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The content of available macro- and microelements against the background of enzymatic activity in soils affected by the soda industry

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Abstract

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Salinity is one of the main causes of degradation in the soil environment. The long-term salinity affects the properties physicochemical and activity of the biological parameters in soil considerably. The aim in the study was to assess the content of selected macro- and microelements and enzymatic properties in soils in areas adjacent to the soda plant. The research was conducted in the area adjacent to the CIECH Soda Polska S.A. in Inowrocław. This study presents an assessment of the content of selected bioavailable macroelements (P, K, Mg) and microelements (Zn, Cu, Mn, Fe) against the background of the enzymatic activity (dehydrogenases, catalase, peroxidase, and alkaline and acid phosphatase) of soil. The soil was sampled from surface mineral horizon (0–30 cm deep). Seven sites were selected for soil sampling (S1–S6 on the grounds of a soda plant, and area C – the control). The effect of long-term salinity on the studied soil properties was found. The content of available macro- and micronutrients varied significantly depending on the soil sampling site. The presented results did not show unidirectional changes in the content of available P, K, Mg, Zn, Cu, Mn and Fe, nor in the activity of dehydrogenases, catalase, peroxidase, or alkaline and acid phosphatases in the technogenic soil. In the analysed soil samples, contents of the bioavailable forms of all the tested microelements were found to be low. The activity of the tested enzymes was found to be lowest in soil from S6 (in the vicinity of waste dumping sites, a sewage treatment plant and the soda plant).

1. Introduction

Soils are salinated through complex processes that can be caused by the natural accumulation of salt in the soil or by inappropriate human activity (Zhou et al., 2012). In the temperate climate zone, salt-affected soils are formed under the impact of salt springs during natural soil-forming processes (Piernik and Hulisz, 2011), and also due to anthropogenic saline habitats. The problem is anthropogenic salinity, which mainly relates to large industrial centres established near to natural mineral deposits. It is also associated with mining waste dumps and areas around soda industry plants (Cieśla et al., 1981; Hulisz and Piernik, 2013), the use of mineral salts for de-icing roads (Daliakopoulos et al., 2016) and the intensive use of mineral fertilisers (Widłak, 2016). Salination can affect high-class arable soils of high utility value. This occurs because an excess of sodium ions negatively affects soil structure, aggregate stability and soil hydraulic properties. High sodium concentrations degrade soil through the dispersion of clay and slaking. This can affect water and air movement and root penetration (Crescimanno et al., 1995). Salt toxicity is one of the major edaphic factors

limiting plant growth (Khatar et al., 2017). Natural and induced soil salinity reduce plant growth and the activity of microorganisms by lowering water availability (as a result of the high osmotic potential of the soil solution) and by raising ion toxicity (Wang et al., 2013). High concentrations of salts may also change the chemical behaviour of metals accumulated in soils, increasing or decreasing their availability. The method of soil use also affects the content of macro- and microelements by changing the physics, chemistry and biology of the soil. Long-term salinity can considerably degrade the parameters of soil biological activity. That is why, in recent years, enzyme activity testing has been used to assess soil fertility. For several years there has been a progressive devastation of arable soils in the immediate vicinity of the waste tanks of the CIECH Soda Polska S.A. plant in Mątwy (Hulisz et al., 2018; Lemanowicz, 2019; Bartkowiak et al., 2020). The infiltration of collected waste water into the ground is increasing the salinity and alkalinity of adjacent soils.

This study aims to assess the content of selected macro- and microelements and enzymatic properties in soils in areas adjacent to the soda plant.

2. Material and methods

2.1. Location of soil sampling

The research was conducted in the area adjacent to CIECH Soda Polska S.A. (a soda plant founded in 1882) in Inowrocław. The soda factory produces, for example, light and heavy soda ash, sodium bicarbonate, calcium chloride, and precipitated calcium carbonate. The soil was sampled from the mineral horizon (0–30 cm deep) in summer (July) of 2017 at seven sites within the soda plant and from a control point (Fig. 1). The sites differed in their method of use: site S1 – places around the clarifying-cooling “pond”, where carbonates are precipitated as waste (a non-reclaimed location); sites S2 and S3 – places where technical and agrotechnical reclamation have been completed; site S4 – a dried pond for ash waters; site S5 – an agricultural field where spring barley was grown 500 m from the soda plant; and site S6 – places in the vicinity of waste dumping sites, a sewage treatment plant and the soda plant with numerous communities of halophytes.

2.2. Soil analysis

In the air-dried soil samples with a disturbed structure and sieved through 2-mm mesh, the following physicochemical properties were determined: clay fraction by laser diffraction using a Mastersizer MS 2000 analyser; pH in 0.01 M CaCl₂ measured potentiometrically (PN-ISO 10390, 1997); total organic carbon (TOC) using a TOC FORMACTS™ Primacs analyser provided by Skalar; electrical conductivity (ECe) in soil paste; and

total exchangeable bases (TEB) by the Kappen method (Soil Survey Investigation, 1996). Additionally, the contents of available forms of Zn, Cu, Mn and Fe extracted with DTPA solution were assayed according to Lindsay and Norvell (1978). The contents of available forms of phosphorus (P) (PN-R-04023, 1996), potassium (K) were also defined, by the Egner–Riehm method (DL) (PN-R-04022, 1996), as was the content of magnesium available to plants (Mg) following the Schachtschabel method (PN-R-04020, 1994). The content of forms available to plants was determined by atomic absorption spectroscopy and atomic emission spectroscopy using a PU 9100X (Philips) spectrometer. To verify the adequacy of the results and the accuracy of the measurement, the certified material Loam Soil No. ERM–CC141 was analysed, and so-called null tests were performed according to the same analytical procedure as the investigated soil material.

The activity of selected oxidoreductase and hydrolytic enzymes was assayed: dehydrogenases (DEH) in soil by the Thalmann method (1968), catalase (CAT) by the Johnson and Temple method (1964), peroxidases (PER) by the Bartha and Bordeleau method (1969), and alkaline (AlP) and acid (AcP) phosphatase by the Tabatabai and Bremner method (1969).

2.3. Statistical analysis

The results were analysed statistically using STATISTICA 13.0 software. The data were analysed by one-way ANOVA variance analysis, followed by Tukey's *post-hoc* test, which isolated homogeneous groups. Significant differences ($p < 0.05$) were studied further. All analytical measurements were performed in three replicates. Arithmetic mean values are shown in tables,



Fig. 1. Location of the study area

including standard deviation (\pm SD). The analysis results were analysed for simple correlation ($p < 0.05$), which determined the degree of dependence between respective features. The precision and accuracy of the analyses was determined using certified reference material (Loam Soil No. ERM-CC141).

3. Results and discussion

According to the Polish Soil Classification (2019), the tested soils are Anthropogenic soils degraded as a result of technologically induced salination, and can be classified as Industriosols. Selected physical and chemical soil properties are presented in Table 1. The soil pH values ranged from 6.78 to 7.65 (which is characterised as neutral and alkaline) and clay content ranged from 4.8 to 9.0%. Total exchangeable bases (TEB) were at low levels. Total exchangeable bases in soil varied significantly by soil sampling site. The highest TEB values were highest in the soil of S6 ($51.63 \text{ mmol}(+)\cdot\text{kg}^{-1}$), but no significant differences in TEB were found in soil sampled from S1–S4 (Table 1).

Total organic carbon content varied depending on the studied habitat, ranging from 2.36 to $77.02 \text{ g}\cdot\text{kg}^{-1}$. The highest content of TOC was determined in the soil from the S1 site and the lowest in the S6 and control soil. Salt-affected soils usually have low organic matter contents due to insufficient plant growth leading to low inputs of organic materials. According to Wong et al. (2010), in saline soils there are a number of opposing processes, some of which often inhibit the humification process. Salinity measured by electrical conductivity varied from 10.1 to $60.5 \text{ dS}\cdot\text{m}^{-1}$. Similar results were obtained by Hulisz et al. (2018). The highest conductivity was detected for sample S6, having been under the impact of three sources of salinity: the sedimentation tanks, the sewage treatment plant and the municipal waste dump. Most of the soils were, according to Jackson's (1958) classification, very strongly saline ($\text{ECe} > 16 \text{ dS}\cdot\text{m}^{-1}$).

The content of bioavailable elements in the studied soil profiles ranged from 5.58 to $128.00 \text{ mg}\cdot\text{kg}^{-1}$ for phosphor, 40.9 to $310.0 \text{ mg}\cdot\text{kg}^{-1}$ for potassium, and 121.0 to $590.5 \text{ mg}\cdot\text{kg}^{-1}$ for magnesium (Table 2). The content of available macronutrients var-

ied significantly depending on the soil sampling site. The highest contents of P and K were recorded for the control (C) and the highest Mg was in S3 (soil where technical and agrotechnical reclamation had been conducted). ANOVA showed that contents were by far the lowest at S6 for P and Mg, and at S3 for K. No significant differences in P content were found between soils sampled from S1, S2, S4 and S5. The content of P was significantly negatively correlated with ECe ($r = 0.834$, $p = 0.019$) and pH ($r = 0.860$, $p = 0.013$). The variation in available phosphorus in the soil was 69.9% determined by ECe and 73.9% by pH (Table 5). The toxic effect of soil salinity may increase in the presence of chlorides, dramatically reducing absorption of nutrients (mainly of N, P and K). Available phosphorus was positively correlated with acid phosphatase activity ($r = 0.905$, $p = 0.005$). Variation in phosphorus was found (using the value of the coefficient of determination [R^2]) to account for as much as 82% of the variation in the activity of AcP . The linear regression equations shows that with a $1 \text{ g}\cdot\text{kg}^{-1}$ increase in AP the activity of AlP increase by $29 \text{ mM pNP}\cdot\text{kg}^{-1}\cdot\text{h}^{-1}$ (Table 5). According to Choudhary and Yaduvanshi (2016) saline soils often contain medium to high amounts of available K. However, during reclamation, K may be lost due to leaching, leading to its low availability. But Xie et al., (2019) stated that long-term reclamation could radically decrease soil salination and promote soil nutrient accumulation (SOM, TN, TP, AN, AP, CEC).

In the analysed soils the content of available Zn, Cu, Mn and Fe forms fell by the following ranges of values: Zn – 0.89 – $13.94 \text{ mg}\cdot\text{kg}^{-1}$; Cu – 0.75 – $5.69 \text{ mg}\cdot\text{kg}^{-1}$; Mn – 3.38 – $5.24 \text{ mg}\cdot\text{kg}^{-1}$; and Fe – 2.88 – $28.06 \text{ mg}\cdot\text{kg}^{-1}$ (Table 3).

The highest content of zinc available to plants was noted at sites C, S2 and S3, and at those points it was higher than the content specified as a deficit for plants; namely, $0.8 \text{ mg}\cdot\text{kg}^{-1}$ (Lindsay and Norvell, 1978). Alkali soils usually contain less than 0.6 mg DTPA-extractable Zn per kg of soil and show a marked deficiency of this element (Choudhary and Yaduvanshi, 2016). Meanwhile, the content of copper extracted with DTPA, which is considered to be the content available to plants, exceeded the deficit contents below which plants' growth and yield worsen; namely, 0.12 – $0.25 \text{ mg}\cdot\text{kg}^{-1}$ (Sims and Johnson, 1991). However,

Table 1

The content of clay [%], pH in CaCl_2 , total organic carbon (TOC) [$\text{g}\cdot\text{kg}^{-1}$], ECe [$\text{dS}\cdot\text{m}^{-1}$] and TEB [$\text{mmol}(+)\cdot\text{kg}^{-1}$]

Sites	Clay	pH	TOC	ECe	TEB
C	9.00	6.78	$2.36^c \pm 0.20$	$10.1^c \pm 0.57$	$30.42^d \pm 1.77$
S1	7.94	7.47	$77.02^a \pm 26.98$	$36.5^b \pm 8.49$	$48.81^b \pm 0.51$
S2	7.52	7.43	$9.06^c \pm 1.99$	$17.6^c \pm 2.97$	$49.60^b \pm 0.07$
S3	5.60	7.44	$8.38^c \pm 0.36$	$18.1^c \pm 3.96$	$49.84^b \pm 0.10$
S4	6.61	7.41	$33.24^b \pm 20.39$	$20.6^c \pm 0.07$	$49.78^b \pm 0.09$
S5	4.80	7.22	$28.7^b \pm 0.77$	$34.6^b \pm 1.63$	$39.05^c \pm 0.16$
S6	6.61	7.65	$2.36^c \pm 2.08$	$60.5^a \pm 2.12$	$51.63^a \pm 0.51$

Values followed by the same letter are not significantly different at $p < 0.05$. Different small letters indicate comparison between sites. (\pm)Standard Deviation

Table 2

The content of available phosphorus (P) [$\text{mg}\cdot\text{kg}^{-1}$], potassium (K) [$\text{mg}\cdot\text{kg}^{-1}$] and magnesium (Mg) [$\text{mg}\cdot\text{kg}^{-1}$]

Sites	P	K	Mg
C	$128.0^a \pm 24.04$	$310.0^a \pm 1.41$	$174.0^d \pm 15.56$
S1	$55.69^c \pm 5.40$	$278.5^b \pm 3.54$	$250.0^c \pm 12.73$
S2	$56.32^c \pm 9.65$	$177.5^d \pm 9.19$	$391.0^b \pm 8.49$
S3	$88.87^b \pm 29.8$	$40.9^e \pm 1.94$	$590.5^a \pm 9.19$
S4	$51.50^c \pm 19.4$	$111.5^c \pm 9.19$	$128.5^e \pm 9.19$
S5	$61.10^c \pm 8.61$	$240.5^c \pm 6.36$	$150.0^{de} \pm 1.41$
S6	$5.58^d \pm 2.84$	$150.5^d \pm 10.61$	$121.0^e \pm 8.49$

Table 3The content of available zinc (Zn), copper (Cu), manganese (Mn) and iron (Fe) [mg·kg⁻¹]

Sites	Zn	Cu	Mn	Fe
C	11.38 ^a ±1.41	1.37 ^b ±0.10	5.24 ^a ±0.32	2.88 ^c ±0.34
S1	0.89 ^b ±0.00	0.75 ^b ±0.06	3.38 ^c ±0.03	3.61 ^c ±1.71
S2	13.94 ^a ±3.51	5.36 ^a ±2.82	4.35 ^{bc} ±0.33	28.06 ^{ab} ±2.01
S3	10.32 ^{ab} ±2.93	5.69 ^a ±0.39	4.70 ^b ±0.11	29.12 ^a ±7.13
S4	1.59 ^b ±0.9	1.66 ^b ±0.04	4.87 ^b ±0.06	18.04 ^{ab} ±8.26
S5	4.46 ^b ±0.62	1.69 ^b ±0.16	4.72 ^b ±0.20	16.96 ^b ±2.04
S6	4.40 ^b ±1.77	0.81 ^b ±0.21	5.04 ^{ab} ±0.17	22.26 ^{ab} ±1.70

we should note that various plant species differ in their nutritional requirements. Therefore, the content of absorbable forms of copper in the tested soil samples was above deficiency levels. In turn, the levels of bioavailable forms of manganese found can be considered insufficient for the nutritional requirements of plants. Critical levels for DTPA-extractable Mn range from 2.5 to 5.0 mg·kg⁻¹ (Moraghan and Mascagni, 1991). Finally, low contents of bioavailable iron were also found in the analysed soil samples. There is a significant negative correlation between iron content and potassium content ($r = 0.839$, $p = 0.018$) (Table 5).

The phyto-availability of elements is affected by: reaction; granulometric and mineral soil composition; water properties; and organic matter content. A factor limiting the solubility of most metals, and therefore their availability to plants is, alongside pH, the amount of organic matter. In the case of copper, manganese and zinc, increased organic matter content causes them to bind strongly to the organic substance and lowers their bioavailability to plants. In contrast, in humic soils, iron forms chelate bonds that are water-soluble. Unlike the other elements mentioned, iron is easily taken up by plants (Łukasiewicz, 2012). The available forms of zinc, copper and iron were not found to correlate statistically significantly with content of total organic carbon or soil pH. The only significant correlation found was a negative one between the content of available manganese and TOC ($r = 0.839$, $p = .018$) (Table 5).

Table 4The activity of dehydrogenases (DEH) [mg TPF·kg⁻¹·24h⁻¹], catalase (CAT) [mg H₂O₂·kg⁻¹·h⁻¹], peroxidase (PER) [mM PPG·kg⁻¹·h⁻¹], alkaline (ALP) and acid (AcP) phosphatase [mMpNP·kg⁻¹·h⁻¹]

Sites	DEH	CAT	PER	ALP	AcP
C	1.724 ^c ±0.10	0.288 ^a ±0.004	0.736 ^d ±0.010	1.599 ^b ±0.011	3.842 ^a ±0.953
S1	2.710 ^b ±0.24	0.237 ^{bc} ±0.001	0.884 ^c ±0.011	1.563 ^b ±0.088	1.898 ^{bc} ±0.756
S2	2.634 ^{bc} ±0.07	0.232 ^{bc} ±0.026	1.142 ^b ±0.047	1.657 ^b ±0.225	1.729 ^c ±0.806
S3	1.845 ^c ±0.13	0.204 ^{bc} ±0.006	1.396 ^a ±0.013	1.729 ^b ±0.161	2.434 ^b ±0.956
S4	1.701 ^c ±0.19	0.194 ^c ±0.012	0.595 ^e ±0.009	0.412 ^c ±0.210	0.318 ^d ±0.050
S5	3.743 ^a ±0.30	0.250 ^{ab} ±0.008	1.231 ^b ±0.034	2.349 ^a ±0.206	1.596 ^c ±0.243
S6	0.737 ^d ±0.02	0.145 ^d ±0.006	0.428 ^f ±0.022	0.269 ^c ±0.044	0.448 ^d ±0.055

ANOVA indicated that the activities of the studied enzymes were dependent on the soil sampling site ($p < 0.05$) (Table 4). The lowest activities of DEH (0.737 mg TPF·kg⁻¹·24h⁻¹), CAT (0.145 mg H₂O₂·kg⁻¹·h⁻¹) PER (0.428 mM PPG·kg⁻¹·h⁻¹), ALP (0.269 mM pNP·kg⁻¹·h⁻¹) and AcP (0.448 mM pNP·kg⁻¹·h⁻¹) were reported in soil from site S6 (the location near city waste dumps, the sewage treatment plant and the soda plant). Similar results were obtained by Lemanowicz (2019). According to Telesiński (2012), increased salt content in soil decreases enzymatic activity. Osmotic potential is changed by the toxicity of specific ions and the drying of enzymatic proteins. However, this study showed no correlation between enzyme activity and salinity (ECe). The highest activity of dehydrogenases (3.743 mg TPF·kg⁻¹·24 h⁻¹) and alkaline phosphatase (2.349 mM pNP·kg⁻¹·h⁻¹) was found in soil from S5. The activities of catalase (0.288 mg H₂O₂·kg⁻¹·h⁻¹) and acid phosphatase (3.842 mM pNP·kg⁻¹·h⁻¹) were much the highest in the control soil (C). Peroxidase activity was highest in S3. According to Pan et al. (2013), salinity affects the enzymatic activity of soil in different ways (depending on enzyme type and degree of salinity).

The research showed significant positive correlations between dehydrogenases and alkaline phosphatase ($r = 0.826$, $p = 0.022$), between catalase and acid phosphatase ($r = 0.805$, $p = 0.028$) and between peroxidases and alkaline phosphatase ($r = 0.839$, $p = 0.018$) (Table 5). This indicates that any enzyme activity can considerably reflect other enzyme activities in soil

Table 5
Relationship between selected soil properties

Variables		Equation	<i>r</i>	<i>R</i> ²	<i>p</i>
Dependent	Independent				
CAT	P	$y = 0.1579 + 0.001x$	0.814	0.663	0.025
AcP	P	$y = -0.1006 + 0.029x$	0.905	0.820	0.005
P	pH	$y = 914.71 - 115.87x$	-0.860	0.739	0.013
P	ECe	$y = 115.58 - 1.8295x$	-0.834	0.696	0.019
K	Fe	$y = 317.64 - 7.5593x$	-0.839	0.704	0.018
Mg	Cu	$y = 75.89 + 73.498x$	0.892	0.796	0.006
Mn	TOC	$y = 5.0555 - 0.0192x$	-0.839	0.704	0.018
DEH	ALP	$y = 0.709 + 1.0577x$	0.826	0.682	0.022
CAT	AcP	$y = 0.1675 + 0.0606x$	0.805	0.649	0.028
CAT	pH	$y = 1.251 - 0.1403x$	-0.855	0.731	0.014
CAT	TEB	$y = 0.4367 - 0.0047x$	-0.815	0.664	0.025
PER	ALP	$y = 0.3332 + 0.3966x$	0.839	0.704	0.018
AcP	pH	$y = 26.620 - 3.3868x$	-0.784	0.616	0.036

(Lemanowicz, 2019). The activity of the enzymes was not found to significantly correlate with TOC. This may be due to the proportion of humic substances in total soil organic content being low. This reduces the availability of easily absorbable carbon, which affects the growth of the microorganisms that produce soil enzymes.

4. Conclusions

Technogenic salinity causes soils to form that have no natural equivalents in nature. The long-term influence of salinity significantly conditioned the properties of the studied soil. The presented results did not show unidirectional changes in the content of available P, K, Mg, Zn, Cu, Mn and Fe, nor in the activity of dehydrogenases, catalase, peroxidase, or alkaline and acid phosphatases in the technogenic soil. There was no reduction in activity of the tested enzymes in the soil at the soda plant (except for the S6 soil). There is also no danger of an increase in the content of available forms of the tested heavy metals in the 0–30 cm soil horizons.

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Zawartość przyswajalnych makro- i mikroelementów na tle aktywności enzymatycznej gleb zmienionych w wyniku oddziaływania przemysłu sodowego

Słowa kluczowe

Aktywność enzymatyczna
Przyswajalne makroelementy
i mikroelementy
Gleby technogeniczne
Gleby zasolone

Streszczenie

Zasolenie jest jedną z głównych przyczyn degradacji środowiska glebowego. Długotrwałe zasolenie gleb wpływa na ich właściwości fizykochemiczne oraz parametry biologiczne w tym aktywność enzymatyczną. Celem pracy była ocena zawartości wybranych makro- (P, K, Mg) i mikroelementów (Zn, Cu, Mn, Fe) na tle aktywności enzymatycznej (dehydrogenazy, katalazy, peroksydazy oraz fosfatazy alkalicznej i kwaśnej) w glebach na obszarze w sąsiedztwie zakładów sodowych CIECH Soda Polska S.A. w Inowrocławiu. Próbkę glebowe pobrano z powierzchniowych poziomów mineralnych gleb na głębokości 0-30 cm. Do badań wyznaczono siedem miejsc różniących się sposobem użytkowania (S1 – S6 na terenie zakładów sodowych i C – kontrola). Stwierdzono wpływ długoterminowego zasolenia na badane właściwości gleby. Zawartość dostępnych przyswajalnych form makro- i mikroskładników różniła się znacznie w zależności od miejsca pobierania próbek glebowych. Przeprowadzone badania nie wykazały jednakowych trendów zmian zawartości przyswajalnych P, K, Mg, Zn, Cu, Mn i Fe oraz aktywności dehydrogenazy, katalazy, peroksydazy oraz fosfatazy alkalicznej i kwaśnej w glebie technogenicznej. W analizowanych próbkach glebowych stwierdzono niskie zawartości form przyswajalnych wszystkich badanych mikroelementów. Wzrost zawartości soli w glebie może powodować obniżenie aktywności enzymatycznej, jednak na terenie zakładów sodowych nie wykazano takich zależności. Najniższą aktywność badanych enzymów uzyskano w glebie pobranej z miejsca S6, które zlokalizowane było w pobliżu składowisk odpadów, oczyszczalni ścieków i zakładów sodowych.