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STATISTICAL APPROACH
TO THE DETERMINATION OF THE INDEX
OF SOIL SUPPLENESS ON REDUCTION

STATYSTYCZNE PODEJŚCIE
DO OKREŚLANIA WSKAŹNIKA PODATNOŚCI GLEBY
NA REDUKCJĘ

Abstract: The decrease in the redox potential in time can be determined on the basis of measurements in the aqueous soil suspension under standard laboratory conditions and time limits, as well as expressed as an index of soil suppleness on reduction (SSR index). Assuming that the SSR covers certain aspects of the relatively stable characteristics of the soil or soil properties, it can be practically considered as a useful indicator of the soil quality and fertility. The main aim of this study was to present a statistical method for determination of soil suppleness on reduction. The most practically useful statistical method to determine the soil susceptibility to reduction was the difference between the maximum and minimum Eh values obtained in the experiment. The Eh decrease values were within the range from 184 mV/week to 70 mV/week, while the soil suppleness on reduction index soil suppleness on reduction index (SSR index) for three soils tested was in the range from 25 to 10 mV/day. Results of statistical calculations and considerations upon the determination of soil suppleness on reduction index value based on the redox potential measurements in the soil-water suspension under laboratory conditions during a one week period, made it possible to significantly simplify the practical determination of the SSR index value.

Keywords: Eh measurements in soil, pH measurements, statistical calculations, soil suppleness on reduction index

Introduction

Phenomenon of the redox potential in soil refers to the soil solution and the extensive literature on redox potential studies in the soil was considered in different directions such as explaining the essence of the phenomenon of redox processes in the soil.

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Research upon the associations between different constants of soil properties and redox potential, identifying the relationships between variable soil conditions and redox potential, as well as a lot of attention was also paid to methodological aspects of redox potential measurements in the soil [1].

The redox potential (Eh) measurements actually show the current state of the redox potential in soil or in the soil-water medium and only at the time of measurement. However, measurements of redox potential in the aqueous suspension of soil under standard laboratory conditions and particular time limits, can be a base for determining the redox potential decrease over time and express it as an indicator of soil suppleness on reduction (SSR index). Assuming that SSR index covers certain aspects of the relatively stable characteristics of the soil, *ie* soil properties, it can be considered as practically useful indicator of soil quality and fertility [2]. The main aim of this study was to present statistical method for determination of soil suppleness on reduction index.

Methods

The research was conducted in the laboratory using three soil samples representatively collected from the topsoil of three arable fields near Lublin in 2012. Soil samples were dried under natural conditions and sieved through a plastic sieve with a diameter of 1 mm. The basic properties were determined in the soil samples. Distribution of soil particles was determined by means of Cassagrande's areometric method modified by Proszynski. Organic carbon was analyzed by Tiurin's method due to oxidation of soil organic matter in concentrated H_2SO_4. Total nitrogen was determined by Kjeldahl's method after digestion in concentrated H_2SO_4. The contents of extractable Fe and Mn were determined according to Rinkis after extraction with 1 mol HCl dm^{-3}, and the measurement of elements was performed by means of atomic absorption spectrometry applying Hitachi Z-8200 device. All chemical analyzes were performed in 4 replicates. The ratios of determined elements as molar ratios of elements were also calculated based on the amount of substance in the system of a given element to the subsequent one. The study included pH and redox potential measurements (Eh) and statistical experimental unit consisted of the soil suspension in a 100 cm^3 capacity beaker, prepared from 50 g of air-dried soil and 50 cm^3 of distilled water, *ie* at the soil to water ratio equaled to 1 : 1. Air temperature in the laboratory during measurements was about 25 ^{o}C. The pH measurements were made by means of potentiometer applying P-730 device provided with automatic temperature compensation and combined electrode ESAgP-30IW. Measured pH values were expressed as real values of pH and also as recalculated onto H^+ based on the dependence between pH and H^+, *ie* hydrogen ions concentration [3].

Redox potential measurements were made using P-730 device with the range of ±960 mV, and provided with platinum electrode of 5 mm length and 0.5 mm diameter, against calomel electrode EK-602. Polarization time of Pt electrode was 300 seconds. Eh values were calculated by adding to the value of redox potential, constant value of

calomel electrode potential that for the conditions adopted in the experiment was +245 mV.

The pH and Eh measurements were carried out for three soils in 4 replicates and 8 terms of measurements, *ie* after 1 hour and then every 24 hours, *ie* after 1, 2, 3, 4, 5, 6, and 7 days of incubation; the whole experiment was repeated 5 times. Therefore, the experimental design consisted of 3 soils in 4 replicates for 8 measurement terms and in 5 replicates of the whole experiment. Thus the total number of experimental data of pH and Eh was $n = 480$.

Experimental data of the H^+ and Eh were calculated according to analysis of variance and *Tukey's-test*. Selected statistical characteristics were also calculated:

1. Arithmetic mean:

$$\bar{y} = \frac{\sum y}{n}.$$

2. Confidence interval $\bar{y} - L < \mu < \bar{y} + L$, where $\pm L$ is:

$$L = t_\alpha \sqrt{\frac{nS^2}{n(n-1)}},$$

where t_α is a value from t-Student's table *t-distributions* at the significance level of $\alpha = 0.05$, sum of squares:

$$nS^2 = \sum y^2 - \frac{\left(\sum y\right)^2}{n}$$

was calculated for the sample size of n, and degree of freedom $v = n - 1$.

3. Standard deviation $S = \sqrt{S^2}$ was calculated as the square root of variance:

$$S^2 = \frac{\sum y^2}{n} - (\bar{y})^2.$$

4. Standard error for mean value:

$$SE_{\bar{y}} = \frac{S}{\sqrt{n}}.$$

5. Minimum value *Min*.
6. Maximum value *Max*.
7. Range between maximum and minimum values *Range = Max − Min*.
8. Variance coefficient was calculated as a ratio of standard deviation to arithmetic mean $CV = S / \bar{y}$ expressed in %.
9. The linear regression equation $y = b_{yx} + c$ for selected measurement characteristics.
10. Determination coefficient R^2 [4].

Results

Soils in presented laboratory experiment were little diverse referring to their physical properties, although soils No. 1 and No. 3 were sandy loam, while soil No. 2 was silt loam. In contrast, soils were more diverse in terms of chemical properties such as pH, as well as the content of organic carbon, total nitrogen, and contents of extractable Fe and Mn. The characteristics of soils also shows the calculated ratios of determined elements as an additional feature of the soil chemical properties, *ie* ratios of elements calculated on a base of the significant and particular property of matter that is variable molecular weight of elements. Therefore, this method of calculating the molar ratios of elements may be considered as one and only correct way for calculating the ratios of chemical elements (Table 1).

Table 1

General properties of soils in the experiment

Feature	Soil		
	No. 1	No. 2	No. 3
Soil fraction and particle size [%]			
Sand $\varnothing$ 2–0.02 mm	66	71	74
Silt $\varnothing$ 0.02–0.002 mm	30	23	23
Clay $\varnothing$ < 0.002 mm	4	6	3
The pH index in 1 mol KCl pH [-]			
Value of pH	4.6	5.5	6.0
Element content [mg/kg]			
Organic-C	8585	7135	7773
Total-N	1876	1568	1764
Extractable-Fe	1386	792	853
Extractable-Mn	223	132	182
Elements ratio	Molar ratio [-]		
C/N	5	5	5
C/Fe	29	4	42
C/Mn	176	247	195
N/Fe	5	1	8
N/Mn	33	47	38
Fe/Mn	6	59	5

Biological processes associated with decreasing the oxygen content in the soil-water suspension increased the advantage of reduction processes, which can be represented as $Fe(OH)_3 + e^- \leftrightarrow Fe(OH)_2 + OH^-$. Table 2 presents ranges of the pH value, for which every range was represented by sample size of $n = 20$.

Minimum and maximum values of the pH in the soil-water suspension
during subsequent terms of measurement

Soil No.	Term of measurement							
	Hour 1	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
	pH [-]							
1	4.67–5.08	4.99–5.35	5.18–5.74	5.37–5.79	5.46–5.97	5.52–5.97	5.72–6.09	5.72–6.15
2	5.71–6.28	6.08–6.40	6.14–6.64	6.26–6.64	6.37–6.60	6.37–6.63	6.48–6.73	6.48–6.75
3	6.16–6.84	6.48–6.84	6.58–7.00	6.65–7.00	6.79–6.97	6.79–6.99	6.83–7.03	6.84–7.10

Oscillations of pH values were slightly variable in subsequent measurement terms, and during the whole experiment, differences between the ranges for soils and terms were significant, although values of the pH ranges varied with a clear tendency of pH
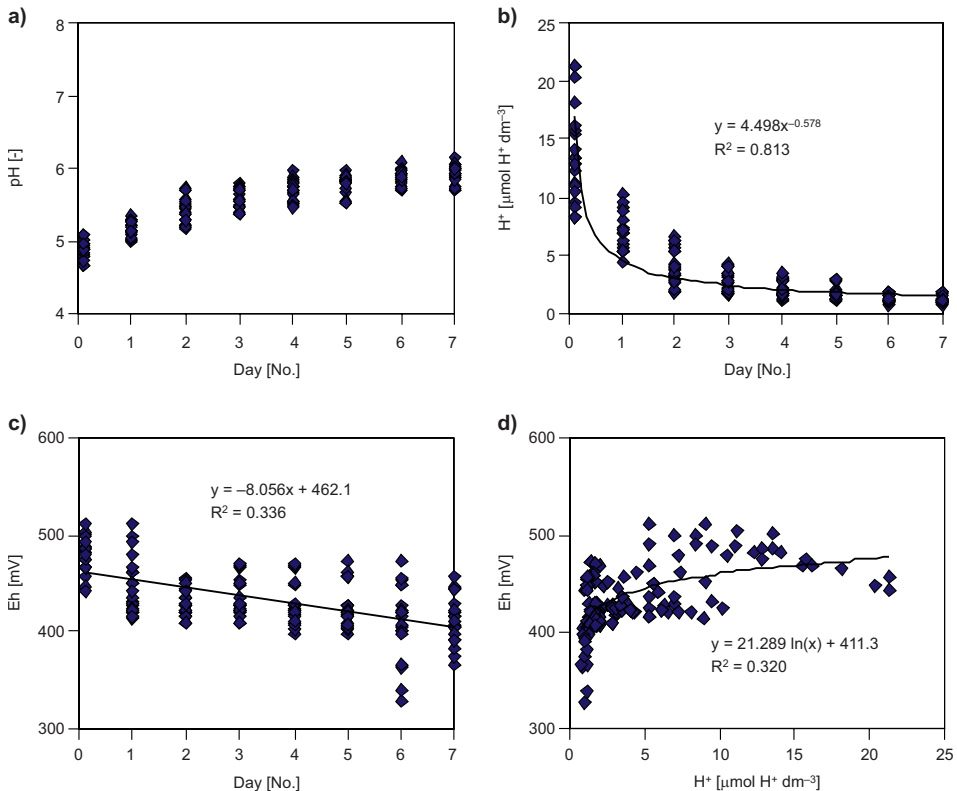

Fig. 1. Dependencies and associations among examined measurement features in soil-water suspension for the soil No. 1 at the sample size $n = 160$; a) Dependence between measurement term in days and pH index, b) Relationship between measurement term in days and H^+ ions concentration, c) Relationship between measurement term in days and Eh values, d) Relationship between H^+ ions concentration and Eh values

increase, which was illustrated for three tested soils in Fig. 1a, Fig. 2a, and Fig. 3a. Quantitative representation of the decreasing the hydrogen ions concentration H^+ and possibility of calculating the $f(x)$ was presented in Fig. 1b, Fig. 2b, and Fig. 3b, where relatively wide range of H^+ in three experimental soils can be seen. Associations between pH values and Eh values can be also compared in Fig. 4 for soils No. 1, No. 2, and No. 3 as well as calculated data for the relationship $f(x)$ between H^+ concentration and Eh values in Fig. 1d, Fig. 2d, and Fig. 3d. These graphs show that such features as pH and Eh may be compared and present altogether, while calculation of $f(x)$ may be performed only when the pH values are converted into the hydrogen ions concentration H^+. Ranges of the pH values presented in Table 2 also indicate data such as pH cannot be statistically calculated. The explanation would be three theses presented without proving them. First: pH 4 is not 4, and pH 8 is not 8. Second: mean for pH 5 and pH 7 is not pH 6. And third: difference between pH 5 and pH 6 is not pH 1. Anyone can easily verify by reviewing the scientific literature in serious and well-known scientific journals that statistical calculations using pH are simply very often incorrect [5].
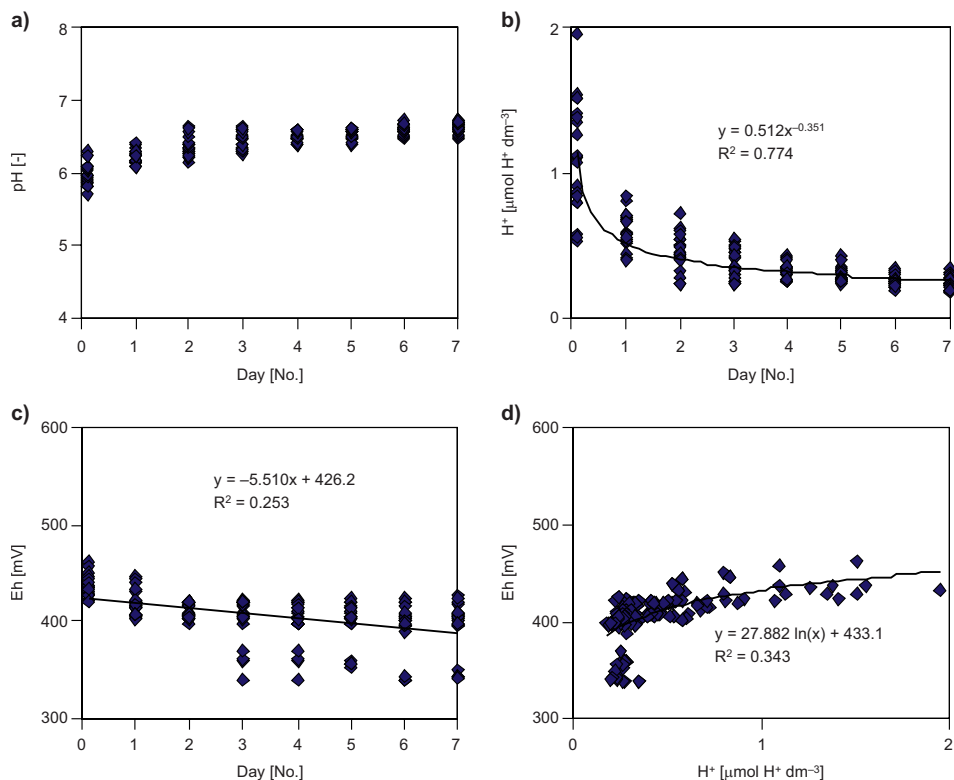

Fig. 2. Dependencies and associations among examined measurement features in soil-water suspension for the soil No. 2 at the sample size $n = 160$, a) Dependence between measurement term in days and pH index, b) Relationship between measurement term in days and H^+ ions concentration, c) Relationship between measurement term in days and Eh values, d) Relationship between H^+ ions concentration and Eh values

a)

b)

$y = 0.219x^{-0.342}$
$R^2 = 0.789$

c)

$y = -2.184x + 413.2$
$R^2 = 0.198$

d)

$y = 9.504 \ln(x) + 422.5$
$R^2 = 0.184$

Fig. 3. Dependencies and associations among examined measurement features in soil-water suspension for the soil No. 3 at the sample size n = 160, a) Dependence between measurement term in days and pH index, b) Relationship between measurement term in days and H^+ ions concentration, c) Relationship between measurement term in days and Eh values, d) Relationship between H^+ ions concentration and Eh values
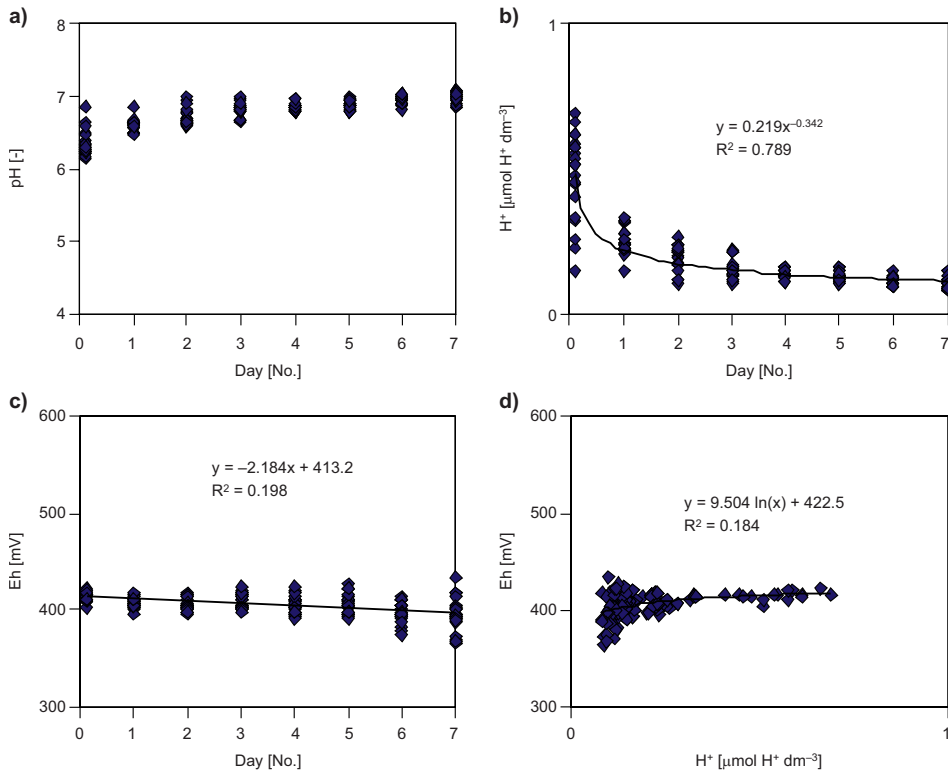
Table 3 presents the H^+ concentration in soil-water suspension under the influence of experimental treatments.

Table 3

Concentration of the H^+ ions in the soil-water suspension during subsequent terms of measurement

| Soil	Term of measurement								Mean
	Hour 1	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	
	H^+ [μmol H^+ dm^{-3}] for sample size $n = 480$								
No. 1	13.8032	6.8614	4.0537	2.7692	2.0878	1.7136	1.3830	1.2322	4.2380
No. 2	1.1090	0.6070	0.4573	0.3747	0.3171	0.2938	0.2564	0.2390	0.4568
No. 3	0.4758	0.2515	0.1949	0.1537	0.1394	0.1305	0.1120	0.1000	0.1952
Mean	5.1293	2.5733	1.5686	1.0992	0.8481	0.7126	0.5838	0.5237	1.6298
LSD at p = 0.01, for soils 0.2617, for terms 0.5538, for soil × term 1.1202									
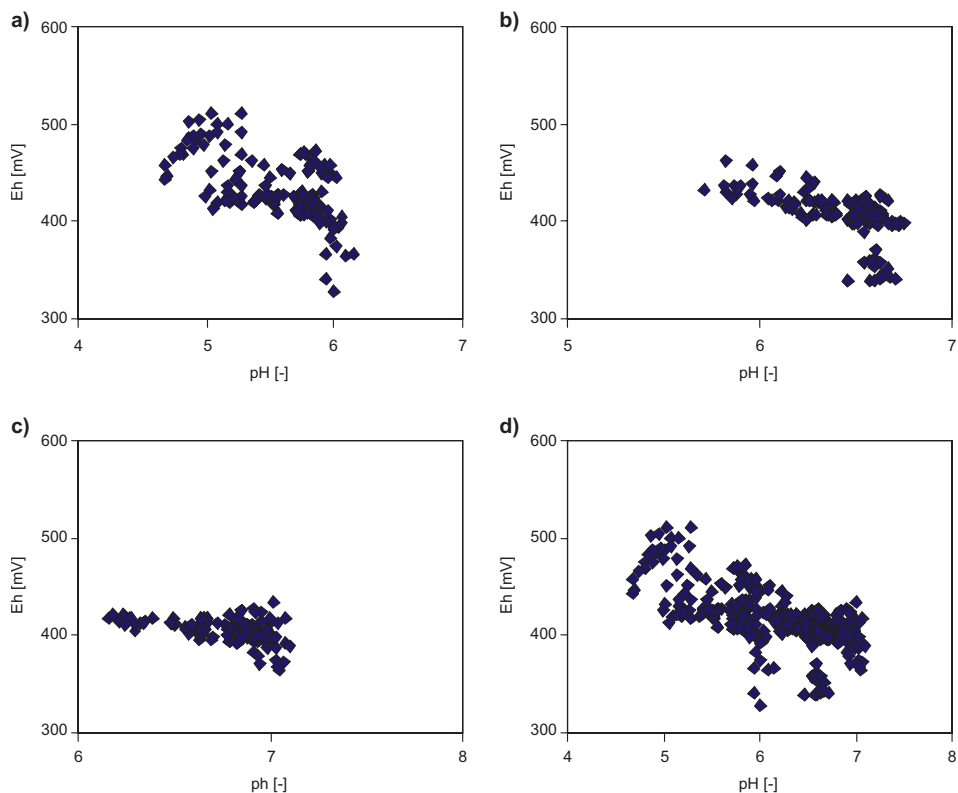
Fig. 4. Dependencies between pH and Eh values in soil-water suspension for examined soils and sample size
$n = 160$: a) No. 1, b) No. 2, c) No. 3 as well as d) for three soils (No. 1, No. 2 and No. 3 altogether)
and sample size $n = 480$

Values of hydrogen ions concentration H^+ were significantly varied especially between soils No. 1 and No. 2, and in majority the H^+ values were also significantly diversified between soils No. 2 and No. 3. Considerable differences between measurement terms occurred just after several days of incubation. The results for H^+ can be also recalculated onto pH, but such presenting of pH values would, however, be only of the demonstration importance with a very clear stipulation, that the calculated value of the least significant difference (LSD) expressed in the concentration of hydrogen ions H^+ cannot ever be converted into the pH value, because it would be completely incorrect.

Table 4 presents statistically calculated Eh values for 3 soils and 8 measurement terms. The highest significant mean Eh value was found for soil No. 1, while no significant differences were recorded between mean values for soils No. 2 and No. 3. There were significant differences between mean values of Eh in the subsequent measurement terms, but most of them were observed rather in the initial measurement terms and a few days intervals; these differences were smaller in the final terms of measurement. It can be also assumed that the least significant difference for the

interaction of soil × term of 22.5 mV, can be the Eh value indicate for a significant difference for each mean of Eh values in the soil-water suspension, but with the restriction that this value is characteristic only for this experiment.

The Eh values in soil-water suspension during subsequent terms of measurement along with statistical characteristics

Soil	Term of measurement								Mean for soil
	Hour 1	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	
	Eh [mV] for sample size $n = 480$								
No. 1	470.7	455.3	429.7	434.0	428.7	427.6	411.6	411.9	433.7
No. 2	435.9	419.6	411.4	403.0	398.0	398.8	393.1	399.4	407.4
No. 3	414.5	407.4	405.9	409.4	405.9	407.2	398.4	394.7	405.4
Mean	440.4	427.5	415.7	415.5	410.8	411.2	401.0	402.0	415.5
LSD at p = 0.01, for soils 5.2, for terms 11.1, for soil × term 22.5									

The list of Eh values in Table 4 best illustrates the average dynamics of the redox potential decrease both for soils and measurement terms. Nevertheless, calculated statistical characteristics for the soils are appropriate statistical evaluation of all Eh measurements in soil-water suspension during the incubation (Table 5).

Statistical characteristics of the Eh values in soil-water suspension for experimental soils

Statistics	Soil		
	No. 1	No. 2	No. 3
	Eh [mV] for sample size $n = 160$		
Mean for soil*	433.7	407.4	405.4
Confidence interval	± 4.9	± 3.9	± 1.7
Standard deviation	31.7	24.9	11.2
Standard error	2.5	2.0	0.9
Minimum	327.6	339.2	364.6
Maximum	511.7	462.2	434.2
Range	184.1	123.0	69.6
Coefficient of variation	7 %	6 %	3 %

* See Table 4.

Under natural circumstances of the soil environment, *ie* in the system with dynamic air-water conditions, redox potential measurements can be performed, but the soil must be moist in order to the measurement to be possible. In the soils moisturized with water,

the redox potential values change rapidly and reach values indicating the current state of oxygenation or reduction. However, in the simulated soil solution at a predetermined ratio of soil to water, reducing conditions at the level of 332 mV can be achieved after several days of incubation at a specific temperature [6]. The decrease of redox potential values in the period, e.g. one week, may be an index of soil susceptibility on reduction as a important indicator for physical, chemical, and biological soil properties. To simplify the determination of such SSR index, only two series of measurements Eh should be conducted. The first, one hour after preparation of the soil-water suspension, and the second measurement after 7 days, *ie* one week of incubation. Of course, the pH measurement could be some additional characteristics of the soil. However, a research should be made at first to be able to develop an interpretation of these Eh and pH measurements.

All the results from chemical analyzes and measurements are more or less variable, which is related to methods, apparatus, and conditions, hence all analytical data should be statistically processed [7]. Redox potential measurements, even in a relatively homogeneous environment such as soil and water suspension, are so variable that a statistical processing of Eh values is actually necessary. Table 5 presents eight important statistical characteristics referring to Eh values achieved during the whole experiment (Table 4). Among statistical measures listed in Table 5, two groups can be distinguished. The first consists of the position measures: arithmetic mean, confidence interval, and standard deviation. Second includes the variability measures: standard deviation and variability coefficient. The only relative measure among all statistical characteristics is variability coefficient that is suitable for comparisons of variability of features expressed in different units.

Table 6 illustrates statistical characteristics of Eh values that can be a basis for determination of the soil suppleness on reduction index.

Table 6

Methods for determining the soil suppleness on reduction index based on statistical characteristics

Statistical way	Soil			Mean
	No. 1	No. 2	No. 3	
	Decline of the Eh [mV] per week			
First and last*	58.8	36.5	19.7	38.3
Range**	184.1	123.0	69.6	125.6
Coefficient b_{yx}***	8.0	5.5	2.1	5.2
Mean	83.6	55.0	30.5	56.4

* Difference between the first and last mean Eh value for measurement terms, see Table 4; ** Range as the difference between maximum and minimum of Eh value for all measurements, see Table 5; *** Value of regression coefficient b_{yx} for linear equation $y = b_{yx} + c$ describing Eh features and measurement terms, see Fig. 1c, Fig. 2c, Fig. 3c.

First: 'First and last', *ie* the difference between the first and the last mean Eh value measured for terms from Table 4. Second: 'Range', *ie* the difference between maximum

and minimum values of Eh for all redox potential measurements in soil-water suspension during a one week. Third: 'Coefficient b_{yx}', *ie* value of regression coefficient b_{yx} from the linear equation describing Eh values and measurement terms. Comparison of the average decline in the redox potential values for the three approaches to determine the soil suppleness on reduction shows good agreement of the trend for all soils (Table 6).

In general, the largest values of redox potential decrease were recorded in soil No. 1, smaller in soil No. 2, while the smallest in soil No. 3 for all three ways. The lowest values of the redox potential decrease were observed for the soil suppleness on reduction determined on a base of the regression coefficient b_{yx}. Higher values occurred for the process of determining the difference from the first and last average Eh value for the measurement terms. Therefore, it can be assumed that both the 'First and Last', and 'Coefficient b_{yx}' methods are not useful to determine the soil suppleness on reduction index. 'Range', as the difference between maximum and minimum Eh values obtained during the experiment, was the most practically useful statistical way to determine the soil suppleness on reduction. Approximate value of Eh decrease for soil No. 1 was 184 mV/week, for soil No. 2 was 123 mV/week, and for soil No. 3 was 70 mV/week. Therefore, the SSR index for soil No. 1 was expressed in a decrease of 25 mV/24, for soil No. 2 the drop was 18 mV/24 hours, and for soil No. 3 this fall amounted to 10 mV/24 hours.

The results in Table 1 and Table 6 may suggest – because it was not a subject of the research – that the sequence of soil suppleness on reduction for the experimental soils lines up as follows: No. 1 > No. 2 > No. 3, which means that soil No. 1 is characterized by the highest susceptibility to reduction and in addition, this soil contained the highest quantities of organic carbon, total nitrogen, as well as extractable Fe and Mn. It may also indicate that the soil suppleness on reduction index may be compatible with chemical properties of the soil, which would give a greater chance to accept the SSR ratio as one of many important indicators of soil properties.

Figure 4 shows the dependence between the values of pH and Eh for soils used in the experiment, and the overall dependence between pH and Eh for 3 soil altogether. The Eh value ranges are different in reference to Eh scatter, although they cover a similar range for the three experimental soils between 300 and 500 mV. Due to the comparison of examined dependences between pH and Eh, such dependencies cannot be calculated statistically, but of course they can be graphically assembled to be able to see how these dependencies run. Thus, the pH ranges in soil No. 1 were mainly from pH 4.5 to pH 6, in soil No. 2 from pH 5.5 to pH 7, and in soil No. 3 from pH 6 to pH 7, *ie* from pH 4.5 to pH 7 for a total of three soils examined. Dependencies between pH and Eh described in literature conclude that with the silent assumption this value is –59 mV/pH [8], meanwhile, such value of decline in the redox potential expressed in mV, when the pH changes by 'unity' between of two pH values, what from a statistical point of view is incorrect.

And here, a simple theory without any evidence may be announced: the concentration of hydrogen ions in the solution expressed in mol H^+ dm^{-3} for the range between pH 4 to pH 5, are completely different values the H^+ than in the range between

pH 5 to pH 6, and further than in the range from pH 6 to pH 7, as well as than from pH 7 to pH 8, and thus for subsequent intervals between pH values, there are indeed different the H^+ values for each 'space' between certain values of pH index [6].

Conclusions

Presented results of statistical calculations and considerations upon determination of the soil suppleness on reduction index based on measurements of redox potential the soil-water suspension under laboratory conditions during a period of one week, made it possible to significantly simplify the practical determination of the SSR index.

Practically, it is possible on the basis of only two series of independents of Eh measurements in soil suspension, *ie* the first: the five Eh measurements after one hour of incubation, and the second: the five Eh measurements in the same soil suspension after 7 days of incubation.

Determination of the range between the maximum and minimum values for all redox potential measurements in soil-water suspensions, can be accept as a good enough way of determining the index of soil suppleness on reduction in laboratory conditions.

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STATYSTYCZNE PODEJŚCIE DO OKREŚLANIA
WSKAŹNIKA PODATNOŚCI GLEBY NA REDUKCJĘ

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Abstrakt: Na podstawie pomiarów potencjału redoks w wodnej zawiesinie glebowej w standardowych warunkach laboratoryjnych i określonych terminach pomiarów można określić spadek wartości potencjału redoks w czasie i wyrazić to jako wskaźnik podatność gleby na redukcję (SSR index). Przyjmując, że wskaźnik podatności gleby na redukcję obejmuje pewne aspekty względnie stałych cech glebowych, czyli właściwości gleby to może być rozważany jako praktycznie przydatny wskaźnik jakości i żyzności gleby. Głównym celem pracy było zaprezentowanie statystycznego sposobu wyznaczania wskaźnika podatności

gleby na redukcję. Najbardziej praktycznie przydatny statystyczny sposób do określania podatności gleby na redukcję to była różnica między maksymalnymi a minimalnymi wartościami Eh uzyskanymi w doświadczeniu. Wartości spadku Eh były w przedziale od 184 mV/tydzień do 70 mV/tydzień. A wskaźnik podatności gleby na redukcję SSR index dla trzech gleb testowanych był w przedziale od 25 do 10 mV/dobę. Przedstawione wyniki obliczeń statystycznych i rozważań nad określeniem wartości wskaźnika podatności gleby na redukcję na bazie pomiarów potencjału redoks w zawiesie gleba–woda w warunkach laboratoryjnych w okresie jednego tygodnia, dały możliwość znacznego uproszczenia praktycznego określania wartości wskaźnika podatności gleby na redukcję (SSR index).

Słowa kluczowe: pomiary Eh w glebie, pomiary pH, obliczenia statystyczne, wskaźnik podatności gleby na redukcję