Analysis of the Soil Selective Potassium Content, using Multifrequency EC Sensors

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Abstract: The relative salinity of soil is an important issue in today's sustainable, site-specific Precision Agricultural practice. The need to replace salt has an enormous impact on production costs, regarding current input prices. The standard method for measuring the salinity of a soil, based on a laboratory test method, is expensive and cumbersome and only the total salinity content can be determined. Growers need to get data faster and cheaper. With the usage of on-the-go soil sensors, this process can be significantly improved. This article presents the first step of developing a quick, cost-saving and easy-to-use measurement method for determining the selective potassium content on the spot, in real time. In our work, we developed a new measurement and calculation method using the analytical application of multifrequency conductometry, to determine selective salinity. It is proved that the electrical conductivity (EC) develops according to different functions, by applying solutions containing K+ ions with a defined concentration, by saturating the soil with a continuously, but reasonably varying measuring frequency. Based on this recognition, the selective salinity of soil can only be determined by interpolation from a frequency-series of EC measurements, in soils containing only one electrolyte.

Keywords: digital agriculture; electrical conductivity; soil salinity; variable frequency; interpolation

1 Introduction

1.1 Digital Farming Concept

Nowadays, there is a constantly growing demand to know our soils as thoroughly as possible and the precision technology toolkits already present in our machines

provides effective support. Numerous smart solutions help farmers in all areas of Precision Agriculture, onboard and offboard the tractor.

Digital agriculture is still a young concept. Synonymous terms are agriculture 4.0, smart farming or precision farming, whereby the terms are to be considered evolutionary. The latter term has its origins back in the 90s. As Figure 1 shows, Griepentrog differentiates between the terms as follows [1]:

- **Precision Farming** focuses on site-specific processing in which the growth conditions are optimized by means of sensors and application technology

- **Smart Farming** adds a level of decision support through fusion and analysis of information

- **Digital Farming** (or Agriculture 4.0 or Farming 4.0) includes the Internet of Things as well as Cloud Computing and Big Data
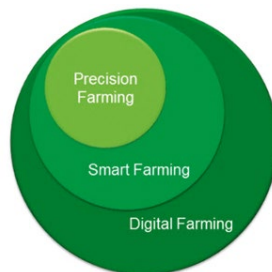

Figure 1
The development of Precision to Digital Farming [1]

In the universe of digital agriculture, the basic goal of site-specific management of agricultural inputs is to increase the profitability of crop production by improving the quality of output and protecting our environment. The process of getting to know our soil as is the Farm Management Information System (FMIS) designed considering the benefits of data processing and data use provided by digitization, functions as a unit of 5 processes [2]:

- **Data acquisition**: measurement and recording of data on the field, part-fields

- **Data collection and storage**: collecting data on a specific field, a specific location within a field

- **Data processing**: providing useful information for decision-making

- **Decision-making**: defines the cultivation technology in detail, broken down for each operation, as well as the details of tillage, nutrient replenishment, planting and other operations, together with characteristic parameters and setup

－　**Executing the operation**: turning the decision-making results into practice

As a first step of the recognition process, it is necessary to obtain data on the variability of the parameters describing the soil properties within the field for geo-positioning. With the correct and sensible use of sensor technology, farmers can better understand their crops and soils while conserving resources and at the same time saving resources and reducing their impact on the environment [3]. If we do not have access to the necessary and relevant data, then the decision-making and the performance of the operation are affected by the quantity and quality of the collected data, which determines the intervention and the cost-effectiveness of the intervention on the field.

1.2　Testing Soil Properties with Sensors

Today, sensor technology is one of the fastest growing areas of technology. A sensor is a device that can detect a change in the physical or chemical environment, which then converts it into electrical signals, both electric current and voltage. The precision agriculture mainly relies on real-time monitoring of soil conditions using information technology and (Global Position System) GPS technology, and then analyzing and managing the spatial-temporal variability of soil and field crops. These information help make decision on precision application of crop inputs including water and fertilizer. It can improve the efficiency and reduce the losses of water and fertilizer [4].

The ever-increasing prices of fertilizer and growing ecological concern over chemical run-off into sources of drinking water, have brought the issues of precision agriculture and site-specific management to the forefront of present-day technological development within agriculture and ecology. Due to increases in the cost of fertilizer production inputs—predominantly nitrate (N), phosphate (P), and potassium (K) those in agriculture are looking for ways to optimize plant yield while minimizing the application and consumption of fertilizer. Since these macro-nutrients vary even on a small scale throughout a cultivated field, numerous researchers have attempted to develop an on-the-go sensing apparatus that can map the presence of these chemicals in situ so that this map, once overlaid with parameters such as pH, electrical conductivity (EC), crop yield, and mechanical properties of the soil, can give a precise spatially varying prescription for fertilizer application [5].

The global Agricultural Sensors market was valued at USD 1,505.4 million in 2020 and expected to reach USD 3200.8 million by the year 2028, at a Compound Annual Growth Rate (CAGR) of 11.04%. Smart Sensors allow farmers to maximize yields using minimal reserves such as fertilizer, water, and seeds. By utilizing sensors and mapping fields, farmers can commence realizing their crops at a micro-scale, conserve resources, and lessen influences on the ecosystem. Smart agriculture has

roots moving back to the 1980s when the Global Positioning System (GPS) capability became accessible for civilian use. Once farmers were able to map their crop fields accurately, they could monitor and use fertilizer and weed treatments only to parts that required it. During the 1990s, early precision agriculture users implemented crop yield monitoring to create fertilizer and pH correction suggestions. As more variables could be calculated and entered into a crop model, more accurate recommendations for fertilizer application, watering, and even peak yield harvesting could be made. The device capture analyzes and transmits information like temperature, humidity, pressure, water content, etc. using radio signals. It gathers the data and sends it to the base station. The base station then analyses the data and carries it for further processing. Usually, sensor networks have a base station known as sink and several other sensors too, which sense and transmit the signals along with sending information to other nodes. Weather stations are self-contained units placed at various sites throughout growing fields. These stations have a mixture of sensors suitable for the local crops and climate. Data such as air temperature, soil temperature at various depths, dew point temperature, wind direction, relative humidity, rainfall, leaf wetness, chlorophyll, wind speed, solar radiation, and atmospheric pressure are measured and recorded at predetermined intervals [6].

For sensor measurement, the following sensor groups can be found in crop production: crop sensors, environment sensors, function monitoring sensors and soil sensors. From the growth of forecast solutions using sensors, it is very easy to recognize that the future of site-specific crop production is moving towards a sensor-based approach because obtaining important soil characteristics quickly and cheaply is still one of the biggest challenges in precision agriculture today. A number of researchers and manufacturers are trying to develop on-the-go soil sensors, to directly measure the mechanical, physical, and chemical properties of the soil. The disadvantage of the practical use of the increasingly widespread ground sensors is that they are less accurate than individual sampling and laboratory tests, but the advantage is that they are suitable for rapid measurement and are therefore cheaper in practice [7]. In the future, mechanized soil testing and nutrient mapping solutions will become widely available using faster and more cost-effective measurement tools. To implement sustainable agricultural and environmental management, a better understanding of the soil at increasingly finer scales is needed. Conventional soil sampling and laboratory analyses cannot provide this information because they are slow and expensive [8].

1.3. Proximal Topsoil Sensors

Proximity detection is defined as using sensors applied in the field to obtain signals from the ground with the sensor unit in contact with or near the ground (within 2 m) [9]. The following measurement principles and sensors are distinguished in practice [10]:

–   Electrical Conductivity (EC) Sensors

–   Ground Penetrating Radar and Reflectometers

–   Visible (VIS) – Near-Infrared (NIR) – Mid-Infrared (MIR) Diffuse Reflectance Sensors

–   Magnetic Susceptibility Sensors

–   Gamma-Ray sensors

–   X-ray Sensors

–   Other Proximal Soil Sensors like photoacoustic spectroscopy, laser-induced breakdown spectroscopy laser-induced fluorescence spectroscopy and inelastic neutron scattering. Mechanical sensors that measure soil penetration resistance have also been used extensively and integrated with other sensors in soil science and precision agriculture, with applications ranging from soil compaction assessment to 3D modelling of soil layers.

Of course, the different sensors can be combined as desired, which is defined as using a multisensory measurement.

1.4   Conductometry - Measurement of Electrical Conductivity

Soil properties often vary significantly within a field, and one of the challenges in precision agriculture is collecting enough soil data to accurately delineate this variability. Soil electrical conductivity (EC) has become a widely used tool for mapping soil variability within fields. Soil EC measurements are typically correlated with soil texture, moisture, and salinity. Soil texture is an important factor for crop yields, because it relates to the water-holding capacity, cation-exchange capacity, rooting depths, drainage and other properties, that can impact crop production [11].

The good conductance of parts is characterized by electrical conductivity. Conductometry is an analytical method based on the measurement of the electrical conductivity of solutions. In analytical chemistry, the electrical conductivity of electrolyte solutions is measured by conductometric methods, and analytical information is derived from this and its changes due to chemical reactions. The conductivity of materials (symbol G, the unit is siemens, S) is the reciprocal of their electrical (ohmic) resistance (symbol R, the unit is ohm, Ω). Electrical conduction requires the presence of charge carriers (e.g., electrons or anions and cations) that are able to move under the action of the electric field. The ion migration in the solution due to the electric field is the reason for the electrical conduction of the solutions. Based on this, electrical conductors and insulators are to be distinguished. Pure (distilled) water, since it contains only very small charge carriers in the concentration $[H^+] = [OH^-] \approx 10^{-7}$ mol l^{-1} corresponding to autoproteolysis, conducts electricity only to a very small extent, therefore it can be

considered as an insulator. However, the concentration of cations and anions in aqueous solutions of electrolytes can be significant, making them mostly conductive depending on the degree of electrolytic dissociation. Each ion in the solution makes some contribution to the conduction value. These are inseparable, so conductometry is not suitable for the selective measurement of individual ions, i.e., it is not an ion-specific method [12]. Therefore, its analytical application is limited to the study of systems that

- Contain only a single electrolyte (so the contribution of the "background" is negligible)

- Chemical reactions take place in them, during which the mobility of the ions that make up the system changes significantly (compared to non-zero "background") [12]

1.5   Principles of Soil Electrical Conductivity

What about special solutions to sample the soil? Do conductivity measurements only provide information on all salinity in the soil and are they not suitable for ion-selective measurements? The measured conductivity is therefore only approximate information [13]. Figure 2 shows the possible directions of displacement of the charged particles during the 3-phase, unsaturated soil conductivity measurement. The air acts as an insulating medium [13] [14]. Three pathways of current flow contribute to the EC of a soil:

(i)   A liquid phase pathway via dissolved solids contained in the soil water occupying the large pores

(ii) A solid–liquid phase pathway primarily via exchangeable cations associated with clay minerals

(iii) A solid pathway via soil particles that are in direct and continuous contact with one another [15].

These three pathways of current flow are illustrated in Figure 2, the air is shown in white, the liquid in gray, and the soil particles are scored [14].
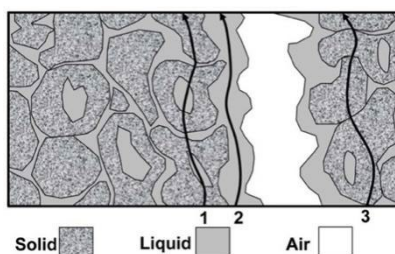

Figure 2
Direction of electrical conduction in 3-phase soil [13] [15]

Nevertheless, the explosive spread of EC measurements since the 90s can be attributed to 2 reasons. On the one hand, with the development of technology, the size of conductivity measuring devices has been significantly reduced and are also available as mobile devices, and on the other hand, satellite positioning systems have become a part of our lives and conductivity is an easily measurable feature.

1.6   Chemical Composition of Soil Moisture (Soil Solution)

Various inorganic salts, organic matter and gases are dissolved in the soil moisture. Dissolved mineral salts are dissociated into positively and negatively charged ions and the ions are surrounded by a hydrate shell. The following ions are mainly present in the soil solution:

- Ca^{2+}, Mg^{2+}, Na^+, K^+, NH^{+4} and in some soils Al^{3+}, Fe^{3+} or Fe^{2+} (cations)

- HCO^{3-}, CO_3^{2-}, Cl^-, SO_4^{2-}, NO^{3-}, H_2PO^{4-}, HPO_4^{2-} (anions)

Most of the dissolved organic compounds are organic acids and low molecular weight humic substances, with CO_2 and O_2 being the most significant dissolved gases. Soluble substances in the soil are mostly products of weathering and soil formation, but salts also enter the soil solution with groundwater close to the surface. In addition, fertilizers and compounds applied to irrigation water in agricultural areas modify the salinity of the soil [16].

1.7   Hydrated Ions

The interaction between solvated ions and water has been a subject of great interest due to its importance in various chemical, biological, and environmental processes [17]. Various experimental and theoretical studies have explored aqueous solutions of ions, both in the bulk [18] [19] and at interfaces [20-22].

The size of the hydrate shell in aqueous solution is illustrated in Figure 3. The smaller the diameter of the dehydrated cation and the larger its charge is, the thicker the hydrate sphere in aqueous solution is. This also affects the mobility of hydrated ions. Potassium is the most mobile element in the soil and as such readily available to plants however care must be taken to avoid it leaching out of the vadose zone of the soil because of its high mobility [23]. Explanation for conclusion:
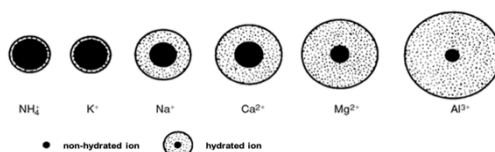

Figure 3
The size of the aqueous hydrate cover for each element in the soil [16]

In fact, the common cations in soil have a wide range of sizes, as shown in Fig. 4. Rengasamy and Sumner [24] showed that the dispersive effect of Na is greater than that of K, and that the flocculating effect of Ca is greater than that of Mg. Conventionally, these cations have been called base or base-forming cations [25]. Hydrated radii are critical to the composition of soil.
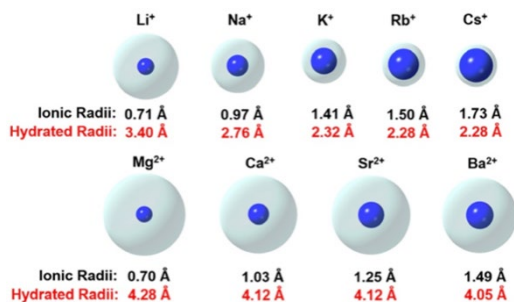

Figure 4

The ionic and hydrated radii (effective sizes) in angstroms, of some of the common ions [26]

1.7   Electrical Conductivity Sensors

Because soil salinization is one of the critical global problems threatening land productivity [27]. Saline soils have been reported from more than 100 countries around the world and covers more than 1125 million hectares of land [28]. It is increasing at about 1-2% every year and is estimated that 50% of the available arable lands can be affected by soil salinity by 2050 and would pose a serious threat to the sustainable development of global agriculture [29].

It is obvious that the measurement of electrical conductivity (EC) is one of the in-situ tools for soil testing required for precision farming and accordingly there is a large-scale literature on the development of EC detectors. These detectors typically and traditionally have 4 electrodes [30-32]. The traditional current-voltage four-electrode method to better fit for in-situ measuring and aimed at developing an in-situ soil EC detector with low price, easy operation, high measurement precision, integral control procedures and data processing procedures [30]. Apparent soil electrical conductivity is one of the simplest, least expensive soil measurements to obtain useful information about soil characteristics, which have a vital role in precision agriculture [31].

The aim of this paper is to present the basic research of measuring selective potassium content of the soil by multifrequency electrical conductivity measurements based on principles of conductometry (EC measurement), which is being developed within the framework of Hungarian University of Agriculture and Life Sciences, Institute of Technology. The methodology development focuses on

how to replace slower, more complex, and thus relatively expensive laboratory determination of salinity with as many on-the-go measurements as possible. It is noted that to the best of our current knowledge, conductivity tests with soil sensors alone are not sufficient to infer the salinity properties of a given field. The measurement is affected by the unequal distribution of different nutrients, different pH conditions, different particle compositions, differences in organic matter content or even temperature [13].

2 Methods and Materials

2.1 Specification of Solution

The measurements were performed in the Lab of Institute of Technology, in Hungarian University of Agriculture and Life Sciences. The laboratory temperature was set at a constant 22°C. The solutions and the soil were used for the measurement at 22°C.

During the measurement we wanted to study the effect of the cations of the dissolved salts in the soil moisture under natural conditions. The selected cation was K^+, because the reasonable replacement of the use of K^+ microelements in cultivated plants is a burning and expensive issue. We had to introduce these ions into the soil solution in the form of water-soluble salts. The salts and chlorides are very soluble in water, so we chose the chlorine salts of the K^+ cation for the experiment. We prepared 1 M concentration (1 M = 1 mol dm^{-3}) stock solutions from Potassium chloride, ACS Reagent, 99.0-100.5% CAS: 7447-40-7. These solutions were further diluted with the dilution series: 1 M; 0.66 M; 0.5 M; 0.33 M and distilled water i.e., 0 M was the reference measurement. Between the measurements we always washed the system with distilled water, so that any ions left on the electrode during the previous measurement, do not cause a measurement error.

2.2 Specification of Soil

As a model soil for our investigations, the same pH_{H2O} <7 soil has been chosen which had no detectable $CaCO_3$ content. The test soil was collected in Szárítópuszta in Gödöllő town, using 0-20 cm layers of arable land (47° 35' 47.65" N, 19° 21' 18.54" E). Soil type based on IUSS Working Group WRB, 2015 was Eutrict Arenosol (Aeolic, Aric, Ochric, Raptic) from elevation: 232 m with texture of sand.

2.3   Hardware Specification

Elements of the measuring circuit as in Figure 5:

1.   SOURCETRONIC ST2829C Precision LCR meter with USB stick

2.   SOURCETRONIC ST26011B Test Fixture: the resolution of the instrument is 0.00001 nS, the basic accuracy is 0.05 %. During the measurement, the voltage of the electrodes is 10 V DC. The output impedance of the instrument was 100 Ω

3.   KSP-F01A Dosing Pump

4.   Measuring cup (500 ml, 50 ml increments) to store the input solution

5.   Measuring cylinder $\varnothing$ 61 mm x 137 mm

6.   Measuring electrodes 2 pcs, $\varnothing$ 2 mm, length: 80 mm full length insulated, uninsulated part length 5 mm, probe distance 12 mm, material is stainless steel

7.   Piping, inner $\varnothing$ 3 mm

8.   Tray with raiser

Figure 5
The measuring circuits

The LCR meter measured and recorded the electrical conductivity between the two stainless steel electrodes placed in the ground paste in an endless series at a predetermined program frequency and restarted the measurement in an endless cycle upon completion of the measurement sequence. The frequency values used for the measurements were recorded in the range of 20 Hz and 10^6 Hz.

Each new measurement was started by calibrating the peristaltic pump. The liquid delivery of the pump was set to 0.5 g s^{-1}. During the measurements, the liquid

solution was pushed into the cylinder (Figure 6) by a pump at the bottom of the measuring cylinder, where the solution was introduced into the cylinder through a distribution plug made of 3D printing so that the soil in the cylinder could saturate evenly. The solution left the measuring cylinder on the perforation formed at the top of the cylinder and collected on the tray under the measuring cylinder, from which the used output liquid was continuously emptied.
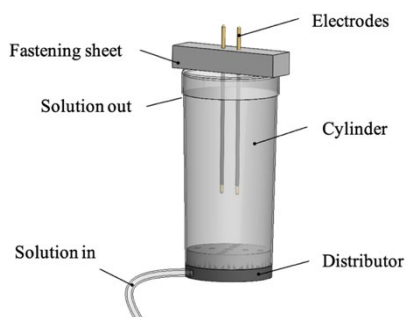

Figure 6
The measuring cylinder and connecting parts

Before starting each new measurement, we washed the soil paste with distilled water in the measuring cylinder so that the ions remaining after the previous measurement did not affect the measurement results. The experiments were repeated three times with each mixed input solution by adding 300 ml of the input solution and each filling was followed by neutralization with 300 ml of distilled water. If the soil paste was not reduced to less than 0.1 mS during the washing, an additional washing with 100 ml of distilled water was performed. Each measurement was repeated three times.

3 Results and Discussions

3.1 Reference Functions to Determine the K Content

From the reference measurements, we determined the arithmetic mean of the measured EC values for each concentration and frequency by examining the adjacent values and averaging them from the values where the change did not exceed 5% ($\Delta < 5\%$). Therefore, the frequency-dependent averages for a complete measurement were determined. After performing the calculations, it was possible to record the measurement results for the calculated data sets per ion, concentration and measurement frequency.
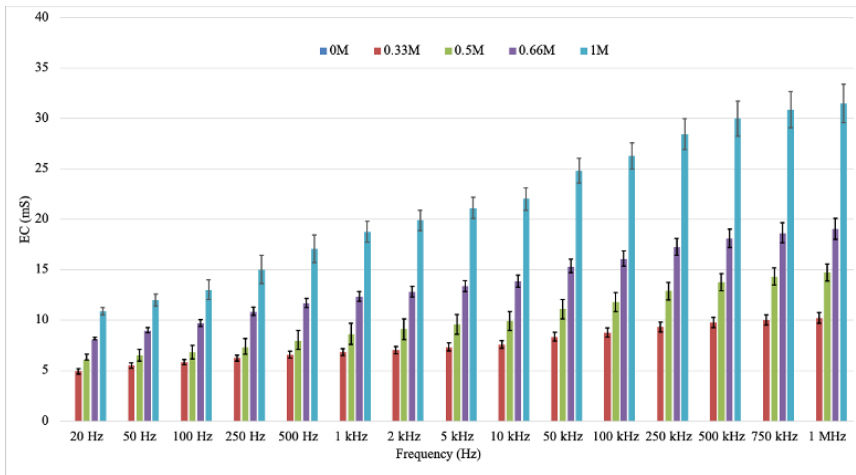
Figure 7

EC values for different concentrations of KCl solutions with standard deviation

When examining the quality of the derived data through the standard deviation of the mean estimate (Figure 7). It is much more informative to plot the change in EC of the solutions of the investigated concentration as a function of the logarithmic frequency. Figure 8 not only shows that the EC grows with increasing concentration, but it is also clear that if the measurement frequency is increased, the measured EC value also increases.
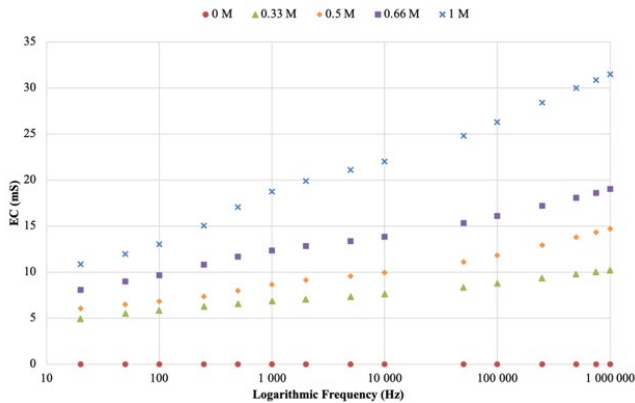

Figure 8

EC as a function of the logarithmic measurement frequency for different concentrations of KCl solutions

Plotting the measurements in a 3D space, the following diagram shown in Figure 9, can be drawn:
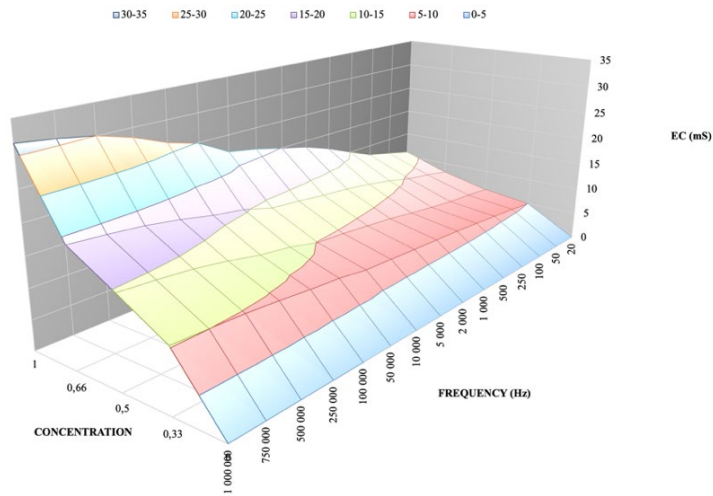
Figure 9
EC as a function of K$^+$ concentration and frequency

Finally, when plotting the frequency on a linear scale and plotting the EC measurement results of the solutions of different concentrations and fitting a function to the obtained points, considering that $R^2 > 0.9$. The reference functions of the different K$^+$ cation concentrations are illustrated in Figure 10.
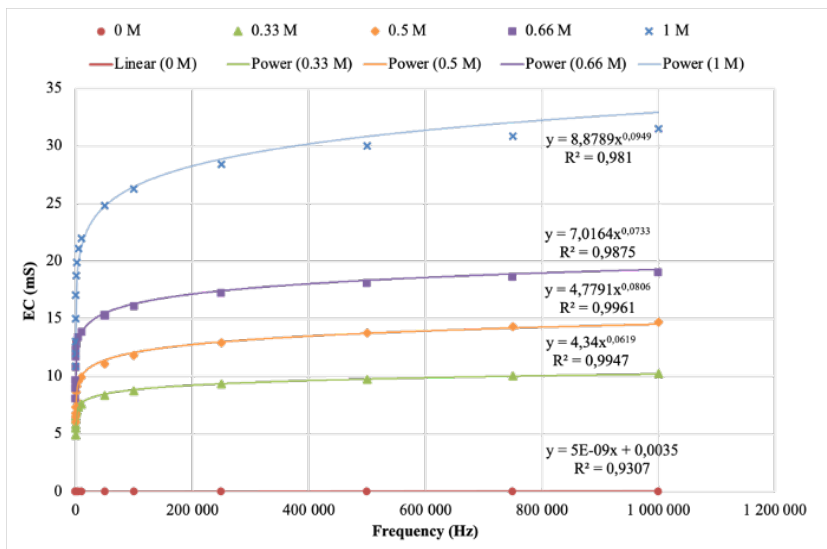

Figure 10
EC saturation curve for solutions with different K+ concentrations on a natural frequency scale

3.2 How to Determine the Concentration of an Unknown Solution Based on our Experiments

Relative EC is measured, but the selective salinity of the soil must be inferred. The solution requires interpolating the obtained measurement results. The solution is to determine the concentration by interpolating the reference values. Premises:

– The analytical application of conductometry is limited to the study of systems that contain only a single electrolyte, so the contribution of the "background" is negligible

– A complete measurement is a measurement performed over the entire frequency range

– It is necessary to use the same frequencies

3.3 The Application of the Following Notations for Deduction

– $x_1 < x_2 < ... < x_n$ the preliminary measurement frequencies

– $z_1 < z_2 < ... < z_n$ EC values for the measurement frequencies in a series of measurements

According to Figure 11 at the given frequency x_1 of the previously measured reference EC values, let u_1 and v_1 be the reference values which enclose the measured value of z_1 i.e.:

$$u_1 \leq z_1 \leq v_1 \tag{1}$$

and so on, in x_n it is u_n and v_n that enclose the value of z_n, that is:
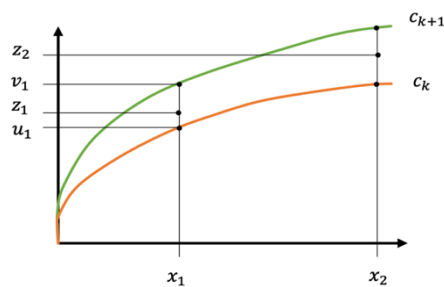
$$u_n \leq z_n \leq v_n \tag{2}$$

Figure 11

Determination of concentration by interpolation of reference values

Suppose that all points (x_i, z_i) fall between the same two adjacent EC curves.

Use:

$i = 1, 2, \dots, n$ fixed $\hspace{6cm}$ (3)

and these two curves correspond to the concentrations c_k and c_{k+1}

We are looking for a $x_i \in [0,1]$ value, to which:

$z_i = x_i u_i + (1 - x_i)v_i$ $\hspace{6cm}$ (4)

from

$x_i = \frac{v_i - z_n}{v_i - n_i}$, where $\hspace{6cm}$ (5)

$i = 1, 2, \dots, n$ $\hspace{6cm}$ (6)

x_i is the average of interpolation constants

$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i$ $\hspace{6cm}$ (7)

This interpolates between c_k and c_{k+1} reference concentrations:

$\bar{c} = \bar{x}c_k + (1 - \bar{x})c_{k+1}$ $\hspace{6cm}$ (8)

Ergo, the estimated concentration of the unknown solution measured will be the average of the concentrations that interpolated between the EC values given by the reference measurements at the measurement frequency occurring in one measurement.

Conclusions

With the current development of soil sensors, it becomes possible to measure soil properties in real-time, so growers can now get immediate information about the state of the most important resource, the soil, which can save resources and reduce the impact on the environment. The use of soil sensors enables rapid, immediate and cost saving soil testing [33] [34] and nutrient mapping solutions.

In our current research, we were looking for the answers to whether one of the simplest and cheapest measurement methods, i.e., the new approach to measuring the electrical conductivity as a parameter of the soil, offers the possibility to measure selective salinity in the laboratory. Our experiments demonstrated that there is a significant correlation between the salt concentration of the soil, the measurement frequency and the measured EC. As the frequency has been increased, the measured EC of the soil saturated with KCl solution of a given concentration changed and increased according to the functions published in the results.

We have also developed a calculation model that can provide guidance for calculating the selective salinity of soil in laboratory conditions using the analytical application of conductometry. The model shows that by consistently changing the measurement frequencies, the selective salinity of a known soil can be determined by EC measurement under laboratory conditions. Because with solutions containing K^+ ions of the same concentration, the EC is saturated according to different functions depending on the measurement frequency.

As we advance our research on reference measurements, we would like to prove our thesis, by testing soils saturated with Ca^{2+} cations. If that test yields positive results, we will refine our model, under actual field conditions.

Acknowledgement

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