Determining the relationship between the dielectric properties and the basic physical and chemical parameters of the air-dry soil

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Abstract- Measurements of the electric conductivity, σ, and relative dielectric permittivity, ε_r, were conducted (0.1Hz –15 MHz) on 40 air-dried soil samples that were subsequently analyzed for pH, total organic matter in soil ($\Delta M/m_1$), P_2O_5, , Fe_2O_3 and heavy metal concentrations (Pb, Cd, Cr, Ni, Cu и Zn). The pH of soil samples varied between pH 5.25 and pH 7.73 (mean pH 6.71), the $\Delta M/m_1$ varied between 1,49 % and 9.96 % (mean 4.56 %). The mean content of Fe_2O_3 was 44352.5 mg/kg, which was 114 fold higher than the mean concentration of the heavy metals (Pb, Cd, Cr, Ni, Cu и Zn). The mean content of P_2O_5 was 0.26% . We found a linear relation between σ (1 MHz) and the indicated physicochemical parameters; P_2O_5 (coefficient of correlation, r = 0.637), pH (R = 0.530) , Fe_2O_3 concentration (r = 0.450), content of $\Delta M/m_1$ (r = 0.545) and heavy metal concentration (r = 0.460). Similar relationships and correlation coefficients were found between ε_r (10 kHz) and the same physicochemical parameters. As the latter represent general biogeochemical parameters, our findings suggest that dielectric spectroscopy may provide useful approach to probing soil geochemistry, iron cycling and anaerobic microbial activity. Furthermore, our results yield insights into the impact of various physicochemical parameters on the induced polarization of soils.

Index Terms- Dielectric spectroscopy, Soil, Conductivity, Relative permittivity, Total content of heavy metals (Pb, Cd, Cr, Ni, Cu и Zn), Total organic matter ($\Delta M/m_1$)

I. Introduction

The quality of soil is controlled by physical, chemical and biological components of soil and their interactions [1, 2]. The soil has physical, chemical as well as electrical properties. Colour, texture, grain size, bulk density etc., comprise the physical properties; Nutrients, organic matter, pH, etc., comprise chemical properties while, electrical properties include dielectric constant, electrical conductivity and permeability. The concept of soil health and soil quality has consistently evolved with an increase in the understanding of soils and soil quality attributes. Perveen S. et al. [3] have studied micronutrient status of soils and their relationship with various physico-chemical properties. Chhabra G. et al. [4] have shown that available manganese decreased with soil pH and available copper increased with clay and organic carbon content. Results of physical and chemical tests provide information about the capacity of soil to supply mineral nutrients. Martin C. et al. [5] have shown that the electrical conductivity of soil water is a good indicator for absorbing the amount of nutrients available for crops.

Bell R. W. and Dell B. [6] demonstrated that the deficiency of nutrients has become major constraint to productivity, stability and sustainability of soils. The status of available micronutrients in soils and their relationship with various physico-chemical properties have been attempted by several investigators [7,8,9]. Avnimelech Y. et al. [10] estimated the organic content and bulk density of flooded mineral soils and found that the sediment bulk density was inversely related to the organic carbon concentration. The measurements of dielectric constant of soils as a function of moisture content over wide microwave frequency range were carried out in the past by many investigators [10-17]. These investigators have used soils covering different parts all over world and with different texture/structures. Almost all these investigators have concluded that the dielectric constant of soils is strongly dependent on moisture content. Further, Sami S. [19] has reported the effect of chemical and mineral composition of dust on dielectric constant. Srivastava S. K. and Mishra G. P. [13] studied the characteristics of soils of Chhatisgarh at X – band frequency and showed the dependence of dielectric constant of soils on their texture. Calla O. P. N. et al. [20] have studied the variability of dielectric constant of dry soil with its physical constituents at microwave frequencies. Dawood N. K. et al. [21] have evaluated the dielectric constant by clay mineral and soil physico–chemical properties and showed that texture and mineral content of soil had different impact on dielectric constant. Chaudhari H. C. and Shinde V. J. [22] have reported that the dielectric properties of dry soil at microwave frequency in X-band are function of its chemical constituents and physical properties. In a detailed study, Sengwa R. J. and Soni A. [23] have reported the variation of dielectric constant with density of dry minerals of soil at 10.1 GHz.

Below 50 MHz the frequency dependence of dielectric permeability ε and the specific conductivity σ of soils strongly depend on their water content [22,25,26] and on the soil type [27]. A strong dependence of inductive polarizability on Fe content in wet and rich in organic matter marshy soils was established[28].

The properties of dry soil along with its type have a great importance in agriculture. For microwave remote sensing applications, dielectric constant is the primary important electrical property for dry soil. Further, due to dependence of dielectric constant on the physical constituents and chemical composition of the soil, the detailed study of its variability with these soil parameters will be useful for better understanding of soil physics[2,29].

The aim of the present study was to investigate the relationship between soil dielectric properties and some basic physicochemical parameters in air-dried samples of arable soils.

II. MATERIALS AND METHODS

Reagents. The reagents are qualified "p.a". Stock standard solution (Merk, Germany) with a concentration of 1000 mg / l for the determination of Fe, Pb, Cd, Cr, Ni, Cu and Zn was applied. In all procedures double-distilled water was used.

Soil samples. The study included five major regions of the central part of South Bulgaria (Asenovgrad, Pazardzhik, Plovdiv, Stara Zagora Parvomai). Sampling was carried out in accordance with ISO 10381-1, 2002[30]; ISO 10381-2, 2002[31]; ISO 10381-3, 2002[32]. A total of 40 fixed sites with an area of 100 m^2 were defined. From each site 3 separate samples from depth of the plow layer 0-30 cm were taken. The average sample was prepared by mixing and homogenizing of incremental samples and stored in sealed glass containers for analysis in the laboratory.

Determination of pH and total organic matter of soil . pH determinations were accomplished by pH – meter (pH-meter Consort C932, Belgium), with a glass electrode in a suspension containing soil / water 1:5 according to ISO 10390 [33] . The moisture content and the $\Delta M/m_1$ were determined in accordance with standard tests for organic and clay soils / ASTM D2974 /. $\Delta M/m_1$ dried sediment samples were determined on the basis of weight loss on ignition .

Mineralization of the samples. The mineralization of the samples was carried out according to EPA Method 3052 procedure (1996) [34] . 1g air - dry soil to the nearest 0.001 g in PTFE vessels was weighted. HNO_3, HF, HCl and H_2O_2, were added using a microwave system Multiwave 3000 . The maximum power was 1400 W, and the maximum pressure in Teflon vessels - 40 bar .

Determination of iron and heavy metals in soils. In the determination of iron and heavy metals in the soil samples Atomno absorption spectrometer "AAnalyst 800 with graphite furnace HGA" Company "Perkin Elmer", at wavelengths: Fe - 248.3 nm, Pb - 217.0 nm, Cd - 228.8 nm, Cr -357,9 nm, Ni - 232.0 nm, Cu - 324.8 nm and Zn - 213.9 nm was used.

Determination of phosphorus in the soil. In determining the content of P in the soil samples UV / VIS DR 5000 spectrophotometer (Hach Lange, Germany), at a wavelength of P - 410 nm. (BDS ISO 11263:2002) was used[35].

Determination of the electrical properties of soils. The resistance, R (Ohm), and capacitance, C (F), of each soil sample were measured after placing the soil sample between two electrodes mounted in a conductometric cuvette. Alternating voltage of 1000 mv with frequency f (Hz) was imposed between the electrodes and the current and its phase difference to the voltage were measured by impedansomer. Using these data the values of R and C were determined based on an appropriate electric model of the sample. The model of resistor and capacitor, connected in parallel, was the most appropriate for the poorly conductive soil samples. The R and C of soil samples were measured at large number of frequencies in the range 0.001 Hz - 5 MHz using Solartron 1260A Impedance/Gain-phase analyzer (England) interfaced to a computer.

The values of R and C depend on sample material as well as on the geometry of electrodes. Instead of R and C it is more convenient to use another variables, electric conductivity σ (mS/m) and relative permittivity ε_r, which describe the intrinsic electrical properties of sample material and are independent on the electrode configuration. The dimensionless ε_r reflects the concentration of bound charges in the sample and the propensity of these charges to be displaced under the influence of an incident electric field. σ is proportional to the concentration of free charges in the sample and to their ability to travel a certain distance without collision with the particles of sample. At low frequencies the dissipation of energy depends primarily on σ, while at high frequencies (e.g. above 1 MHz) the dissipation of energy is dominated by the dielectric losses due to bound charges.

According to (Davey et al., 1992) σ and ε_r were calculated from equations (1) and (2)

$$\varepsilon_r = K.C/\varepsilon_o \quad \text{...} \ (1)$$
$$\sigma = K/R \quad \text{...} \ (2)$$

Here, ε_o = $8.854.10^{-l2}$ (F/m) is the dielectric permittivity of vacuum and K is the constant of the conductometric cuvette. In case of flat electrodes, distanced at d (m), each having the area of S (m^2), the K= d/S. In this case the conductometric cell had a cylindrical shape with a diameter of 17 mm and volume of 4.5 ml. The wall of the cylinder and a thin rustles rod in the axis of cylinder served as electrodes. Based on equation (2) the constant, K, was calculated equal to 1.55 cm^{-1} for the frequencies less than 1 MHz measuring the resistance of the conductometric cuvette filled with salt solution with known conductivity.

Prior to measuring the soil samples were dried and break to pieces with approximately the same size. As a control sample containing no organic material we used fine sea sand taken from the shore of the Baltic Sea (Poland) after subjecting it to heat treatment (300^{o}S, 15 min), washing with distilled water and drying. Distilled water with ε_r = 80 was used as control in measuring the dielectric permittivity of soil samples.

Accuracy and precision

Soil materials used for accuracy and precision tests include three certified soil samples corresponding to two main soil types in Bulgaria:

1. Light Alluvial–deluvial Meadow Soil PS-1, SOOMET No. 0001-1999 BG, SOD No. 310a-98.

2. Light Meadow Cinnamonic Soil PS-2, SOOMET No. 0002-1999 BG, SOD No.311a-98.

3. Light Alluvial–deluvial Meadow Soil PS-3, SOOMET No. 0003-1999 BG, SOD No. 312a-98.

SPSS (Statistical Package for Social Science) program for Windows was used for statistical data processing.

III. RESULTS AND DISCUSSION

The obtained values of some of the basic physic-chemical parameters- pH, $\Delta M/m_1$, P_2O_5, Fe_2O_3 and total heavy metals content (Pb, Cd, Cr, Ni, Cu and Zn) of the studied air-dried soil samples are presented in Table 1. The pH of the soil samples was in the range from medium acid (pH = 5.25) to slightly basic (pH = 7.73) with a mean value of pH = 6.71. The average iron content in the dry soil samples was 44352,50 mg/kg which is 114 times higher as compared with the average value of the total mass of the other investigated heavy metals(Pb, Cd, Cr, Ni, Cu and Zn). The mean value for P_2O_5 was 0,195%, and for $\Delta M/m_1$ – 4,561%. In the table, there were found lower values of ε_r at higher frequencies, which was due to frequency dispersion.

Table I: Principal physicochemical data collected for the soil probes under study

Sample	Soil type FAO	pH	ε_r, 10 kHz	ε_r, 100 kHz	σ (mS/m), 1 MHz	Fe_2O_3, mg/kg	Total heavy metals, mg/kg	P_2O_5, %	$\Delta M/m_1$, %
1	Fluvisols	7.30	11.30	6.90	1.05	49700	845.7	0.263	6.154
2		7.36	14.17	9.70	1.38	46800	948.2	0.258	8.692
3		7.28	10.53	7.22	0.57	29600	600.9	0.192	3.796
4		7.31	12.99	8.60	0.78	68800	1055.4	0.193	4.718
5		7.48	10.81	6.77	0.67	50900	1121.0	0.165	2.056
6		7.54	11.26	7.01	0.76	37100	802.6	0.166	2.089
7	Leptosols	6.41	11.04	8.06	0.88	38400	349.2	0.210	6.864
8		6.32	10.45	7.46	0.84	45200	404.0	0.156	7.077
9		6.11	10.15	7.34	0.77	44300	411.8	0.237	6.629
10		6.37	10.56	7.35	0.95	37000	303.4	0.187	6.390
11	Vertisols	5.55	10.36	7.40	0.92	51900	298.0	0.165	7.930
12		5.30	7.64	5.83	0.27	49600	519.5	0.147	1.546
13		5.34	9.16	6.98	0.51	53400	280.3	0.156	8.000
14	Chromic	6.11	9.75	7.18	0.75	42200	197.7	0.229	7.317
15		7.59	11.66	7.19	1.33	31500	196.9	0.192	8.964
16		6.72	10.18	6.78	0.93	47600	268.6	0.210	5.992
17		6.68	9.72	6.59	0.91	27900	213.1	0.229	6.024
18		6.61	11.04	7.12	1.10	45100	223.5	0.238	6.730
19		6.42	10.00	7.02	0.90	45400	325.4	0.256	7.467
20		6.54	9.35	6.76	0.72	53900	314.4	0.238	7.488
21		6.50	10.19	6.95	0.90	55000	388.7	0.238	8.309
22	Leptosols	5.82	4.02	3.52	0.08	19800	446.5	0.120	2.571
23		5.76	5.45	4.28	0.18	22700	149.6	0.170	2.281
24		5.25	4.12	3.52	0.10	22900	132.9	0.120	1.579
25		6.05	7.25	4.86	0.35	55900	165.7	0.165	2.040
26		6.98	8.67	5.73	0.39	58100	225.4	0.156	2.825
27		6.09	7.19	4.93	0.33	58100	385.2	0.137	2.264
28		6.79	5.26	4.14	0.19	41500	142.7	0.137	1.490
29		5.71	5.27	4.24	0.17	46300	163.9	0.165	1.694
30		6.66	5.39	4.25	0.19	39300	114.2	0.156	1.599
31		7.70	7.48	4.87	0.36	32800	181.8	0.210	1.886
32		7.68	11.30	7.12	0.69	42500	538.9	0.165	3.196
33		7.73	6.81	4.98	0.27	36400	222.3	0.192	1.795
34	Vertisols	7.41	9.37	6.12	0.57	45700	555.3	0.238	4.294
35		7.00	13.18	8.69	0.62	61500	272.5	0.229	4.901
36		7.01	14.20	9.19	0.72	62200	316.1	0.210	5.346
37		6.99	11.20	7.34	0.61	53700	341.9	0.210	4.409
38		7.58	11.21	7.34	0.54	42800	599.6	0.229	2.273
39		7.68	12.00	7.69	0.65	42400	279.9	0.229	2.896
40		7.70	12.07	7.80	0.60	38200	207.5	0.238	2.852

Max. value	7.73	14.20	9.70	1.38	68800	1121.0	0.263	8.964
Min. value	5.25	4.02	3.52	0.08	19800	114.2	0.120	1.490
Average	6.71	9.59	6.57	0.64	44352.50	387.75	0.195	4.561
Standard deviation	±0.75	±2.62	±1.52	±0.33	±11192.22	±255.24	±0.04	±2.473

The regression-correlation analysis displayed that the influence between the studied soil parameters was very slight). The obtained correlation coefficients were presented in Table 2 and they characterized with low values. Statistical significance was established only for the effect of heavy metals content on soil pH: r = 0,366 and determination coefficient R^2 = 0.134 at $p<0.05$. The results obtained corresponded to the data from other investigations [36,37,38].

Table II: Correlation coefficients between some of the principal physicochemical parameters (pH, $\Delta M/m_1$, P_2O_5, Fe_2O_3, total amount of heavy metals (Pb, Cd, Cr, Ni, Cu и Zn) of air-dried soil probes

Correlation	Correlation coefficient, r	Statistical significance
Fe_2O_3 as a function of the content of ΔM/m1	0.211	p > 0.05
Fe_2O_3 as a function of pH	0.044	p > 0.05
Fe_2O_3 as a function of the content of heavy metals	0.270	p > 0.05
pH as a function of the content of heavy metals	0.366	p <0.05
$\Delta M/m_1$ content as a function of the content of heavy metals	0.052	p >0.05
pH as a function of the content of ΔM/m1	0.097	p >0.05

The apparatus for measuring the dielectric parameters of the soil samples was calibrated by the use of materials with known parameters. The value for dH_2O ε_r was approximately 80 (10 kHz – 5 MHz) and varied sand and between 2.5 and 4.0 for the separate mineralized soils(500 Hz – 5 MHz). The value of ε_r increased monotonously, while that for each of the investigated soil samples when the frequency was reduced from 5 MHz to 1 Hz. These data agreed with the results of other authors [39,40,41] and emphasized the reability of the applied dielectroscopic method for the investigation of soil samples in the studied conditions. The values of ε_r and σ for each soil sample determined at selected current frequency are presented in Table 1.

Fig. 1 and Fig. 2 display the distribution diagrams of the couple values EPSr(10kHz) - $\Delta M/m_1$, % , EPSr(100kHz) - $\Delta M/m_1$, %, EPSr(10kHz) - P_2O_5, % and EPSr(100kHz) - P_2O_5, % for the studied soil samples. The plots show the presence of linear correlation between EPSr - P_2O_5, % , EPSr - $\Delta M/m_1$, % of the soil samples. A similar linear graphical distribution was defined in the rest of the cases when a correlation between the cited basic physic-chemical parameters and EPSr of the soil samples was examined. The strength of the established linear correlations between the basic physico-chemical parameters and the dielectric properties - ε_r and σ of the soil samples was assessed by calculation of the corresponding correlation coefficients presented in Table3.

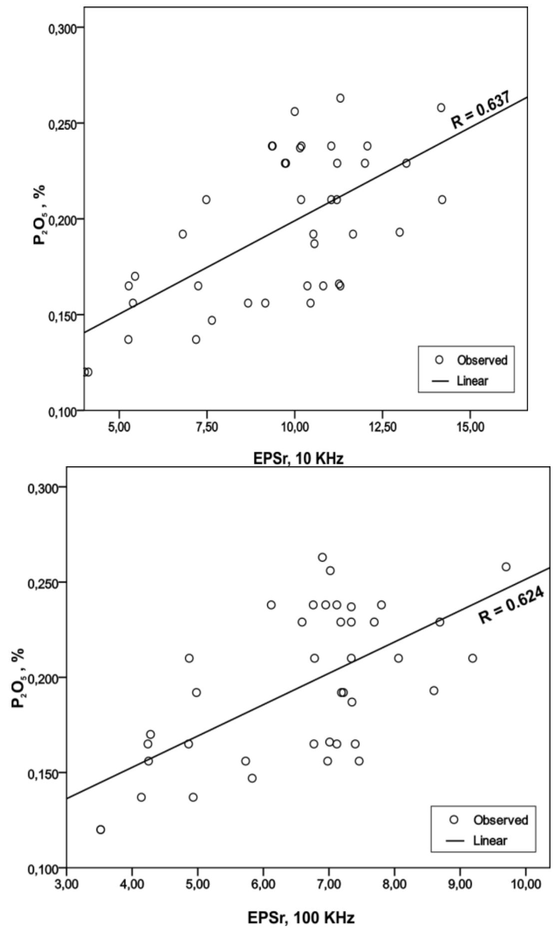

Figure 1: Statistical dependence between EPSr (10kHz) and P_2O_5 %; EPSr (100kHz) and P_2O_5 % of air tempted soil samples

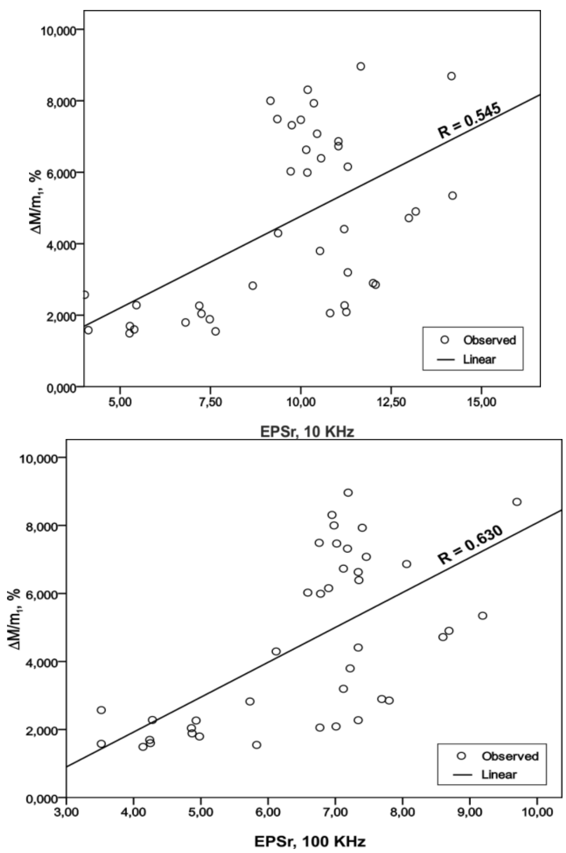

Figure 2: Statistical dependence of EPSr (10kHz) - $\Delta M/m_1$ %, EPSr (100kHz) - $\Delta M/m_1$ of air tempted soil samples

Table III. Correlation coefficients between the dielectric parameters ε_r (10 kHz) and σ (mS/m) and some of the principal physicochemical parameters (pH, $\Delta M/m_1$ %, P_2O_5, , Fe_2O_3, total amount of heavy metals (Pb, Cd, Cr, Ni, Cu и Zn)) of air-dried soil probes.

Correlation	Correlation coefficient, r	Statistical significance
ε_r, 10 kHz as a function of pH	0.530	$p < 0.05$
ε_r, 10 kHz as a function of Fe_2O_3 (mg/kg)	0.451	$p < 0.05$
ε_r, 10 kHz as a function of the content of heavy metals (mg/kg)	0.463	$p < 0.05$
$\Delta M/m_1$, % as a function of ε_r, 10 kHz	r = 0.545	$p < 0.01$
P_2O_5, % as a function of ε_r, 10 kHz	r = 0.637	$p < 0.01$
σ, 1 MHz as a function of pH	0.316	$p < 0.05$
σ, 1 MHz as a function of Fe_2O_3 (mg/kg)	0.203	$p > 0.05$
σ, 1 MHz as a function of the content of heavy metals (mg/kg)	0.336	$p < 0.05$
$\Delta M/m_1$, % as a function of ε_r, 100 kHz	r = 0.630	$p < 0.01$
P_2O_5, % as a function of ε_r, 100 kHz	r = 0.624	$p < 0.01$
σ, 1 MHz as a function of ε_r, 10 kHz	r = 0.794	$p < 0.01$
σ, 1 MHz as a function of ε_r, 100 kHz	r = 0.786	$p < 0.01$

The data from Table 3 displayed that the dialectical properties ε_r and σ of the dried soil samples wore moderately influenced by the studied basic soil parameters – pH, Fe_2O_3 content, P_2O_5, total organic compounds and total heavy metals content. The calculated correlation coefficients (r from 0,20 to 0,64) have moderately low values. The determined correlation coefficients with regard which estimate the strength of the linear correlation between the studied basic physic-chemical parameters of the samples and the dielectric properties (ε_r and σ) characterized with statistical significance ($p < 0.05$) except for that regarding to the correlation between σ and Fe_2O_3 of the samples ($p > 0.05$). Statistically greater effect ($p < 0.05$) on ε_r (10 kHz), ε_r (100 kHz) and σ (1 MHz) was established for P_2O_5(r = 0.637 and0.624, respectively), $\Delta M/m_1$(r = 0.545 and 0.63, respectively), pH (r = 0.53, respectively), Fe content (r = 0.45, respectively) and total heavy metals content (r = 0.46, respectively). The latter results demonstrated the necessity of additional investigation on soils with higher total organic compounds content, where higher correlation coefficients are expected.

The determined correlation coefficients displayed that each of the studies basic physic-chemical parameters could be independently analyzed in the range of 20% to 40% from the variation of the proper dielectric parameter, ε_r and σ of the soil samples. In total, however, as independent between each other (Table 2), they could explain practically the entire interval of statistical variation of the dielectric properties of the soils.

According to the data from Tables 1,2 and 3, each of the studied basic soil parameters influences individually and independently on the dielectric soil properties. As stated, the latter could explain the determined moderately low correlation coefficients. More informative is the frequency dependence of σ as ε_r of the soil samples was influenced in a similar way by the strong dielectric polarization of the organic compound and the studied salts.

IV. CONCLUSIONS

The determined correlation coefficients displayed that each of the studies basic physic-chemical parameters could be independently analyzed in the range of 20% to 40% from the variation of the proper dielectric parameter, ε_r and σ of the soil samples. In total, however, as independent between each other, they could explain practically the entire interval of statistical variation of the dielectric properties of the soils.

More informative is the frequency dependence of σ as ε_r of the soil samples was influenced in a similar way by the strong dielectric polarization of the organic compound and the studied salts.

The obtained physicochemical correlations by means of the IP method with a different extend of correlation could be applied for solving of various scientific and practical tasks to predict and control the soil characteristics.

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