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Comparison of two- and three-electrode systems in measurements of potential oxygen flux density in soil*

A. Bieganowski

Institute of Agrophysics, Polish Academy of Sciences, Doświadczalna 4, P.O. Box 201, 20-290 Lublin 27, Poland

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Abstract. This paper presents a comparison of the results of potential flux of oxygen in soil, determined by the voltammetric method, OFD, with the use of a three-electrode set of electrodes in the system of potentiostat, and a symmetrical two-electrode set of platinum electrodes. The studies have been performed on artificially prepared saturated or nearing saturation soil samples, representative for mineral soils in Poland. The discussion of the results obtained leads to the conclusion that a possibility exists to use a symmetrical two-electrode system to determine the potential flux of oxygen in soil; however, uncertainty of the results of measurements performed with the use of the voltammetric method, when the classic three-electrode system is used, is smaller than in the case of application of a symmetrical two-electrode system. Regardless of the system of electrodes used (symmetrical two-electrode or three-electrode with the system of potentiostat), the obtained values of OFD should be treated as indicators and not as measures of oxygen availability for plant roots.

Keywords: soil aeration, soil oxygen flux, ODR, OFD

INTRODUCTION

Monitoring of the oxygenation conditions in soil is important, taking into consideration the possibility of the occurrence of aeration stress. Oxygen deficiency in soil can limit or completely stop plant roots growth, and in special cases it can even cause their atrophy. The phenomenon of oxygen deficiency is especially important in soils which are permanently or temporarily waterlogged (Przywara and Stępniewski, 2000).

Monitoring of oxygenation can be done in many different ways (Gliński and Stępniewski, 1985). The most important of them are: measuring the concentration of oxygen, and determination of the potential density of oxygen flux in soil. The latter mean is especially significant, because it informs about the availability of oxygen for plant roots (defined as a resultant of oxygen concentration in the air/soil solution and the possibility of its replenishment through diffusion after its consumption, *eg* by the plant roots or soil organisms) (Malicki, 1990).

The use of an amperometric method for the evaluation of the potential flux of oxygen in soil was proposed by Lemon and Erickson in the middle of the fifties of the previous century (Lemon and Erickson 1952; 1955). The potential flux of oxygen in soil (oxygen diffusion rate - ODR) was evaluated by them from the current of reduction of molecular oxygen on the negatively polarized (- 0.65 V) platinum cathode (the anode was a saturated calomel electrode).

Due to the ambiguous determination of the real potential of a platinum cathode in relation to the soil solute (the measurement was performed in the two-electrode system) under a specific voltage applied to the electrodes, this method became a subject of criticism (Mc Intyre, 1970).

The problem of precise control of the cathode potential was solved by Malicki and Walczak (1983) by the incorporation of a third electrode (a reference electrode) in the measuring system, which, by the system of potentiostat, made it possible to control the potential of the platinum cathode. The application of the three-electrode system brought, however, new technical problems. To increase the precision of measurement, the comparative electrode (most frequently a saturated calomel electrode or a chlorosilver electrode - *ie* electrodes which have a construction based on a glass body) should be situated as close to the cathode as possible. This is inconvenient, due to the fact that it is difficult, from the technical point of view, to insert an

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Corresponding author's e-mail: biegan@demeter.ipan.lublin.pl

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electrode into some kinds of soil characterized by high density, and the situation of the comparative electrode close to the cathode causes each time a considerable disturbance of the environment measured.

The next stage of the work connected with the improvement of electrochemical methods of evaluation of the potential oxygen flux in the soil was the proposal, by Malicki and Bieganowski, of the voltammetric method *ie* the determination of the discussed quantity on the basis of the analysis of the current-voltage curve. Similarly as in the amperometric method, the reduction current of the molecular oxygen dissolved in the soil solution is measured in this method. The difference is that the potential of the platinum cathode is not established on a specific level but it is changed in a linear manner towards the negative values. This way a possibility exists to register the whole current-voltage curve whose shape is next analyzed and integrated, and the integer value obtained is converted into the potential density of oxygen flux in soil. To distinguish the results obtained by the voltammetric method, these values are called oxygen flux density - OFD values (Malicki and Bieganowski, 1999).

The determination of results on the basis of the analysis of the set of points on the current-voltage curve (not from an individual measuring point specified by the potential value and a current registered for this potential, as was done before) has the following adventages:

– it enables preliminary discarding of recordings referring to soils with too small a water content, which cannot be interpreted in the category of measurement of potential oxygen flux in soil (the curve, being a graphical representation of the $I(U)$ dependence, does not possess a quasiplateau area, *ie* such a range of potentials that the current of the measuring system is mainly limited in it by the possibility of diffusive supply of oxygen to the cathode surface) (Bieganowski and Wolińska, 2001).

In amperometric measurements there is a lack of a criterion for the preselection of results being not interpretable because of insufficient soil water content:

– it enables the rejection of results which, because of the high concentration of hydrogen ions, cannot be interpreted in the category of measurement of the potential oxygen flux in soil (at low pH, the reduction of hydrogen ions in the electrolysis reaction of water starts in the same range of the potential at which the particles of oxygen reduce - this causes an overestimation of the reduction current of oxygen and leads to a big error in the calculated value of the potential oxygen flux in soil) (Mc Intyre, 1970).

In amperometric measurements there is a lack of such information (unless additional investigations are performed) which would enable the rejection of results uninterpretable because of too high acidity of soil:

– preliminary investigations, realized in water solutions (the model system enabling to obtain fully repeatable measuring environment) and on chosen soils, show that the results obtained by the voltammetric method are characterized by better precision (Bieganowski, 2003).

When analyzing the essence of measurements with the application of the voltammetric method, one more advantage can be expected, important from the practical point of view. Because the voltammetric curve is registered, the problem of precise control of the cathode potential becomes insignificant. The lack of this control will result in the quasiplateau range ‘shifting’ along the potential axis. It means that, depending on *eg* the water content and/or pH, the quasiplateau can, in such a case, occur at higher potential values, and otherwise at lower potentials. However, it is not a problem because when the whole curve is recorded, the proper range of the potential can be selected. The consequence of this is the possibility of returning to the measurement in the two-electrode system. However, as distinct from the set of electrodes used by Lemon and Erickson, in which the anode was a saturated calomel electrode (Lemon and Erickson, 1952), the use of an anode made of a first type conductor seems to be possible.

The aim of this paper is a comparison of the results obtained with the use of the symmetrical two- and three-electrode measuring system in the context of application of the symmetrical two-electrode system for the evaluation of the potential oxygen flux in soil by the voltammetric method.

MATERIAL AND METHOD

The studies were performed on soil material taken from the soils specified in the Soil Samples Bank Representing Mineral Soils of Poland (Gliński *et al*., 1991). The description of the selected properties of the soil material studied is contained in Table 1.

The collected soil material was dried at room temperature to reach the air dry state. Then, the soil aggregates were crumbled with a hammer and the lot was screened through a sieve having the mesh of 2 mm.

The soil material prepared this way was wetted with distilled water. By mixing it carefully, the saturation state was obtained. The saturated soil material was placed in small portions in plastic pots (having the diameter of about 6 cm and the height of about 9 cm). When filling the pots, special care was taken to ensure the maximum possible homogeneity of the bulk density. The water content values referring to the weight (determined using the gravimetric method) of the soil samples obtained this way are presented in Table 1. For each soil material, the saturated samples were prepared as well as two samples with water content close to saturation (a total of 76 soil samples).

The current-voltage curves were registered with a voltammeter produced by ForTest, applying three- and two-electrode sets of electrodes.

The three-electrode set consisted of a platinum cathode (platinum wire – purity of 3n, diameter of 0.5 mm, length of

Table 1. Description of the investigated soil samples

Soil	Number in Bank of Soil Samples*	Symbol of soil formation	Pleace of soil sample uptake	FAO		
				sand 2–0.02	silt 0.02–0.002	clay <0.002
Brown soil	553	Bli	Sady com. Skierbieszów	50	41	10
(Cambisol)	568	Bli	Majdan Skierbieszowski com. Skierbieszów	58	30	11
	569	Bli	Majdan Skierbieszowski com. Skierbieszów	71	25	4
	570	Bli	Kol. Lipna Nowa com. Skierbieszów	62	27	11
	591	Bpłg	Rogalin com. Horodło	70	27	3
	594	Apłp. gs	Osówka com. Niemce	77	21	2
	604	Bps.pl	Wólka Kątna com. Markuszów	96	3	1
	605	Bps.pl	Wólka Kątna com. Markuszów	95	4	1
	607	Bps:pl	Bobowisko com. Markuszów	95	4	1
	611	Bps:pl	Kol. Olempin com. Markuszów	94	5	1
Chernozem (Haplic Phaeozem)	586	Cli	Hostynne com. Werbkowice	59	30	11
	601	Cli	Kol. Hostynne com. Werbkowice	60	35	6
	619	Cli	Sahryń com. Werbkowice	86	12	1
	621	Clz	Sahryń gm. Werbkowice	63	31	5
	622	Cli	Terebin com. Werbkowice	60	34	6
	623	Cli	Terebin com. Werbkowice	60	29	11
Alluvial soil (Dystric Fluvisol)	593	Fglp.płi	Kośmin com. Żyrzyn	86	12	2
Muck soil (Terric Histosol)	606	Mps.pl	Wólka Kątna com. Markuszów	97	2	1
Grey brown podzolic (Haplic Luvisol)	597	Apgm.gl	Dęba com. Kurów	95	4	1
Rendzina (Rendzic Leptisol)	597	Rcgc	Bezek com. Chełm	61	21	18
	560	Rcglp:gc	Chojno Nowe com. Siedliszcze	85	13	2
	562	Rbgp.gs	Chojno Nowe com. Siedliszcze	49	35	16
	565	Rcgl.gs	Okszów com. Chełm	78	17	5
	556	Rbgl.gs	Okszów com. Chełm	91	8	1
Black soil (Mollic Gleysol)	613	Dpłi.płg	Pożóg Nowy com. Końskowola	79	20	1

*Gliński *et al*., 1991.

10 mm, sunk in a glass pipe), a steel anode (stainless steel wire, diameter of 2 mm, 40 mm long) and a reference electrode which was a saturated calomel electrode (CSE). The potential of the cathode was controlled by the system of potentiostat (Malicki and Walczak, 1983).

The two-electrode set consisted of two identical platinum electrodes – platinum wire with purity of 3n, diameter of 0.5 mm, length of 10 mm, sunk in a glass pipe. One of the electrodes was the cathode and the other – the anode.

Taking into consideration the lack of repeatability of results obtained during successive replications under single-time installation of electrodes (Bieganowski, 2003), an analysis of the impact of the range of integration on the obtained values of the potential oxygen flux in soil was performed on the basis of the first current-voltage curve registered for a given installation of the electrodes.

In both cases (two- and three-electrode systems), the registration of the current-voltage curves was conducted under the rate of the cathode potential change equal to 4 mV s^{-1}.

RESULTS AND DISCUSSION

The parameters and their values being the basis for the comparison of the results obtained with the use of the three- and two-electrode systems are presented in Table 2.

Table 2. Comparison of results obtained with three- and two-electrode systems in voltammetric measurements of potential oxygen flux density in the soil

	Compared parameter		Three-electrode system	Two-electrode system
1	Subjective assessment of quasiplateau occurrence (population of 76 measurements)		42	28
2	Averaged values of right border of quasiplateau specified on the basis of subjective evaluation of quasiplateau occurrence		- 458 mV	- 1255 mV
	Standard deviation		111 mV	320 mV
	Absolute value of the ratio between standard deviation and averaged value of the right border (coefficient of variation)		0.242	0.255
3	Averaged values of left border of quasiplateau specified on the basis of subjective evaluation of quasiplateau occurrence		- 701 mV	- 1599 mV
	Standard deviation		69 mV	229 mV
	Absolute value of the ratio between standard deviation and averaged value of the left border (coefficient of variation)		0.098	0.143
4	Averaged 'length' of the quasiplateau*		243 mV	344 mV
	Standard deviation		89 mV	167 mV
	Absolute value of the ratio between standard deviation and averaged 'length' of the quasiplateau (coefficient of variation)		0.366	0.485
5	Absolute value of minimal difference OFD between two- and three-electrode systems*		1.14 mV (obtained for soil No. 597; soil water content - 34.9%)	
	Absolute value of maximum difference OFD between two- and three-electrode systems*		66.17 mV (obtained for soil No. 623; soil water content - 41.2%)	
	Absolute value of average difference OFD between two- and three-electrode systems*		24.17 mV	
	Standard deviation		13.97 mV	
6	Number of measurements with low value of extremum (absolute value of difference of maximum and minimal value of current in quasiplateau area is less than 1.2 μA)		8	4
	Number of measurements with high value of extremum (absolute value of difference of maximum and minimal value of current in quasiplateau area is greater than 1.2 μA)		18	1
7	Double quasiplateau	Weakly developed	4	4
		Well developed	4	3
8	Evaluation of monotonicity when soil water content of the soils sample decreases. Population – 25 soils*	Number of cases when with the decrease of soil water content OFD increased	8	9
		Number of cases when with the decrease of soil water content OFD decreased too	3	4
		Number of cases when with the decrease of soil water content OFD showed the extremum	14	12

* The ranges of integration were established in consequence of item 2 and 3: three-electrode system -460 ÷ -700 mV, two-electrode system -1260 ÷ -1600 mV.

Analysis of the quasiplateau range

Both the discussed methods of determination of the potential oxygen flux in soil (amperometric and voltammetric) are based on the assumption that under a specific potential value the current flowing in the system comes only from the reduction of the molecular oxygen dissolved in the soil solution and that the speed of reduction is limited by the possibility of the diffusive completion of the deficiency of oxygen occurring on the electrode surface. The consequence of this is the fact of plateau occurrence at the dependence between the current (*I*) ant the potential (*U*). Nevertheless, in practice, the results obtained during the measurements realized not only on soils but also in solutions do not show the plateau area *ie* a situation when $\delta I/\delta U = 0$. In a majority of cases, for saturated soils (the assumption of the limit in the rate of electrode reaction through oxygen diffusion is applicable only for saturated or nearing saturation soils) the analysis of the current-voltage curves leads to the conclusion that although the phenomenon of the restriction to the current growing takes place (that is manifested by the area of the so called quasiplateau), the current-voltage curve shows in this area some increase of the current with the increase of potential *ie* $dI/\delta U \neq 0$.

The lack of the occurrence of the evident plateau can be caused by many, often overlapping, factors. For instance, the assumption that the only substance being reduced on the cathode is oxygen is not proper (Bieganowski, 2002). After all, in unsaturated soils the speed of diffusion can be too high and the limitation of oxygen availability can be incomplete.

In measurement practice, the registration of the current-voltage curve and checking the criterion of the quasiplateau occurrence can enable the discarding of measurements which are not interpretable in the context of fulfilling the assumptions of the method.

In Table 2, item 1 groups together the numbers of cases where, during the subjective analysis of the current-voltage curves, it was recognized that the quasiplateau exists. The procedure of evaluation consisted in graphical presentation of all the current-voltage curves in the same scale, followed by an arbitrary decision if the quasiplateau occurred or not. In spite of the high level of uncertainty of the interpretative procedure carried out this way, it can be stated that in the case of the use of the symmetrical two-electrode system, the quasiplateau occurs more seldom than when the three-electrode system is applied (37% for the two-electrode and 55% for three-electrode systems, respectively).

In a majority of cases for a given soil material and a given water content value, the occurrence of the quasiplateau under the measurement with the three-electrode system did not correspond to the occurrence of the quasiplateau under the measurement with the two-electrode system. The number of cases where the quasiplateau was observed simultaneously for both systems of electrodes was 18 (from the total of 76). For these cases, a comparison of the OFD values, calculated on the basis of the observed quasiplateau, was made - Fig. 1A.

When analyzing the plot from Fig. 1A, the low correlation should be noted between the results obtained with the use of the three- and two-electrode systems (determination coefficient 0.0917).

The important parameters describing the quasiplateau are the right and left borders of this area (a graphical definition of the right and left borders for the ideal curve is presented in Fig. 2). In Table 2 (items 2 and 3) the averaged values of these parameters are presented, as well as the respective standard deviations. It is worth to note that:

– comparing the right and left borders of the quasiplateau: the ratio of the standard deviation to the mean value for both sets of electrodes is smaller in the case of the left border:

– comparing both sets of electrodes: both for right and left borders of the quasiplateau, the ratio of the standard deviation to the mean value is higher in the case of the two-electrode system.

Because the measurements described in this paper were carried out in artificially prepared saturated and close to saturation samples, it is possible not to take into account, when interpreting the results, the situation when insufficient water content causes the necessity of discarding results. Due to this, for comparison, without analyzing the reasons of not forming the quasiplateau of the dependence *I*(*U*), for all the samples and for both sets of electrodes, the values of oxygen flux density, OFD, was calculated for the range of potentials calculated in Table 2, items 2 and 3 (*ie* -458 ÷ -701 mV for the three-electrode system and -255 ÷ -1599 mV for the two-electrode system, respectively). The comparison of these two values is presented in Fig. 1B.

The determination coefficient is also very small (0.112), although it is a bit higher than in Fig. 1A. The analysis of the two coefficients can lead to the conclusion that uncertainty of the measurement (which is the resultant of numerous, sometimes difficult to be determined factors, *eg* the geometry of the system, the water content on the micro-scale near the electrode surface, etc.) and the resulting dispersion of the results are so high that it is difficult to compare directly the results obtained with the use of the two- and three-electrode systems. The proof that this thesis is true can also be the relatively high dispersion between the minimum and maximum differences, calculated for the three- and two-electrode systems (Table 2, item 5).

This conclusion is a confirmation of the thesis, formulated in earlier papers, that voltammetric determination of the potential oxygen flux in soil should be treated more as an indicator than the measure of oxygenation (Bieganowski, 2002).

Independently from the above conclusion, it is possible and advisable to indirectly compare the measurements

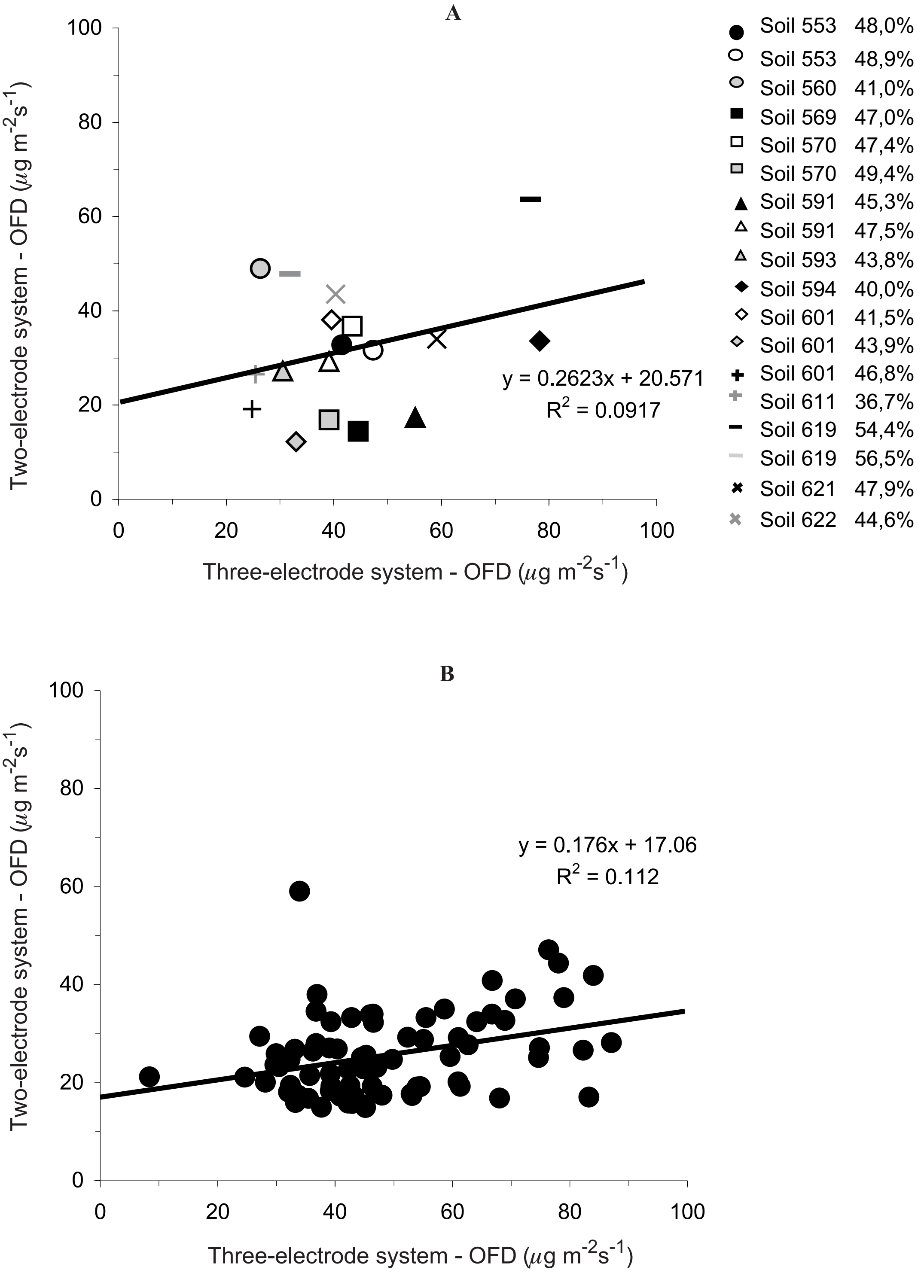


Fig. 1. Comparison of OFD obtained with the three- and two-electrode systems in voltammetric measurements of potential oxygen flux in the soil. A) Selected values of moisture content of investigated soils. The criterion of selection was the simultaneous occurrence of quasiplateau in the current-voltage curves in measurements made with the three- and two-electrode systems. B) All measurements (all water content values in all investigated soils). The range of integration for OFD calculation is shown in Table 2, items 2 and 3.

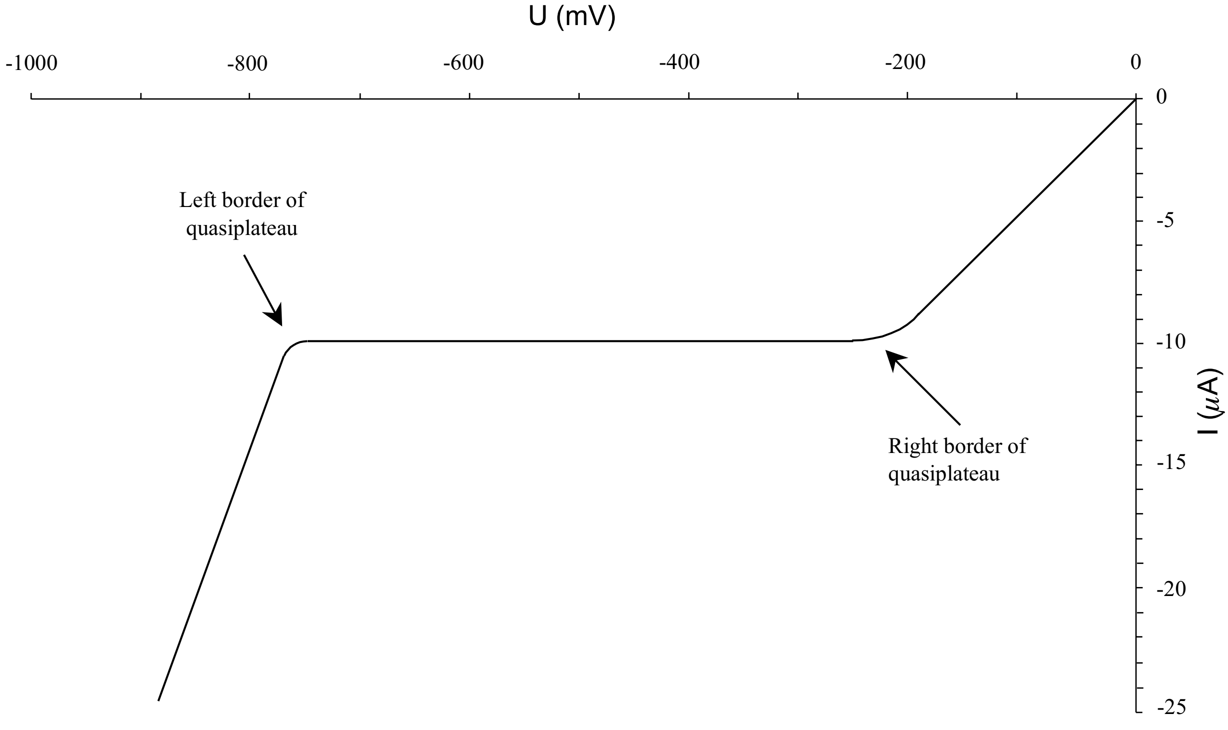


Fig. 2. Idealized current-voltage curve and definition of right and left borders of quasiplateau.

performed with the two- and three-electrode sets to evaluate which of these methods gives more precise results.

The analysis of the ratio of standard deviation to the mean value of the 'length' of the quasiplateau coefficient of variation (Table 2, item 4) leads to the conclusion that better repeatability takes place for the three-electrode system, for which the value of this ratio is 0.366, while for the two-electrode system the analogous value is 0.485.

Analysis of the shape of the quasiplateau

One of the problems described in the literature, dedicated to the electrochemical measurements of the soil properties, is the so-called 'poisoning' of the electrode surface. This poisoning can be understood in different ways, therefore – for the needs of this paper – it is defined as the phenomenon of precipitation, on the surface of the electrode, of deposits which are the effect of migration of chemical substances in the electrical field and of the products of electrochemical reactions taking place earlier than the considered measurement. The poisoning of the electrode can lead to the chemical or physical blocking of a part of the surface and, due to this, to uncontrolled changes of the reaction course.

The phenomenon of poisoning of electrode surface is common and occurs during any electrochemical measurements in the soil (Mc Intyre, 1970). However, as opposed to the amperometric measurements, the results obtained by the voltammetric method enable the evaluation of the quality of this phenomenon and of the possible impact on the results obtained. The feature that makes it possible to conduct this evaluation is the shape of the curve in the range of potentials in which the quasiplateau should occur. The appearance of extremes in this area can confirm the overlapping of some other currents, sometimes difficult to be determined, on the current of the molecular oxygen reduction. An exemplification of this can be the currents of reduction/oxidation of deposits accumulated on the electrode during previous measurements. An exemplary current-voltage curve, on which an extreme appeared, is presented in Fig. 3.

From among different methods of electrode preparation for measurements, one of the most efficient seems to be mechanical cleaning of the surface. This can be realized by rubbing it with absorbent paper or with fine-grained sandpaper (Mc Intyre, 1970; Rankin and Sumner, 1978; Carnell and Anderson, 1986), or just by putting the electrode into the soil and taking it out of it (Armstrong, 1967; Carnell and Anderson, 1986). The latter of the mentioned methods *ie* cleaning the platinum by taking it out of the soil and installing again, was adopted in this study.

As can be seen from Table 2, item 6, extremes of the dependence *I*(*U*) occurred more frequently for the three-electrode system (26 of 76 cases) than for the two-electrode system (5 of 76 cases). Moreover, in the three-electrode system, extremes with high values prevailed (*ie* the absolute value from the maximum difference of the current value and

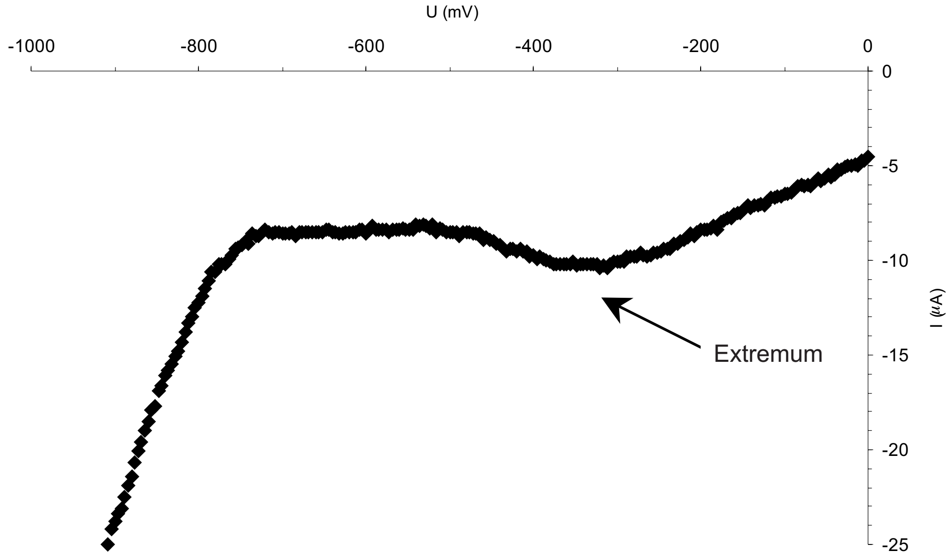


Fig. 3. Example of a current-voltage curve (soil No. 568; soil water content 46.7%) obtained with the three-electrode system. There is a relative extremum in the range of potentials of quasiplateau.

the minimum current value in the area of the quasiplateau was higher than 1.2 mA).

When analyzing the shape of the quasiplateau area, the phenomenon of creation of two waves of current in different ranges of the potential values should also be noted. It took place in the measurements with the three-electrode as well as with the two-electrode systems (Table 2, item 7). The exemplary curves illustrating this phenomenon for the cases of evidently formed two quasiplateau areas (according to the subjective evaluation) are presented in Fig. 4.

Analysis of the relation between OFD and water content

Important information which enables indirect evaluation of the uncertainty of the results is the analysis of the relation between OFD and the water content. As has been mentioned earlier, the assumption which was the foundation of both the electrochemical methods (amperometric and voltammetric) for the estimation of the potential density of the oxygen flux in the soil is the thesis that the value of the current in the system is limited by the possibility, based on diffusion, of the replenishment of oxygen that reacts on the cathode. If it is true, theoretically, every diminishing of the water content as compared to the saturation state should result in a monotonic increase of OFD values because the diffusion coefficient in the gaseous phase is about 10000 times higher than in the liquid phase (Gliński and Stępniewski, 1985).

For practically all the soils, *ie* for 25 cases, saturated soil samples were studied as well as samples of the soils with water content levels nearing saturation (except for the soils with numbers 604 and 566, for which the measurements were performed only for saturated samples). For the sets of results corresponding to the successive soil water content values of a given soil, the relation between OFD and the water content was analyzed. The results are presented in Table 2, item 8.

For both sets of electrodes, most frequently (in about 50% of all cases) a decrease in the soil water content resulted in the OFD values showing extremes. When comparing this tendency with a number of cases where the monotonicity was inverse to the expected *ie* when with lowering of the soil water content the value of OFD decreased (several per cent of all cases), one should conclude that the uncertainty of the measurement is too big to permit the observation of such little differences of OFD in relation to the water content which varied under the conditions of the experiment.

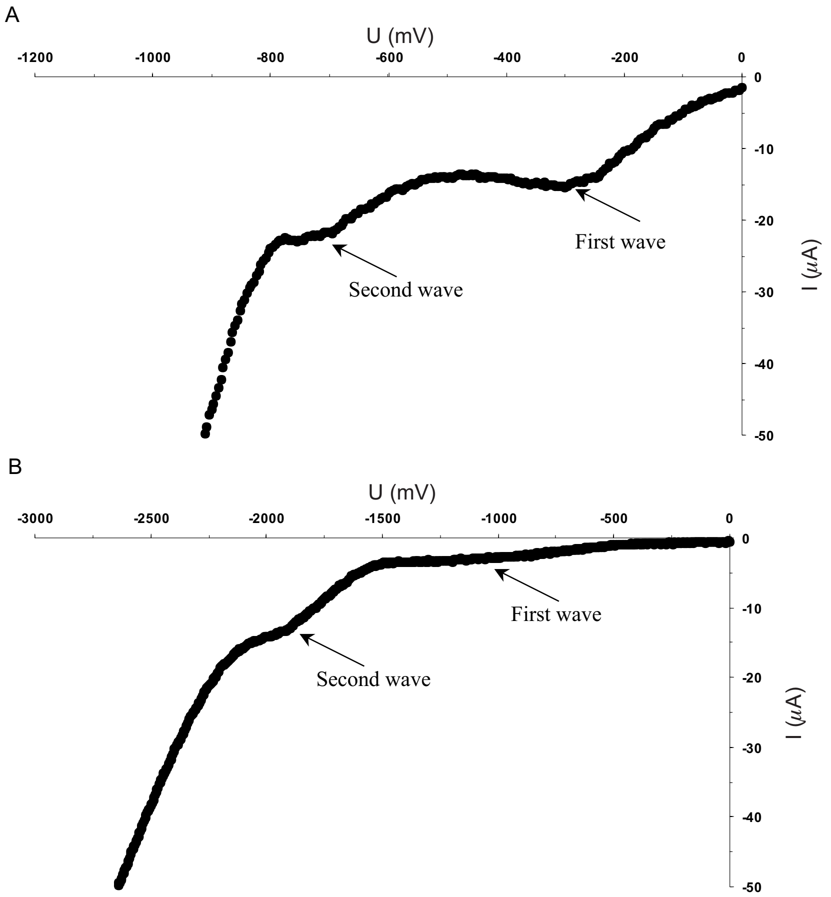
From the practical point of view, in this aspect there were no differences between the three- and two-electrode systems.

CONCLUSIONS

1. A possibility exists to apply the symmetrical two-electrode system for the determination of the potential oxygen flux in the soil

2. Direct comparison of the values obtained with the use of two- and three-electrode systems is difficult because of the high uncertainty of the results of the voltammetric method applied for the determination of the potential oxygen flux in the soil.

3. The uncertainty of the results of the measurement of the potential oxygen flux in soil realized with the voltammetric method is smaller when the classical system of



Rys. 4. Examples of current-voltage curves where two waves can be observed. These waves can be evidence that the reduction of molecular oxygen in not the unique electrochemical reaction in the system. A) three-electrode system in soil 565; soil water content 53.7%; B) three-electrode system in soil 606; soil water content 40.2%.

three electrodes is used than in case of using the symmetrical two-electrode system.

4. Regardless of the electrode systems applied (symmetrical two- or three-electrode with the system of potentiostat) the results obtained should be treated as an indicator rather than as a measures of the availability of oxygen for plant roots.

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