:5}$			Time for 95% of max $EC_{1:5}$		
		Shaking/centrifuging	Shaking	Stirring	Shaking/centrifuging	Shaking	Stirring
	dS m^{-1}	min					
1§	0.96	4285a†	4378a	3356a	1681a	1716b	1307a
2§	1.24	4195a	4913a	2919b	1645a	1932a	1140b
3§	2.05	2385b	3371b	1737c	923b	1315c	662c
4§	2.91	1222c	2256c	1737c	487c	868d	662c
5§	3.14	1188c	1975c	515e	440c	752d	233ed
6	4.66	776d	555d	723d	307d	219e	285d
7	5.33	90hfg	80d	38j	34hg	33e	19i
8	6.91	66hg	126d	286hg	28hg	56e	110gh
9	7.06	404ef	176d	502fe	159ef	72e	197ef
10	7.12	296ehfg	144d	168jhi	114ehfg	59e	63ih
11	9.02	376efg	356d	332fheg	148efg	140e	130gh
12	9.23	223ehfg	115d	259hg	88ehfg	48e	103gh
13	11.3	506ed	444d	394feg	198ed	174e	154gf
14	13.1	132hfg	30d	751d	49hfg	161e	296d
15	13.8	103hfg	117d	204jhi	43hfg	47e	79ih
16	16.8	207ehfg	63d	330fhg	80ehfg	33e	128gh
17	16.8	163hfg	101d	176jhi	62hfg	44e	66ih
18	17.1	23h	26d	69ji	13h	16e	28i
19	19.6	177hfg	158d	180jhi	69hfg	60e	70ih
20	21.2	143hfg	49d	251hig	53hfg	19e	96gh
LSD‡	–	321	537	184	120	213	67

† Within columns, means followed by the same letter are not significantly different according to LSD (0.05).

‡ LSD, least significant difference.

§ The first five soils have soil EC_e less than 4 dS m^{-1}.

No significant differences ($p > 0.05$) for equilibration time were observed within the shaking method when the EC_e values were greater than 4 dS m^{-1}, indicating that as little as 26 min (0.43 hr) is needed to achieve 98% equilibration in shaking method (Table 9). The equilibration time for the 98% ranged from 23 to 776 min (0.38 to 12.9 hrs), 26 to 555 min (0.43 to 9.24 hrs), and 38 to 723 min (0.63 to 12.1 hrs) for shaking/centrifuging, shaking, and stirring, respectively, for soils having EC_e values greater than 4 dS m^{-1}. In contrast, for soils with EC_e values less than 4 dS m^{-1}, the average time for 98%

equilibration of (soil ID 1–5) was obtained by taking the average equilibration time of the first 5 soils in Table 9, and the values were about 2655, 3378, and 2053 min (44.3, 56.3, and 34.2 hrs) for shaking/centrifuging, shaking, and stirring, respectively. Generally, soils having the lowest EC_e values require significantly highest ($p \leq 0.05$) equilibration times.

The 95 and 98% of equilibration times had a similar trend, but as expected, less time was required to reach 95% equilibration for each soil across respective agitation methods (Table 9). For the shaking method, soils with EC_e values greater than 4 dS m^{-1} were not significantly different and had equilibration times ranging from 16 to 219 min (0.27 to 3.65 hrs). However, for soils with EC_e values less than 4 dS m^{-1}, equilibration times averaged about 1035, 1317, and 801 min (17.3, 21.9, and 13.3 hrs) for the shaking/centrifuging, shaking, and stirring methods, respectively. Considering the overall average equilibration time, the longest equilibration times was recognized for shaking/centrifuging and the shortest time was for the shaking. Across all methods, the equilibration time needed for obtaining 98% of equilibration was about 2.5 times of that needed for 95%.

Discussion

Although the low EC_e soils (soils 1–5; Table 6) fit a quadratic equation very well (data not shown), the data were fit to an inverse second order model because majority of soils (15 in our study) fit an inverse second order better so that comparison of soils and methods could be more easily accomplished with consistent models. Supporting data from VanderGheynst et al. (2004), who looked at changes in EC in 1:5, 1:6, 1:7, 1:10, and 1:12 dilution ratios for compost with time also showed that as time increased, EC of the stirred extract also increased ($EC_{1:7}$ of GGRS, a mixture of green waste, grape waste, and rice straw compost samples ranged from about 3.2 to 4 dS m^{-1}). From the VanderGheynst et al. (2004) study, there was not a significant difference in the EC values agitated for 30, 60, and 180 min, but EC was significantly different ($p \leq 0.05$) between 180 min (3 hrs) and 15 hrs of agitation time. Their reasons for this were assumed to be related with biofilm protection the diffusion of salts from interior of soil particles. Although the media from which salts were dissolved were different (soil vs. compost), the results shown in Figure 1 had a similar trend as those reported in VanderGheynst et al. (2004).

In our study, soils with low salinity (soils 1–5; Table 6) perform differently than the rest of soils with high salinity in Figure 1. The reasons are not clear, possibly related with soil chemical composition. For soils (soils 1–5; Table 6) with salinity less than 4 dS m^{-1}, the time points of 10, 20, and 40 min and the corresponding $EC_{1:5}$ values at those points were deleted for analysis, because we are more concerned with the equilibration time. The salts dissolved in water will undergo a process of equilibrium dissociation, and when sparingly soluble salts in our study are present, the dissociation equilibration is slowed down, which may affect salts absorbed on soil particles to go into solution (Freeze and Cherry, 1979). Therefore, exclusion of the first few time points (a short time period) will not influence the overall equilibration time development. Study from VanderGheynst et al. (2004) also state that presence of biofilm would not change the value of soil EC but affect the equilibration of salts in soil with water (VanderGheynst et al., 2004).

In our study, variance among agitation methods used to obtain 1:5 soil extracts lead to differences in $EC_{1:5}$ values. Significant differences in $EC_{1:5}$ values between the three agitation methods used in our study for the same soil suggest that comparison of EC across different methods should be done with caution. These differences may be due to the fact that salts can be more easily dissolved using mechanical shaking, which would tend to destroy micro-aggregates and expose entrained salts compared to stirring. In addition, mechanical shaking would also increase dissolution of salts because the concentration gradient between solid and liquid phases would be more dynamic (greater differences between partial pressures) compared to the stirring method where the zone around the salts would become saturated with ions (smaller differences between the partial pressures) and thus dissolution would be more limited.

Mineral dissolution may also lead to overestimation of EC and change in solute composition (Reitemeier, 1946). For example, in soils containing gypsum or calcite, the amount of calcium and sulfate that are dissolved can increase when the amount of water used in extraction is increased, while the concentration of the other ions such as chloride can decrease due to dilution since chloride salts are likely pre-dissolved due to their relatively high solubility (Al-Mustafa and Al-Omran, 1990; FAO, 1973; Hogg and Henry, 1984; Robbins and Wiegand, 1990). In addition, salt dissolution may be affected by biological activities in solution (VanderGheynst et al., 2004) by a reduction in diffusion of salts from particle interiors of particles due to the presence of biofilms. Although not determined for this study, the Great Groups of the soils used here indicate the presence of high levels of carbonate and sulfate salts (Table 6), which may then support the above statement.

The limits of 95 and 98% equilibration were used to determine time for equilibration due to acceptable error in methodology and to reduce the estimated time for equilibration. For example, for soil 2 (Table 6), which had an EC_e of less than 4 dS m^{-1}, the time for equilibration was greater than 48 hr (Table 9), which is not generally feasible for commercial soil testing laboratories. However, for soils having EC_e values greater than 4 dS m^{-1}, equilibration times of less than 8 hr could be achieved, which is more appropriate for testing laboratories. The dilemma is that many soils that get analyzed will have EC_e values less than 4 dS m^{-1} and the laboratory will not likely know the EC_e prior to analysis. Consequently, analysis errors based on time for equilibration may be unavoidable or the testing laboratory will have to analyze all samples for 24 hrs or longer or potentially rerun samples to achieve 95 or 98% equilibration. However, there is no loss of confidence in $EC_{1:5}$ values when extraction times appropriate for soils having EC_e values less than 4 dS m^{-1} are used (Table 9).

Conclusions

The $EC_{1:5}$ of soils were affected by both agitation methods and agitation time. Significant differences existed within three agitation methods ($p < 0.0001$). In general, shaking/centrifuging resulted in the highest $EC_{1:5}$ values and stirring resulted in the lowest. Selecting an agitation method will depend on equipment availability and the reporting of data should include a detailed description of methods so that $EC_{1:5}$ values can be compared and contrasted between studies.

Times for equilibration were a function of soil salinity. The 95 and 98% equilibration times were all faster for soils having EC_e values greater than 4 dS m^{-1} compared to soils less than 4 dS m^{-1}. Many factors can influence equilibration including salt concentration and species, protection of salts through entrainment or organic matter, and agitation method.

A considerable body of information has been built regarding soil $EC_{1:5}$ and the suitability of soils for crop production based on this data. Globally, numerous extraction ratios are used, which are likely a function of available equipment and time. The factors listed above and the variation in $EC_{1:5}$ values determined from this study indicate that the establishment of a global $EC_{1:5}$ standard method should be considered which would help minimize the variability between laboratories and published results.

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PAPER 2. RELATING THE VALUE OF $EC_{1:5}$ TO EC_e OF THE SATURATED PASTE EXTRACT

Abstract

Because of simple and fast in preparation many commercial laboratories appraise soil salinity from $EC_{1:5}$ measurements. However, the influence of salinity on plant growth is mainly based on saturated paste extract electrical conductivity (EC_e), so it is necessary to convert $EC_{1:5}$ to EC_e in order to assess plant response. The objectives of this research were to develop predictive models to convert $EC_{1:5}$ data to EC_e based on four different 1:5 extraction methods: 1) shaking, 2) shaking plus centrifuging (shaking/centrifuging), 3) stirring, and 4) a USDA-NRCS equilibration technique. Soils evaluated from this study were from north central North Dakota, USA, and 100 samples were used for each agitation method to develop the relationship between EC_e and $EC_{1:5}$. Through regression analysis, the value of EC_e was highly correlated with $EC_{1:5}$ ($p < 0.0001$) across four agitation methods using non-transformed, $\log_{10}$-transformed, and dilution ratio models. The values of r^2 were greatly improved to about 0.87 using $\log_{10}$-transformation compared to other two models (r^2 values of about 0.69). Since agitation methods were determined to be highly correlated with each other, any regression model determined under the four agitation methods were applicable for the estimation of EC_e from another method. Estimating EC_e from $EC_{1:5}$ using different agitation methods after model development is a useful approach to estimate EC_e without the need for saturation paste extracts.

Introduction

Soil salinity is a major factor determining the suitability of soils for crop production (Kruse et al., 1990). Electrical conductivity (EC) measurement of saturated paste extracts are generally used for evaluating soil salinity, where this extract essentially simulates the environment of naturally occurring moisture-saturated soil. Results from these extractions are thought to be the best indicator of plant response to salinity (Rhoades et al., 1989; USDA, 1954). However, 1:5 soil:water mass ratio extractions ($EC_{1:5}$) are easier to prepare, save time and money compared to saturation extracts (Khorsandi and Yazdi, 2007), and are commonly used to determine soil salinity in Australia, China and Central Asia (Committee of Saline Soils of Soil Science Society of China, 1989; Rayment and Higginson, 1992; Shirokova et al., 2000).

Predicting EC_e from $EC_{1:5}$ extractions has been accomplished for different regions including China, Iran, and Australia (Chi and Wang, 2010; Khorsandi and Yazdi, 2011; Slavich and Petterson, 1993; Sumner and Naidu, 1998). Parameters that have been considered that may influence the relationship between EC_e and $EC_{1:5}$ include soil texture, types of salts present in the soil, and water content of the saturated paste soil (Al-Mustafa and Al-Omran, 1990; Chi and Wang, 2010; Khorsandi and Yazdi, 2007; Sonmez et al., 2008). In general, predictions of EC_e from $EC_{1:5}$ have been very good with coefficient of determination (r^2) values greater than 0.91 (Khorsandi and Yazdi, 2007). However, several different extraction methods and equilibration times have been used with the 1:5 ratio without comparison between methods. Thus, uncertainty exists when applying these models developed from different 1:5 extraction methods to data derived from other extraction methods.

Due to different 1:5 sample preparation methods, the relationships between EC_e and $EC_{1:5}$ that have already been established varied between the different studies. Previous studies have generally not compared various 1:5 extraction methods and equilibration times. For example, U.S. Salinity Laboratory Staff (1954) used mechanical shaking as agitation method and 80 min for equilibration, while Visconti et al. (2010) used mechanical shaking as the agitation method and 24 hr for equilibration. In addition, the relationships for converting $EC_{1:5}$ to EC_e are empirical models and should be developed for more regions (Al-Mustafa and Al-Omran, 1990; Landon, 1991; Shaw, 1994; Slavich and Petterson, 1993). No research was found predicting EC_e from $EC_{1:5}$ that took into account the agitation methods, and to date no EC_e to $EC_{1:5}$ relationships have been conducted on soils from Northern Great Plains. The objectives of this research were to develop predictive models to convert $EC_{1:5}$ data to EC_e based on four different 1:5 extraction methods: 1) shaking, 2) shaking plus centrifuging (shaking/centrifuging), 3) stirring, and 4) a USDA-NRCS equilibration technique from Soil Survey Staff (2011).

Materials and Methods

Soil samples

Soil samples used in this study (n = 100) were from Benson and Ramsey counties in North Dakota, USA (approximately 48°11′34″–48°33′36″ N, 99°59′52″–99°41′88″ W). Samples were from 0–15, 15–30 and 30–60 cm depths from varying landscape positions. These samples were initially collected for a larger study looking at changes in soil EC due to irrigation (Steele and Hopkins, 2009). All samples were Mollisols but had different suborder classifications and textural classes ranging from sandy to fine-textured. Each sample was air-dried, ground to pass through a 2 mm sieve, and stored in plastic bags until analysis. Saturated pastes were made as part of the prior study following the methods outlined by U.S. Salinity Laboratory Staff (1954) and electrical conductivity (EC_e) was determined on each extract. Hamerly (fine-loamy, mixed, superactive, frigid Aeric Calciaquolls), Fargo (fine, smectitic, frigid, Typic Epiaquerts), and Embden (coarse-loamy, mixed, superactive, frigid Pachic Hapludolls) soils were used as "check" soils during EC_e determination. The Hamerly, Fargo, and Embden were analyzed 47, 56, and 48 times and have average EC_e values and coefficients of variation (CV) 0.9, 0.6, and 1.5 dS m^{-1}, and 7.2, 8.2, and 15%, respectively.

Extraction preparation (1:5 ratio)

The agitation methods used in this study were shaking, shaking plus centrifuging (shaking/centrifuging), stirring, and an updated NRCS method (Soil Survey Staff, 2011) which will be described in the following. The soil suspension was obtained by adding 35 mL of ultra-pure water to 7 g of soil for shaking, shaking/centrifuging, and stirring methods. The soil suspensions were shaken for 8 hrs using a mechanical shaker (132 rev min^{-1}) for the shaking and shaking/centrifuging methods followed by 5 min of settling or centrifuging at relative centrifuge force (RCF) of 730×g, respectively, followed by the measurement of EC on each supernatant using a conductivity probe (Sension 378; Hach Co., Loveland, CO, USA). For the stirring method, the suspension was stirred using a glass rod for 10 sec every 2 hr. After the final stirring the slurry was allowed to settle for 5 min followed by EC measurement. The 8 hr of equilibration was determined by regression analysis of $EC_{1:5}$ values and agitation times, where equilibration times were determined by calculation from the regression models and comparison by least significant difference (LSD) (paper 1). For the NRCS method, the soil suspension (20 g soil:100 mL ultra-pure water) was maintained at room temperature for 23 hrs followed by agitation by a mechanical shaker (132 rev min^{-1}) for 1 hr. After agitation, the soil solution was filtered through #2 Whatman filter paper (Cat. No. 1002-110, Whatman International Ltd Maidstone, England) into a 100 mL polyethylene bottles from which EC of the solution was determined. All agitation methods and EC readings were done at 25 °C.

For each agitation method, a "check" soil, and one blank (ultra-pure water only) were analyzed following the respective agitation and settling criteria. Although temperature may affect EC readings, the EC meter used for this study was determined to not be influenced by possible temperature differences between the agitation methods (Briese, 2010). The EC meter was calibrated with a NaCl solution (1000 μS cm^{-1}) (Cat. No. 2243-16, Ricca Chemical Company, Arlington, Texas) prior to each measurement.

Data handling and statistical analysis

The relationships between values of EC_e and $EC_{1:5}$ were determined using non-transformed and $\log_{10}$-transformed data. In addition, assuming the 1:5 soil water extract to be a simple 1:5 dilution of a saturated paste extract (Slavich and Petterson, 1993), the EC_e was plotted against the value of $EC_{1:5}$ multiplied by 5 ($5 \times EC_{1:5}$). Correlations of $EC_{1:5}$ values between the different methods of agitation were conducted to determine the errors associated with the different agitation methods.

Analysis of variance (ANOVA) was performed using the PROC ANOVA procedure in SAS 9.2 (SAS Inc., Cary, NC). Regression analysis was used to determine the relationship between values of $EC_{1:5}$ and EC_e, to compare the measured EC_e and EC_e values estimated from the regression model, and to determine the correlation of $EC_{1:5}$ values between different agitation methods. Significance was determined at $p \leq 0.05$.

Results

Relationship between $EC_{1:5}$ and EC_e

Non-transformed

The EC_e versus $EC_{1:5}$ for the four different agitation methods are presented in Figure 3. Highly significant linear relationships ($p < 0.0001$) existed within each of the four agitation methods. The slopes of the regression lines were 2.74, 2.74, 3.42, and 2.86 for shaking, shaking/centrifuging, stirring, and the NRCS equilibration technique method, respectively, with r^2 values of 0.66, 0.67, 0.75, and 0.67, respectively. All methods had similar slopes and intercepts except for the stirring method. For each method, when considering only data with EC_e value less than 4 dS m^{-1}, the data were very linearly correlated ($r^2 > 0.89$), but below the regression line of the overall relationship of EC_e and $EC_{1:5}$ (Fig. 3).

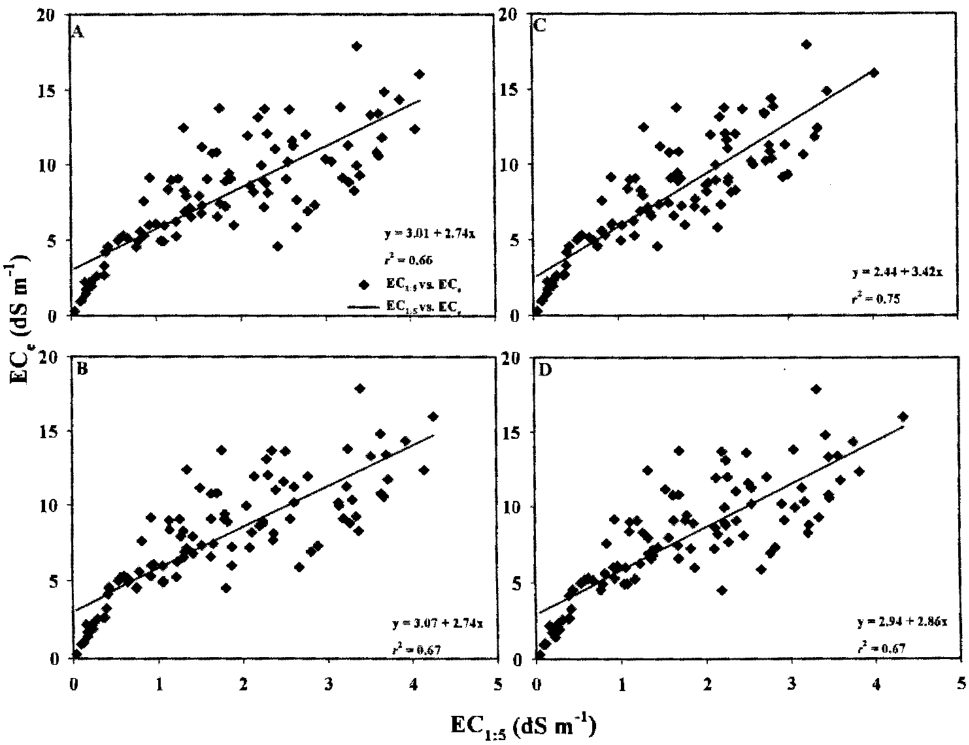

Figure 3. Relationships between $EC_{1:5}$ and EC_e for 100 soil samples for four methods: A (shaking), B (shaking/centrifuging), C (stirring), and D (NRCS).

Log_{10}-transformed

Highly significant linear relationships ($p < 0.0001$) were found between values of $\log_{10}$-transformed EC_e and $EC_{1:5}$ (Fig. 4). After $\log_{10}$-transformation, data clustered around regression lines and r^2 values were greatly improved to between 0.86 and 0.89 for all agitation methods. The simple linear regression models in shaking, shaking/centrifuging, and NRCS method were again very similar, but the stirring method was slightly different (Fig. 4). Each relationship was significant, which indicates that all agitation methods provided satisfactory prediction of EC_e from $EC_{1:5}$ based on $\log_{10}$-transformed data.

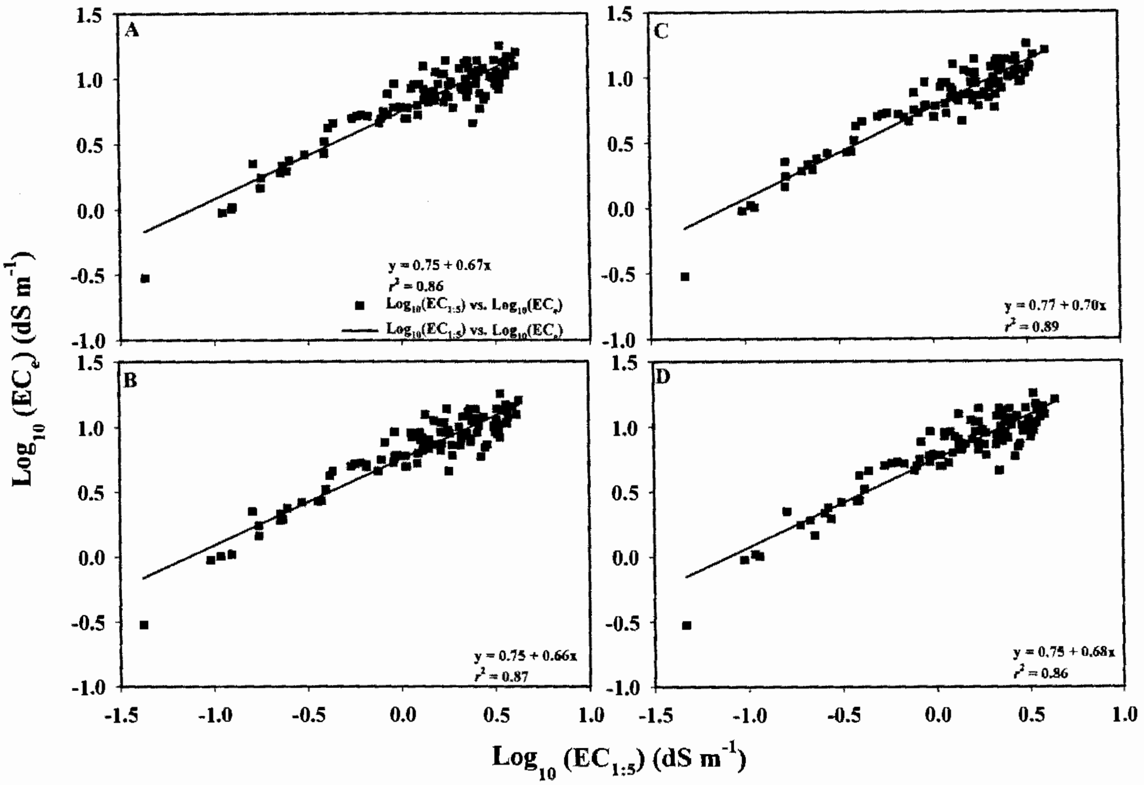

Figure 4. Relationship between $\log_{10}(EC_{1:5})$ and $\log_{10}(EC_e)$ for 100 samples for four methods: A (shaking), B(shaking/centrifuging), C (stirring), and D (NRCS).

Comparison of values of coefficient of determination (r^2) and standard error (SE) in the regression models established before and after EC (both EC_e and $EC_{1:5}$) values were $\log_{10}$-transformed indicates that the $\log_{10}$-transformation significantly improved the relationships (Table 10). The factors (slope and intercept) after $\log_{10}$-transformation also indicate a fair degree of uniformity in the relationship between EC_e and $EC_{1:5}$, where the slopes were 0.75 across the three shaking methods and their intercepts were identical (Fig. 4), while the intercepts and slopes for non-transformed data slightly varied (Fig. 3).

Table 10. Coefficient of determination (r^2), standard error (SE) and regression equations describing the relationship of electrical conductivity between 1:5 and saturated paste extract for 100 soil samples.

Agitation methods	Regression equation	r^2	p	SE[†]
Non-transformed				
Shaking	$y^{‡} = 3.01 + 2.74x^{§}$	0.66	< 0.0001	2.2
Shaking/centrifuging	$y = 3.07 + 2.74x$	0.67	< 0.0001	2.2
Stirring	$y = 2.44 + 3.42x$	0.75	< 0.0001	1.9
NRCS method	$y = 2.96 + 2.86x$	0.67	< 0.0001	2.2
Log_{10}-transformed				
Shaking	$w^{¶} = 0.67 + 0.75z^{\#}$	0.86	< 0.0001	0.12
Shaking/centrifuging	$w = 0.66 + 0.75z$	0.87	< 0.0001	0.11
Stirring	$w = 0.70 + 0.77z$	0.89	< 0.0001	0.10
NRCS method	$w = 0.68 + 0.75z$	0.86	< 0.0001	0.11

† SE, Standard error of estimate.
‡ y, EC_e.
§ x, $EC_{1:5}$.
¶ w, $\log_{10}(EC_e)$.
# z, $\log_{10}(EC_{1:5})$; $EC_e = 10^w$.

Dilution ratio method

Predicting EC_e from $5\times EC_{1:5}$ was significant ($p \leq 0.05$), but the regression line deviated from 1:1 line (Fig. 5). When the soil had a $5\times EC_{1:5}$ value of about 6.7 dS m^{-1}, the predictive EC_e value from the regression line was almost the same as EC_e determined from the 1:1 line. However, the predicted EC_e value was overestimated when soils having $5\times EC_{1:5}$ value less than 6.7 dS m^{-1}, and underestimated when soils having $5\times EC_{1:5}$ value greater than 6.7 dS m^{-1}, compared to the estimation accuracy from 1:1 line. For example, for a soil with 5×$EC_{1:5}$ value of 15 dS m^{-1} determined from the shaking method, the EC_e value estimated from regression equation was about 11.3 dS m^{-1}, which was lower than the theoretical value estimated from 1:1 line. In general, when a soil 5×$EC_{1:5}$ value was greater than about 6.7 dS m^{-1}, differences between predicted and theoretical EC_e value were greater than for soils having 5×$EC_{1:5}$ values less than 6.7 dS m^{-1} (Fig. 5).

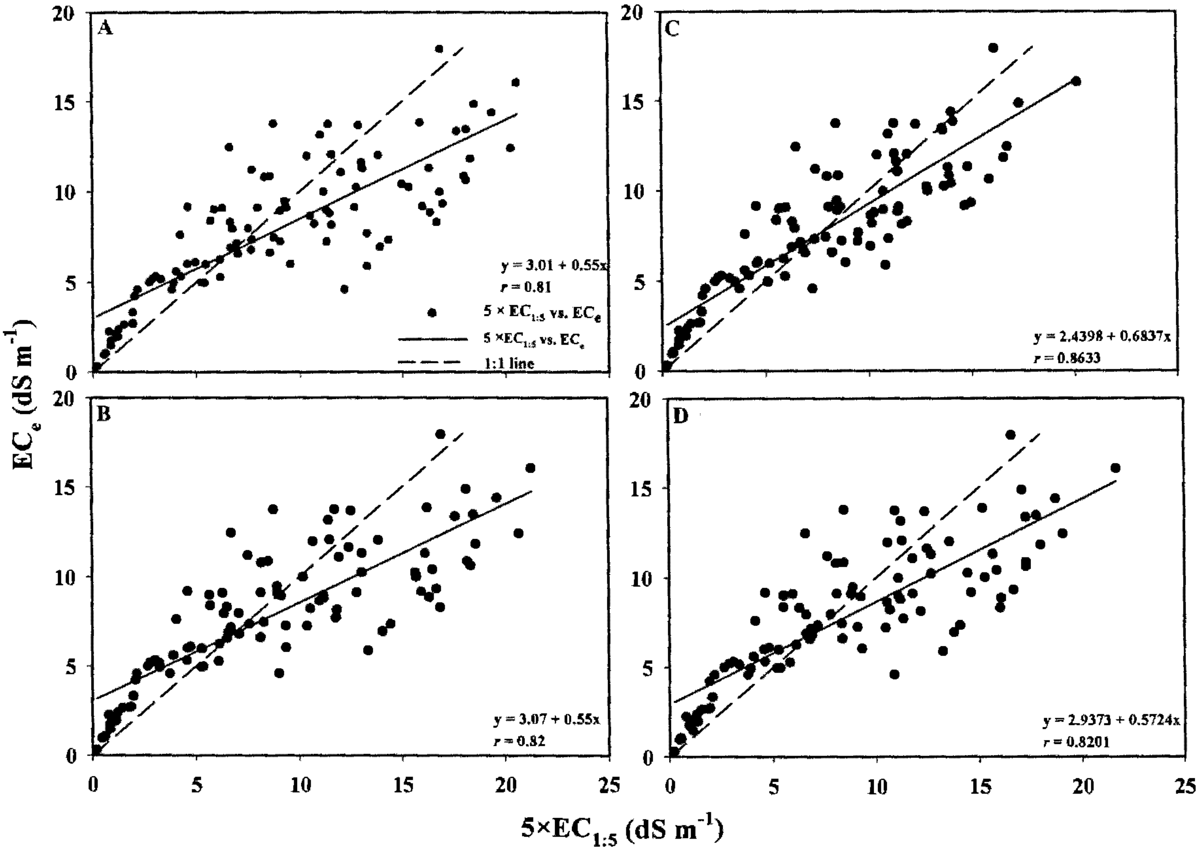

Figure 5. Relationship between actual EC_e and 5×$EC_{1:5}$ for agitation method of A (shaking), B (shaking/centrifuging), C (stirring), and D (NRCS).

Since different models were used to build the relationship between EC_e and $EC_{1:5}$, preference of models was determined by comparing estimated EC_e value determined from each model and actual EC_e value determined from saturated paste extract. Relative percentage difference (RPD) between actual EC_e and 5×$EC_{1:5}$ values were compared to RPD that were calculated from $\log_{10}$-transformed regression models and non-transformed relationship models (Table 11). Comparison of the RPD values indicate that estimation of EC_e from $EC_{1:5}$ from any model depended on the level of salinity, so for soil salinity (EC_e) levels less than 12 dS m^{-1}, log_{10}-transformed models generally predicted EC_e from $EC_{1:5}$ better than if salinity was greater than 12 dS m^{-1}. This relationship was held for each of the agitation methods (Table 11). However, for soils having EC_e values greater than 12 dS m^{-1}, all models have identical accuracy to estimate EC_e with identical average RPD values. In general, log_{10}-transformed models had a better function in predicting of EC_e value from the value of $EC_{1:5}$ for each of the agitation method taking into account the average RPD values. Overall, all the models could result in accurate EC_e estimation in the specific soil salinity range.

Measured EC_e and predicted EC_e value from log_{10}-transformation

Predicted EC_e values from the log_{10}-transformed regression models were validated against corresponding actual EC_e values determined from the saturated paste extract (Fig. 6). Highly significant relationships ($p < 0.0001$) existed between actual EC_e and that estimated from the log_{10}-transformed regression models across all agitation methods. Theoretically, there is a 1:1 relationship between actual and predicted EC_e value if the model between EC_e and $EC_{1:5}$ is accurate. Here, the validation lines between actual and predicted EC_e were close to the 1:1 reference line for all agitation methods, which indicates that the model accurately predicted EC_e, but variation still existed. The regression coefficient of regression lines were 0.82, 0.82, 0.84, and 0.82 for shaking, shaking/centrifuging, stirring, and NRCS methods, respectively, which were lower than theoretical value of 1 (Fig. 6).

Table 11. Statistics for relative percentage difference (RPD) between actual EC_e and EC_e values estimated from dilution (A), non-transformed (B), and $\log_{10}$-transformed models (C) across four agitation methods.

EC_e range	Shaking			Shaking/centrifuging			Stirring			NRCS		
	A†	B‡	C§	A	B	C	A	B	C	A	B	C
$dS\ m^{-1}$	------------------------------------						RPD					------------------------------------
0—2												
Mean	48.9	95.0	22.9	54.2	95.8	24.7	55.7	82.8	20.9	48.6	94.2	24.4
Median	49.5	93.4	11.2	51.4	94.5	21.0	58.9	80.2	15.0	56.3	93.2	17.6
Minimum	64.5	61.0	9.00	35.0	61.6	1.00	21.0	48.4	1.00	25.0	59.9	3.00
Maximum	32.3	165	79.0	68.0	166	80.0	71.0	159	81.0	67.0	165	80.0
2—4												
Mean	54.6	39.7	10.2	57.6	40.5	9.34	59.3	28.5	10.8	52.3	39.3	9.47
Median	53.0	41.7	9.74	56.6	41.1	6.87	61.7	30.9	8.15	50.8	40.4	6.80
Minimum	31.2	20.9	2.00	36.0	22.6	2.00	38.0	12.8	6.00	32.0	21.9	2.00
Maximum	93.8	51.2	30.0	95.0	52.3	29.0	92.0	38.5	29.0	95.0	51.7	32.0
4—8												
Mean	32.7	15.7	20.6	32.2	14.9	19.8	29.0	15.5	19.1	31.1	15.4	19.8
Median	22.0	9.75	16.1	25.6	11.3	16.4	22.7	14.7	18.0	23.1	11.2	15.6
Minimum	1.00	0.71	0.00	1.00	0.01	0.00	1.00	0.29	1.00	2.00	0.15	0.00
Maximum	91.0	71.7	76.0	78.0	55.5	59.0	73.0	50.6	53.0	82.0	66.8	70.0
8—12												
Mean	31.5	19.3	19.6	31.6	19.8	20.1	25.9	18.2	17.8	30.7	19.3	19.8
Median	33.0	15.1	16.5	33.8	16.0	17.3	25.3	14.3	15.3	28.6	16.3	16.9
Minimum	1.00	1.81	3.00	2.00	0.09	4.00	2.00	1.82	1.00	3.00	0.13	3.00
Maximum	67.0	49.0	52.0	68.0	48.1	53.0	66.0	26.5	49.0	65.0	32.9	53.0
> 12												
Mean	24.4	24.1	20.8	24.7	23.3	21.3	16.8	21.3	20.1	24.0	23.4	21.2
Median	22.2	16.5	14.7	20.0	15.0	14.6	10.4	16.9	14.4	22.6	17.3	14.4
Minimum	4.00	4.81	1.00	5.00	1.72	1.00	1.00	1.10	3.00	6.00	2.44	1.00
Maximum	61.0	60.5	58.0	59.0	59.1	58.0	63.0	57.4	55.0	61.0	3.90	59.0

† Model A, dilution ratio model.

‡ Model B, non-transformed model between $EC_{1:5}$ and EC_e.

§ Model C, $\log_{10}$-transformed model.

¶ RPD, relative percentage difference = |(measured EC_e – predicted EC_e)/ [(measured EC_e + predicted EC_e)/2]| × 100.

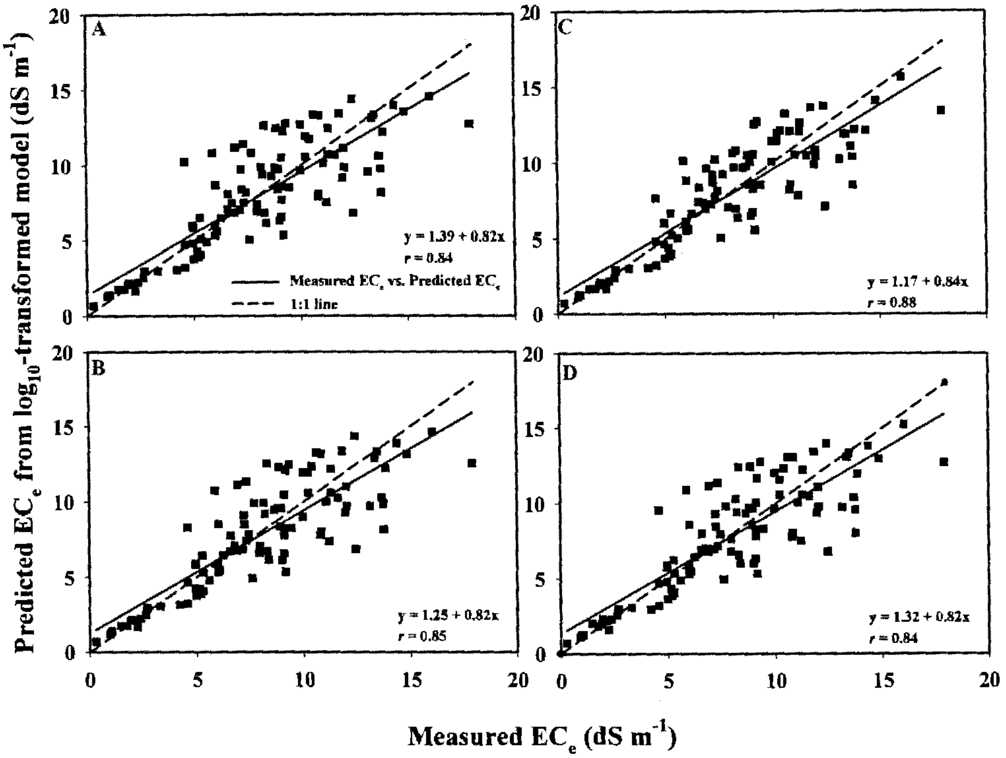

Figure 6. Validation of relationship between measured EC$_e$ and estimated EC$_e$ from log$_{10}$-transformed model when compared with 1:1 regression line for A (shaking), B (shaking/centrifuging), C (stirring), and D (NRCS).

Correlation of EC$_{1:5}$ values within different agitation methods

Due to the fact that agitation method would not affect the relationship when predicting EC$_e$ from EC$_{1:5}$, the EC$_{1:5}$ values across different agitation methods were plotted and were determined to be highly correlated with each other ($p < 0.0001$) (Table 12). Overall, the correlations between the three shaking methods were high ($r \geq 0.98$) and regression coefficients (b) were close to 1. The root mean square error (RMSE) values were higher when EC$_{1:5}$ values obtained from any of the shaking methods were plotted against the stirring method than when the three shaking methods were compared (Table 12). High correlations of EC$_{1:5}$ values within the three shaking methods indicate that any regression model established under different shaking methods would be applicable for the estimation of EC$_{1:5}$ from another shaking method.

Table 12. Statistics for correlation between $EC_{1:5}$ values within different agitation methods.

Parameter	A†	B‡	C§	D¶	E#	F††
b‡‡	1.01	0.83	0.96	0.83	0.96	1.11
r	0.99	0.98	0.99	0.98	0.99	0.98
RMSE§§	0.10	0.20	0.08	0.19	0.08	0.21
p	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001

† A, shaking vs. shaking/centrifuging.

‡ B, shaking vs. stirring.

§ C, shaking vs. NRCS equilibration technique.

¶ D, shaking/centrifuging vs. stirring.

# E, shaking/centrifuging vs. NRCS equilibration technique.

†† F, stirring vs. NRCS equilibration technique.

‡‡ b, slope of regression equation established by plotting $EC_{1:5}$ values between different agitation methods.

§§ RMSE, root mean square errors = $[\Sigma \text{ (predicted N} - \text{measured N)}^2 / (n - 1)]^{1/2}$.

Discussion

The regression coefficients (Fig. 3) in this study were substantially different from those reported in other papers where regression equations were used to convert values of $EC_{1:5}$ to EC_e (Chi and Wang, 2010; Loveday et al., 1972; Sonmez et al., 2008). The regression coefficient from these other studies ranged from 6 (Loveday et al., 1972) to 11 (Chi and Wang, 2010), and were all higher than values determined in this study. Also, the values of r and r^2 determined from the non-transformed and dilution ratio method models in this study (Fig. 3 and 5) were significantly lower than r and r^2 values found in other studies (Khorsandi and Yazdi, 2007; Khorsandi and Yazdi, 2011; Slavich and Petterson, 1993). Irrespective, the regression equations from these studies were also significant for estimation of EC_e from $EC_{1:5}$ data, which indicates that geographic variability (texture, types of salts) and agitation method and time play very important roles in estimating EC_e from $EC_{1:5}$ data.

When salinity values were less than 4 dS m^{-1}, the data were below the regression line of the overall relationship between EC_e and $EC_{1:5}$ for all methods when predicting EC_e

from $EC_{1:5}$ (Fig. 3), but they were linearly correlated. Agarwal et al. (1961), who examined EC_e-$EC_{1:5}$ relationships for low salinity (CO_3-HCO_3) and high salinity (CO_3-HCO_3 and SO_4-Cl) soils, indicated that the relationship between EC_e and $EC_{1:5}$ was separate for two salinity ranges where the data best fit curvilinear and linear models, respectively. Although it is not clear in this study why soils with salinity less than 4 dS m^{-1} exhibited a different trend, the data presented in Figure 3 had some similarity to that reported in Agarwal et al. (1961). Here, the possibility of two salinity ranges exist, one with EC_e less than 4 dS m^{-1}, and another with EC_e greater than 4 dS m^{-1}, being assessed after 8 hr of agitation, which was the equilibration time used in this study. Visconti et al. (2011) also found EC_e and $EC_{1:5}$ values were linearly related when soil $EC_{1:5}$ values were lower than about 1 dS m^{-1}, which was different from the random distribution of the EC_e and $EC_{1:5}$ relationships that characterized higher salinity values. Another possible reason is that soils having salinity less than 4 dS m^{-1} require more than 8 hr for equilibration as shown in paper 1, so the $EC_{1:5}$ values determined after 8 hr here in this study are lower than that expected. The 8 hr equilibration time used here attempts to best mimic conditions of soil testing laboratories (i.e. normal working day length). The data could have been parsed out and separate predictive models could have been developed, but the $\log_{10}$-transformed models described the data very well and thus further investigations were not pursued (Fig. 4).

Scattered data presented in Figures 3 and 5 are not a result from agitation methods, but from soil EC_e values. Good correlation of $EC_{1:5}$ values existed between different agitation methods ($r \geq 0.98$), which indicate that agitation methods would not influence the data distribution. Supporting data from $\log_{10}$-transformed models also provides evidence that data distribution was independent of agitation method (Fig. 4). The three check soils

(Hamerly, Fargo and Embden) used during EC_e determination exhibited an increase in CV from 7.2 to 15% with the increase in soil EC_e values, which further explains that the data scattering in Figures 3 and 5 were reasonable, especially when soil EC_e values are greater than 4 dS m^{-1}.

The $\log_{10}$-transformed models yield significant ($p \leq 0.05$) and better relationships in predicting EC_e (Table 10 and 11). The relationships here between measured and fitted EC_e value using $\log_{10}$-transformed regression line had slight deviation from the reference 1:1 line (Fig. 6), but were still similar to the results of Khorsandi and Yazdi (2011) where the linear regression lines between observed and predicted EC_e values were similar to the 1:1 line.

The difference of models between EC_e and $EC_{1:5}$ developed for the current study and those reported in other studies are most likely due to soil properties and salt composition of soils from different regions (USDA, 1954). Type of salts present in soils can result in different relationships between EC_e and $EC_{1:5}$ based on their salt solubility, rate of reaction and kinetics. Minerals of soils contact with water, dissolute until equilibration concentrations are reached in water. For example, solubility of gypsum at pH 7 is 2100 mg L^{-1} which is lower than halite, but high salt content indicate an increasing solubility due to decreasing of activity coefficient by ionic strength, therefore, sparingly soluble salts such as gypsum will affect the solution ability to conduct current and then EC value (Freeze and Cherry, 1979). Khorsandi and Yazdi (2007) also reported differences in regression equations, particularly between gypsiferous and non-gypsiferous soils. Addition of water beyond that used for saturated paste preparation would increase the dissolution of sparingly soluble salts, but additional water would also result in the fast dilution of chloride salts which have already dissolved in solution because of high solubility (Al-Mustafa and Al-Omran, 1990).

Conclusions

Significant relationships exist between EC values measured in saturated paste extract and 1:5 soil to water extract ($p < 0.0001$). It is reasonable to estimate EC_e from $EC_{1:5}$ with the developed regression models. Different agitation methods for preparing 1:5 extracts result in slightly different regression equations when converting $EC_{1:5}$ to EC_e. However, the correlations between four agitation methods is high ($r \geq 0.98$) and have similar regression equations, which indicates that the relationship between agitation methods is independent, therefore, any regression model established under different methods would be applicable for predicting EC_e from another method. In general, the $\log_{10}$-transformed model yielded better relationships between EC_e and $EC_{1:5}$ compared to non-transformed and dilution ratio models. Although the $\log_{10}$-transformed relationships would be recommended from this study, the relative variability of EC_e appears to be inherently greater than the electrical conductivity of $EC_{1:5}$.

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GENERAL CONCLUSIONS

Soil $EC_{1:5}$ measurement was affected by both agitation methods and agitation time. Significant differences existed within the three agitation methods (shaking/centrifuging, shaking, and stirring) ($p < 0.0001$), and in general, shaking plus centrifuging (shaking/centrifuging) resulted in the highest, and stirring resulted in the lowest $EC_{1:5}$ values. Selecting an agitation method will depend on the availability of equipment and the reporting of data should include a detailed description of methods so that $EC_{1:5}$ values can be compared and contrasted between studies.

The time for equilibration was a function of soil salinity, with soils having the lowest EC_e values resulting the longest equilibration times. The 95 and 98% equilibration were observed faster for soils having EC_e values greater than 4 dS m^{-1} compared to soils less than 4 dS m^{-1}. Considering the feasibility for commercial laboratories, the 8 hr of agitation time could be considered adequate for the soil and water to reach equilibration, but independent tests within each laboratory are recommended.

The EC_e of soil is important for the purpose of soil classification and management, and the severity of soil salinity is an important factor used in relating to plant response and making crop and variety choices. Significant relationships existed between values of EC_e and $EC_{1:5}$, indicating that the prediction of EC_e from $EC_{1:5}$ measurements are acceptable. In general, the $\log_{10}$-transformed models yielded better relationships between EC_e and $EC_{1:5}$ compared to non-transformed and dilution ratio models irrespective of the agitation methods used for equilibration. Although the $\log_{10}$-transformed relationships would be recommended from this study, errors associated with EC_e may likely decrease the reliability of any predictive 1:5 approach.

Once a standard $EC_{1:5}$ procedures has been developed, the accuracy of applying predictive models between EC_e and $EC_{1:5}$ would be increased and the predictive model would help minimize the variability between laboratories and published results. Estimation of soil EC_e from $EC_{1:5}$ would help to minimize time and cost related to salinity assessment, especially if the plant responses are the focus of soil-water equilibration.

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APPENDIX A. SOIL EXPERIMENT SUPPLEMENTAL DATA

Table A1. Time schedule for $EC_{1:5}$ measurement procedures in shaking method for any of the soil samples (Paper 1).

Start shaking solution at	Tube No.	Shaking time period (min.)	Remove tubes from shaker at	Measure $EC_{1:5}$ values (after 5 min of settling) at
7:00AM	1–6†	5	7:05AM	7:10AM
7:00AM	7–12	15	7:15AM	7:20AM
7:00AM	13–18	35	7:35AM	7:40AM
7:00AM	19–24	75	8:15AM	8:20AM
7:00AM	25–30	175	9:55AM	10:00AM
7:00AM	31–36	355	12:55PM	1:00PM
7:00AM	37–42	715	6:55PM	7:00PM
7:00AM	43–48	1435	6:55AM	7:00AM
7:00AM	49–54	2875	6:55AM (next day)	7:00AM (next day)

† Tube No. 1–6, four replications of the same soils, one blank (ultra-pure water) and one standard KCl solution were analyzed in the totally same procedure.

Table A2. Time schedule for $EC_{1:5}$ measurement procedures in shaking/centrifuging method for any of the soil samples (Paper 1).

Start shaking solution at	Tube No.	Shaking time period (min.)	Remove tubes from shaker at	Measure $EC_{1:5}$ values (after 5 min of centrifuging) at
7:40AM	1–6†	5	7:45AM	7:50AM
7:40AM	7–12	15	7:55AM	8:00AM
7:40AM	13–18	35	8:15AM	8:20AM
7:40AM	19–24	75	8:55AM	9:00AM
7:40AM	25–30	175	10:35AM	10:40AM
7:40AM	31–36	355	1:35PM	1:40PM
7:40AM	37–42	715	7:35PM	7:40PM
7:40AM	43–48	1435	7:35AM	7:40AM
7:40AM	49–54	2875	7:35AM (next day)	7:40AM (next day)

† Tube No. 1–6, four replications of the same soils, one blank (ultra-pure water) and one standard KCl solution were analyzed in the totally same procedure.

Table A3. Time schedule for $EC_{1:5}$ measurement procedures in stirring method for any of the soil samples (Paper 1).

Stirring time period (min)	Tube No.	Time point to stir at										Measure $EC_{1:5}$ at
5	1–6[†]	9:05 AM	9:10 AM									9:15 AM
15	7–12	9:05 AM	9:10 AM	9:20 AM								9:25 AM
35	13–18	9:05 AM	9:10 AM	9:20 AM	9:40 AM							9:45 AM
75	19–24	9:05 AM	9:10 AM	9:20 AM	9:40 AM	10:20 AM						10:25 AM
175	25–30	9:05 AM	9:10 AM	9:20 AM	9:40 AM	10:20 AM	12:00 PM					12:05 PM
355	31–36	9:05 AM	9:10 AM	9:20 AM	9:40 AM	10:20 AM	12:00 PM	3:00 PM				3:05 PM
715	37–42	9:05 AM	9:10 AM	9:20 AM	9:40 AM	10:20 AM	12:00 PM	3:00 PM	9:00 PM			9:05 PM
1435	43–48	9:05 AM	9:10 AM	9:20 AM	9:40 AM	10:20 AM	12:00 PM	3:00 PM	9:00 PM	9:00 AM		9:05 AM
2875	49–54	9:05 AM	9:10 AM	9:20 AM	9:40 AM	10:20 AM	12:00 PM	3:00 PM	9:00 PM	9:00 AM	9:00[‡] AM	9:05 AM

[†] Tube No. 1–6, four replications of the same soils, one blank (ultra-pure water) and one standard KCl solution were analyzed in the totally same procedure.

[‡] 9:00 AM, is the time for the next day compared to the stirring time when it starts.