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Response of Capacitance Probes to Soil Solution Nitrate Concentration

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Abstract
Dielectric properties of soil are highly correlated with volumetric water content (VWC) of the medium, but at a relatively low frequency soil salinity has an important effect on permittivity measurements. A laboratory experiment was conducted to understand the potential of monitoring nitrate and chloride ions in soil solutions using capacitance-type soil probes EC-5 and EC-10 operating at frequencies of 70 and 5 MHz, respectively. Dielectric response of soil samples wetted with nitrate and chloride solutions of different concentration were compared at each frequency within the volumetric water content ranging from 0.1 to 0.3 m3/m3. Linear regression models were fitted through data to correlate the actual VWC, concentration of solutions, soil temperature and sensor output. At 70 MHz frequency the sensor response was primarily explained by moisture content for both solutions. Dielectric response of the EC-10 probe to change in ionic concentration was different for each wetting solution. Change in chlorine concentration demonstrated no evidence of having effect on the sensor response, while nitrate solution illustrated that the EC-10 probe is sensitive to the change in nitrate-N concentration within the water content and salinity range tested (from 0.05 to 0.51 dS/m). None of the fitted models demonstrated the statistically significant effect of temperature on dielectric measurements due to the little variation of the temperature (+/- 1.5°C) during the experiment.

Keywords
Capacitance probe, dielectric measurement, nitrate concentration

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Abstract. *Dielectric properties of soil are highly correlated with volumetric water content (VWC) of the medium, but at a relatively low frequency soil salinity has an important effect on permittivity measurements. A laboratory experiment was conducted to understand the potential of monitoring nitrate and chloride ions in soil solutions using capacitance-type soil probes EC-5 and EC-10 operating at frequencies of 70 and 5 MHz, respectively. Dielectric response of soil samples wetted with nitrate and chloride solutions of different concentration were compared at each frequency within the volumetric water content ranging from 0.1 to 0.3 m3/m3. Linear regression models were fitted through data to correlate the actual VWC, concentration of solutions, soil temperature and sensor output. At 70 MHz frequency the sensor response was primarily explained by moisture content for both solutions. Dielectric response of the EC-10 probe to change in ionic concentration was different for each wetting solution. Change in chlorine concentration demonstrated no evidence of having effect on the sensor response, while nitrate solution illustrated that the EC-10 probe is sensitive to the change in nitrate-N concentration within the water content and salinity range tested (from 0.05 to 0.51 dS/m). None of the fitted models demonstrated the statistically significant effect of temperature on dielectric measurements due to the little variation of the temperature (+/- 1.5°C) during the experiment.*

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Introduction

Agricultural production has doubled during the last decades through increased use of chemical fertilizers (Addiscott, 2005). Nitrogen (N) is among the most important elements required to sustain this increased crop production. However the extensive use of N in agriculture can significantly increase a risk of N loss to the environment largely through leaching of nitrate (NO_3) that was not used by the crop to ground water. High level of nitrate in surface and sub-surface waters can cause human health problems such as methemoglobinemia or gastric cancer (Bruning-Fann et al., 1993; Gupta et al, 2000), as well as environmental problems such as eutrophication of the aquatic environment and hypoxia (NRCS, 1997). To minimize nitrate loss from agricultural land a balance should be achieved between maximized crop production and nitrogen application through matching spatial and temporal needs within the field (Hoskinson et al., 1999; Roberts et al., 2002). The traditional method for determining nitrate concentration involves time-consuming and tedious procedures of extracting pore water from soil samples and their chemical analysis using different techniques and equipments (Carter and Gregorich, 2007), which is impractical for a large scale monitoring. Therefore a fast real-time in situ measurement techniques are needed which will allow evaluation of nitrate concentration change with sufficient resolution in space and time.

Intensive development of precision farming techniques has stimulated an increased interest in use of new energy-efficient dielectric soil probes for continuous monitoring of field variables such as soil moisture content (Robinson et al., 2008; Andrade−Sánchez et al., 2004). These commercially available soil moisture probes use a variety of techniques to measure dielectric constant of the medium which is correlated with the volumetric soil moisture content by various calibration equations (Topp et al., 1980; Dalton et al., 1984). However, numerous studies have shown that at relatively low frequencies dielectric permittivity of soil is particularly sensitive to soluble salt content and temperature (Kizito et al., 2008; Thompson et al., 2007; Regalado et al., 2007; Kelleners et al., 2004; Baumhardt et al., 2000; Andrade et al., 2001). Dielectric sensors operating at an effective frequency above 500 MHz benefit from the relatively stable permittivity region (Kellener at al., 2005), while at a frequency below 50 MHz dielectric permittivity measurements are biased by soil electrical conductivity (Gardner et al., 1998; Campbell, 1990). This error in water content estimate can be corrected if soil electrical conductivity and temperature are taken into account (Kizito et al., 2008; Bogena et al., 2007).

Numerous dielectric sensors can simultaneously measure both dielectric permittivity and soil bulk electrical conductivity of the medium, which is correlated to the ionic conductivity of soil solution (Hilhorst, 2000). Calculated electrical conductivity of the pore water has been used to determine soil salinity (Bouksila et al., 2008; Zhang et al., 2004). Several studies have successfully demonstrated the potential to estimate nitrate (NO_3) concentration of soil solution from electrical conductivity measurement using time domain reflectometry (TDR) sensors (Krishnapillai and Ranjan, 2009; Payero et al., 2006; Nissen et al., 1998).

However only a handful of capacitance sensors have been developed that can measure both VWC and soil bulk electrical conductivity by independent measurement of conductive and capacitive components of the dielectric permittivity of a porous material. While the real (capacitive) part of the permittivity is mainly influenced by the soil water content, the imaginary (conductive) part is related to the soil bulk electrical conductivity. Recent studies have shown that the capacitive behavior of soil at relatively low frequencies is affected by the conductive behavior causing an increase in the dielectric permittivity measurement (Thompson et al., 2007; Regalado et al., 2007; Baumhardt et al., 2000). Carr et al. (2007) studied the effect of dissolved ions in the soil moisture on the permittivity measurements using EC-5 and EC-10 probes operating at fixed-frequencies of 70 and 5 MHz, respectively. They found that the EC-10 probe was more sensitive to ionic conductivity than EC-5, hence illustrating its potential to estimate soil salinity.

The goal of the present study is to evaluate the sensitivity of capacitance probes to change in soil solution nitrate concentration at two fixed measurement frequencies (70 MHz and 5 MHz) for a range of soil moisture content using EC-5 and EC-10 probes. Furthermore, to determine the effect of other soil solutes on capacitance probes, we examined the differences in sensor response between soil samples wetted with nitrate and chlorine solutions of same concentration at both measurement frequencies. To increase accuracy of the used sensors calibration curves were developed for a range of soil moisture content by incorporating concentration of the wetting solutions.

Methods

The soil used for the laboratory-scale experiment was collected from a depth of 0.20 m in the Iowa State University Agronomy Research Farm located in Boone County, IA. The soil was air-dried, grinded and sieved to approximately ≤ 2 mm. The particle size distribution was determined using hydrometer method and was classified as a loam based on the USDA textural triangle. Different properties of the soil are presented in Table 1. The soil bulk density was found to be 1.15 g/cm^3 and was later maintained for all soil samples prepared for the experiment.

Six identical cylindrical sample holders were constructed using 10 cm diameter PVC pipe. The length of each cylindrical container was 22 cm. A removable threaded plug and a PVC cup were attached to the top and the bottom of the sample holder, respectively, to prevent leakage or evaporation of soil moisture and falling of the soil particles during experiment. Each cylindrical container was marked at a volumetric interval of 300 cm^3 to insure homogeneous bulk density throughout the soil sample during packing.

Three potassium nitrate (KNO_3) and three chlorine (Cl) solutions were prepared with concentrations of 6, 25 and 60 ppm of nitrate-N and Cl respectively. Electrical conductivities of the six wetting solutions were measured. Values ranged from 0.05 to 0.51 dS/m for the potassium nitrate solutions and from 0.02 to 0.22 dS/m for the chlorine solutions.

The air dried soil with an initial volumetric water content of 0.04 was uniformly moistened with each solution to prepare individual soil samples. Each sample was thoroughly mixed on a tray by gradually adding the proper amount of specific solution to arrive at the designed volumetric water contents (VWC). A total of five VWC levels were used during experiment ranging from 0.10 to 0.30 with increments of 0.05. As a result, 30 samples were prepared; one for each solution and moisture content combination.

The mixed soil was transferred to the cylindrical container and packed to the homogeneous bulk density in sections corresponding to the marked volumetric intervals. The sample holder was sealed at both ends and placed on a rotator device for at least 12 hours to allow the sample to reach equilibrium. The rotator device, similar to one described by Logsdon et al. (1993), held 6 cylindrical container at a time in a horizontal position, while rotating them back and forth nearly 360° about their cylindrical axis at 2 rpm to avoid accumulation of the added solution at the bottom of the sample.

Table 1. Summary of soil properties.

Sand	Silt	Clay	Soil pH	OM (%)	Nitrate-N (ppm)	Chloride-Cl (ppm)	Solble Salts 1:1 (dS/m)
0.43	0.33	0.24	6.6	5.5	24.1	5.6	0.38

After 12 hours, the sample holders were removed from the rotating device, the plugs were unscrewed and readings were taken from each soil sample using four EC-5 and four EC-10 probes. The row probe outputs, in millivolts, were recorded for 10 minutes at 10 readings per minute in each test. The averaged measurement of each probe was used for further analysis. Furthermore, the soil temperature readings were taken simultaneously in each sample using the Analog Device temperature sensor AD22100.

Results

The raw voltage output of both EC-5 and EC-10 probes increased with increasing soil water content at all six wetting solutions. The calibration curves of four EC-5 and four EC-10 sensors for all wetting solutions combined are shown in Figure 1. The fitted linear regression models yielded R^2 values of 0.931 and 0.967 for EC-5 and EC-10 probes respectively.

Sensor-to-sensor variation was observed between the probes during experiment. With increase in moisture content the difference between sensor readings in the same medium increased for both types of probe. However these variations were mostly within the 95% prediction interval (+/- 3.5 and +/- 2.5% for EC-5 and EC-10 probes respectively), which were very close to the model accuracies provided by the manufacturer for all mineral soils (+/- 3 and +/- 4% for EC-5 and EC-10 probes respectively).

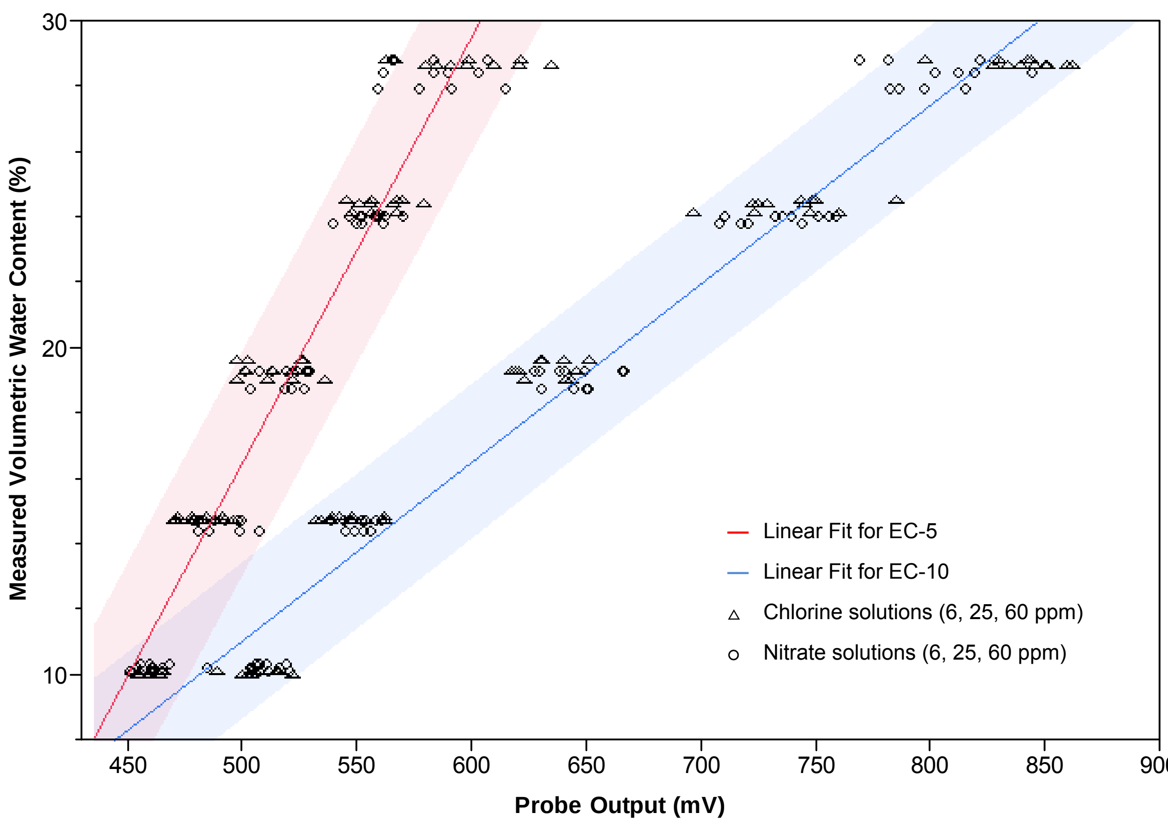

Figure 1. Relationship between VWC and the raw voltage output of EC-5 and EC-10 probes. The shaded areas represent 95% prediction intervals for each regression model.

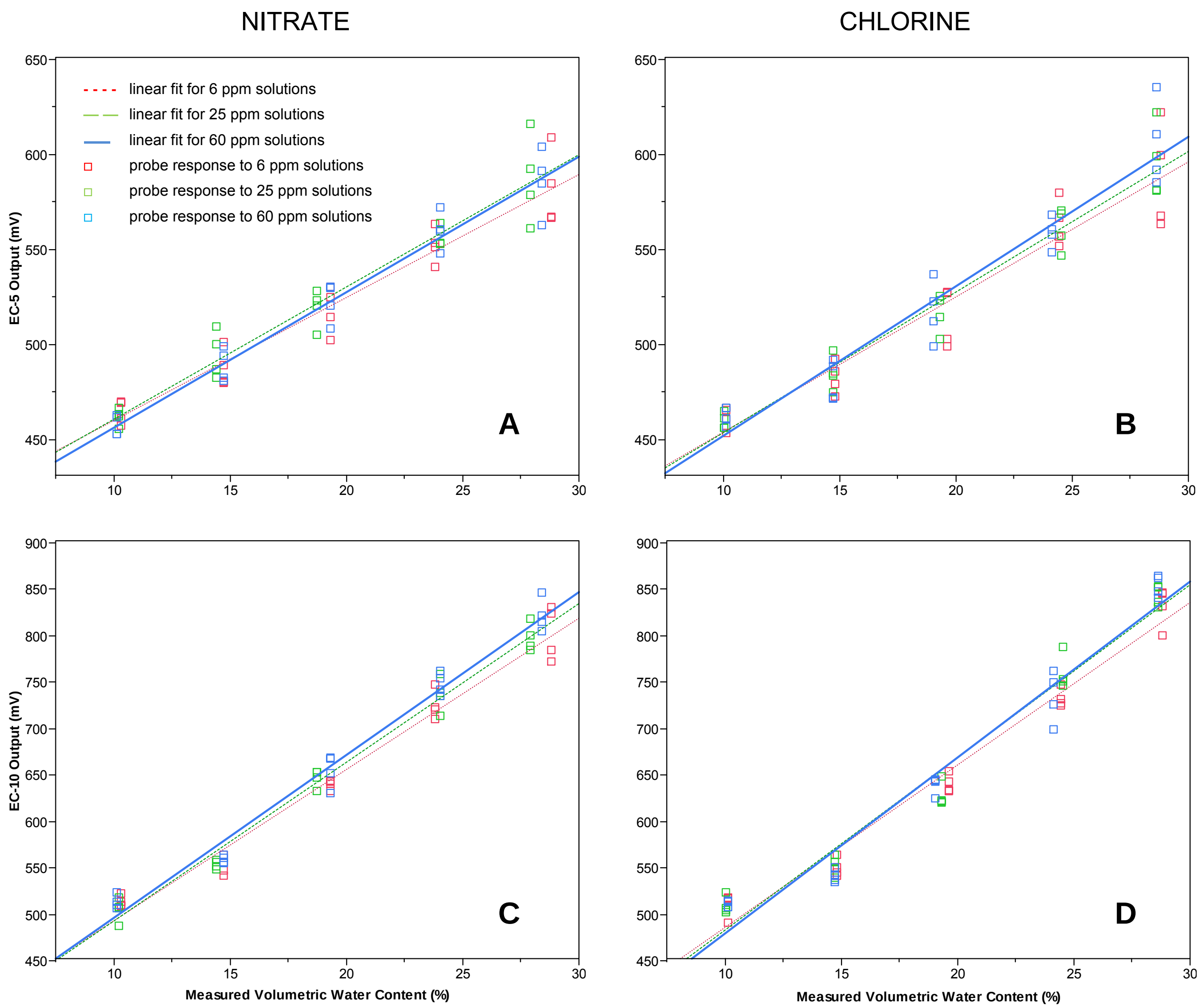

Figure 2. Response of the sensors to different concentrations of nitrate and chloride solutions: (A) and (B) response of EC-5 probes to nitrate and chlorine solutions respectively, (C) and (D) response of EC-10 probes to nitrate and chlorine solutions respectively

Figure 2 illustrates the different calibration curves of EC-5 and EC-10 probes developed for each solution separately. Mismatching responses of individual sensors in the same soil sample, caused by sensor to sensor variation, were treated as replications. Hence four measurements were obtained at each data point representing readings of four different probes. The EC-10 data was found to be more consistent for all six solutions than the EC-5 data, which became scattered with increase of the moisture content (Fig. 2a and 2b).

The R^2 values for EC-10 and EC-5 models ranged from 0.960 to 0.984 and from 0.907 to 0.958 respectively. Linear models fitted to the nitrate solution data had slightly higher R^2 values than models developed for soil samples wetted with different concentration of chlorine. EC-5 data showed no response to change in nitrate-N concentration, while EC-10 data illustrated noticeable sensitivity to it. The voltage output, and hence the dielectric response, of EC-10 probes increased with increasing concentration of added nitrate solution (Fig. 2c). At higher water content the concentration-induced change in probe response was stronger for nitrate solutions, which is illustrated by increasing divergence of the fitted curves with increase in VWC.

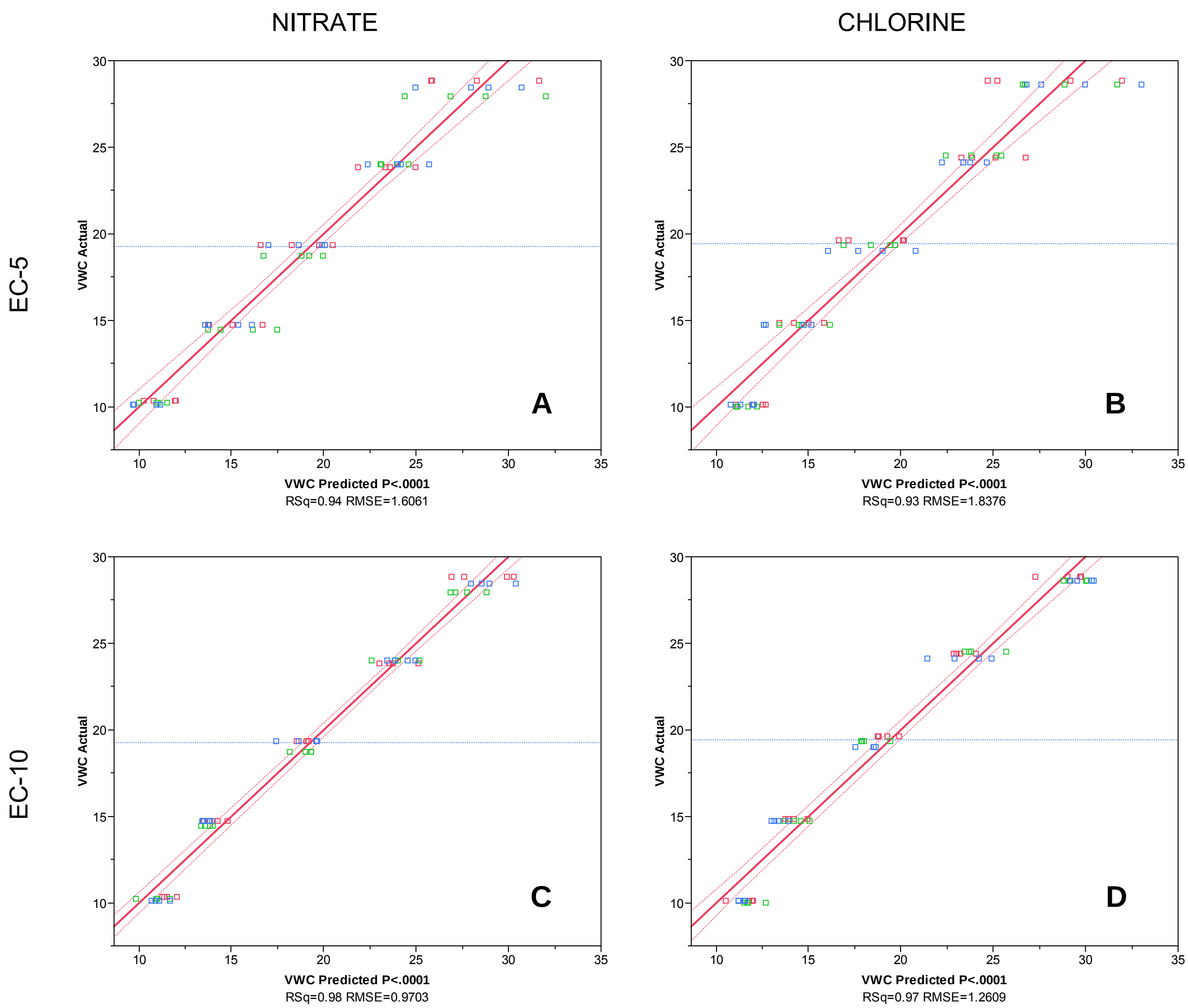

Figure 3. Actual versus predicted soil moisture content based on multiple regression model incorporating sensor output, concentration of wetting solution and soil temperature as variables.

EC-10 probes showed no sensitivity to change in Cl concentration (Fig. 2d), while EC-5 probes demonstrated some sensitivity (Fig. 2b). In general, higher water content and higher concentration of chlorine resulted in higher voltage output of the EC-5 sensor. It is worth mentioning that the slope of all fitted equations increased with an increase of solution concentration, but only two cases (response of EC-5 and EC-10 probes to chlorine and nitrate solutions respectively) showed a recognizable visual pattern between concentration of wetting solutions and sensor response.

To take into account the effect of concentration of the applied wetting solutions and soil temperature on prediction of soil moisture content first-order multiple regression models were fitted to individual sensor type/solute combinations. Regression lines through data for nitrate and chloride solutions showed good agreement between actual VWC and those predicted by developed model for both EC-5 and EC-10 sensors. All four models had high adjusted R^2 values and low root mean square error (RMSE), indicating good prediction abilities (Table 2). In general, the models developed for EC-10 probe performed better than those for EC-5. The actual moisture content plotted against predicted moisture content (Fig. 3) showed that EC-10 data lies close to 1:1 line for both nitrate and chlorine solutions, whereas EC-5 data became scattered with increasing VWC. Furthermore, models developed for nitrate solution had lower RMSE and slightly higher R^2_{adj} values than analogous models developed for chlorine solution.

When these multiple regression models were compared with simple linear regression models that used only sensor output as a predictor (Table 2), no significant improvements were found in corresponding R^2_{adj} values. What is interesting, RMSEs of EC-5 calibration curves were slightly increased for both solutions compared to the RMSE of similar simple regression models. On the other hand, RMSEs of EC-10 were decreased (by 0.025 and 0.060 for chlorine and nitrate solutions respectively) suggesting that inclusion of temperature and solution concentration data can enhance the prediction accuracy of EC-10 probe.

Table 2. Adjusted R^2 and RMSE of models.

		EC-5		EC-10	
Solution	Variables used in Model	R^2_{adj}	RMSE	R^2_{adj}	RMSE
Chlorine (6, 25, 60 ppm)	Probe Reading	0.925	1.824	0.963	1.286
	Probe Reading, Temp, and Solution Concentration	0.924	1.838	0.964	1.261
Nitrate (6, 25, 60 ppm)	Probe Reading	0.940	1.596	0.975	1.030
	Probe Reading, Temp, and Solution Concentration	0.939	1.606	0.978	0.970

To identify the effect of each variable on the prediction models the t statistic of the model parameters were examined (Table 3). As was expected, concentration of the solutions had no additional predictive value for calibration of EC-5 probes. The probe response at frequency of 70MHz was primarily dominated by water content for samples wetted with both solutions. Similar result was obtained for EC-10 probe in chlorine solution. The effect of the chlorine concentration on sensor response was not statistically significant (P = 0.3687) within the range of concentration tested. On the other hand, the nitrate concentration had a statistically significant effect (P < 0.01) on the EC-10 calibration model. None of the model showed enough evidence to conclude relationship between temperature and sensor dielectric measurements, which can be attributed to the fact that variation of the temperature (+/- 1.5°C) during experiments was small enough to have very little effect on probe response.

Table 3. Multiple regression model parameters.

		EC-5				EC-10			
	Term	Estimate	Std Error	t Ratio	Prob>|t|	Estimate	Std Error	t Ratio	Prob>|t|
Chlorine	Intercept	-49.8243	51.4004	-0.97	0.3365	55.6217	37.0384	1.50	0.1388
	Probe Reading	0.1240	0.0062	**19.99**	**<.0001**	0.0549	0.0018	**30.19**	**<.0001**
	Concentration	-0.0115	0.0106	-1.08	0.2850	-0.0066	0.0073	-0.91	0.3687
	Temp	2.8256	31.2086	0.09	0.9282	-41.9304	22.0800	-1.90	0.0627
Nitrate	Intercept	9.1182	59.1728	0.15	0.8781	37.6878	35.8536	1.05	0.2977
	Probe Reading	0.1388	0.0048	**29.06**	**<.0001**	0.0581	0.0012	**49.10**	**<.0001**
	Concentration	-0.0085	0.0101	-0.85	0.4014	-0.0186	0.0061	**-3.04**	**0.0036**
	Temp	-36.1760	34.8882	-1.04	0.3042	-32.4418	21.0119	-1.54	0.1282

Conclusion

In the present study, the EC-5 and EC-10 capacitance-type soil probes were calibrated for measuring volumetric water content at different salinity levels. Responses of each sensor type were compared in soil samples wetted with different nitrate and chloride solutions. The effect of each solute concentration and temperature on probe sensitivity was examined and following conclusions were drawn:

- Response of EC-5 sensor, operating at frequency of 70 MHz, was primarily explained by moisture content within the range of concentration used in the experiments. The simple linear regression lines fitted through nitrate and chlorine solution data showed good correlation between VWC and sensor output. Incorporation of additional parameters in the models slightly decreased the R^2_{adj} values and increased the RMSEs.
- EC-10 probe, operating at frequency of 5 MHz, showed that concentration of the solutions, and hence electrical conductivity of the pore water, has significant effect on sensor response depending on the solute type. Change in chlorine concentration demonstrated no evidence of having effect on the sensor response, while nitrate solution illustrated that the EC-10 probe is sensitive to change in nitrate-N concentration within the water content and salinity range tested. The fitted linear models adequately described the sensor response yielding better RMSE and R^2 values than those for EC-5. Incorporation of solution concentration (in ppm) enhanced the EC-10 calibration curve for soil samples wetted with nitrate solutions.

The results of the study illustrated that EC-10 sensors operating at a relatively low frequency is more sensitive to NO_3 than to Cl concentration in soil solution. Hence there is a potential to use EC-10 probes for estimating nitrate concentration in soil when actual soil moisture content is known. This is consistent with the results of Carr et al. (2007) which pointed out the usefulness of EC-10 probes for determining soil salinity over EC-5 probes.

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