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Drawdown of biophilic elements under winter barley, and maintaining fertility of black soils of Western Ukraine

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ABSTRACT
Climate stabilisation and soil fertility strategies that capture carbon as soil organic matter lag because of ambiguity in stoichiometry, ionomics, and correlations between key chemical components of soil productivity. We analyse the mutual influence of C, N, P, Ca, S, Mg, and K content, N content, microelements, and the yield of winter barley in Luvic Greyzemic Phaeozems of the Ukrainian Western Forest-Steppe. Depletion of biophilic element stocks occurred only with a significant increase in the yield of winter barley which ranged from 6.6 to 7.4 t/ha compared to unfertilised control plots yielding 4.4–4.6 t/ha. The 6 × 6 correlation matrix of the elements indicates 16 significant relationships out of the 21 calculated. Heavy fertiliser application ($N_{120}P_{60}K_{60}$) combined with a nitrification inhibitor poses no great risk of soil depletion but does require supplementation with sulphur-containing Ca and Mg fertilisers and micronutrients.

KEYWORDS
Carbon sequestration; soil organic carbon; stoichiometry; ionomics; phaeozems

Introduction

Climate stabilisation demands both clean energy and sequestration of carbon from the atmosphere. Carbon capture as soil organic carbon [1,2] is held back by a lack of clarity in stoichiometry, ionomics, and correlations between key components of soil productivity. Du *et al.* [3] and Terrer *et al.* [4] identify plant-available N and P as a constraint on carbon capture. The C:N:P ratio plays a crucial role in supporting plant growth and functioning of agroecosystems [5–8]. Chemical element pools and their stoichiometry are key factors in soil potential [9,10] and present thresholds for nutrient availability, particularly in the case of plant-available nutrients as opposed to absolute element contents [11].

Bossio *et al.* [1] reckon that soil carbon accounts for one-quarter of the potential of nature-based climate solutions for balancing industrial CO_2 emissions while delivering ecosystem services: 40% attributed to the protection of existing soil carbon and 60% to the restoration of depleted stocks. Accumulation of soil organic carbon is also essential for maintaining and increasing N and P reserves [12] as well as S, Ca, Mg, and trace

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elements that are functionally linked with organic matter [13,14]. Meta-analysis of 92 studies on changes in C, N, and P following conversion of natural and semi-natural systems into agricultural landscapes [15] demonstrated that C and N stocks mainly depended on soil thickness (≤30 cm), the duration of these transformations, and precipitation; phosphate status was less responsive to these factors.

Hnativ *et al.* [13] showed that carbon stocks increase only in parallel with increasing stocks of N, P, Ca, and Mg; Kirkby *et al.* [16] argued that soil carbon accumulation depends on the presence of stabilising elements such as N, P, and S. The correlation between C and P was weaker than that with N and S, but the stability of C:N:P:S ratio is a fundamental property of humus [17].

Spohn [18] showed that organic carbon accumulation in mineral soils is associated with sequestration of significant amounts of phosphorus, thanks to the formation of phosphorus-rich microbial necromass and sorption of organic phosphorus onto mineral surfaces which prevents its mineralisation (1000 kg of organic carbon in the clay fraction of arable topsoils absorbs 13.1 kg of phosphorus). Hospodarenko *et al.* [19] report changes in the total phosphorus content in Ukrainian phaeozems under the influence of long-term fertilisation: reserves in loess-derived phaeozems ranging from 0.09% to 0.27% after 55 years of fertilisation in crop rotation; at the same time, Dmytruk & Sobko [20] demonstrate that soil phosphorus content depends on several other factors.

According to Spohn & Stendahl [21], carbon concentration in boreal forest soils positively correlates with exchangeable calcium in the humus layer and negatively with soil acidity. The ratio of available N to mobile P is strongly linked to above-ground productivity; the relationship between available N and P is associated with the rate of atmospheric N fixation or its supply from fertilisers [22]. Microbial anabolism underlies many of these relationships; soil organic carbon is an accessible substrate for microbial activity [23,24] and the stoichiometric ratio of available N and P provides a better criterion of the need for mineral fertilisers compared to the stoichiometric ratios of total C:N:P in the soil. This paper evaluates the mutual influence of C, N, P, Ca, S, Mg, and K content, N content, and microelements, as well as the winter barley yield and interrelationships of total concentrations in cultivated black soils of the Western Forest-Steppe of Ukraine.

Materials and methods

The experimental fields of the Stepan Gzhytskyi National University of Veterinary Medicine and Biotechnology (Lviv) are located at lat. 49°53'53.3'N, long. 24°05'15.7'E, 263 m above sea level (Figure 1). A long-term field experiment on soil fertilisation and cultivation under crop rotation was established more than 20 years ago on Luvic Greyzemic Phaeozem (Aric) with a total carbon content of 9.8 g/kg, available nitrogen 99 mg/kg, and phosphorus and potassium contents of 75 and 80 mg/kg, respectively. Figure 1 also shows the distribution of related black soils in Western Ukraine according to the national classification.

In early spring (BBCH[1] growth stage 21–25) and at harvest (BBCH 92), auger samples were collected from the entire area of each experimental plot at a depth of 0–20 cm. Samples were collected 5–6 times along the route and then mixed to produce a combined sample that was immediately air-dried, sieved, and grounded. Agrochemical, physical,

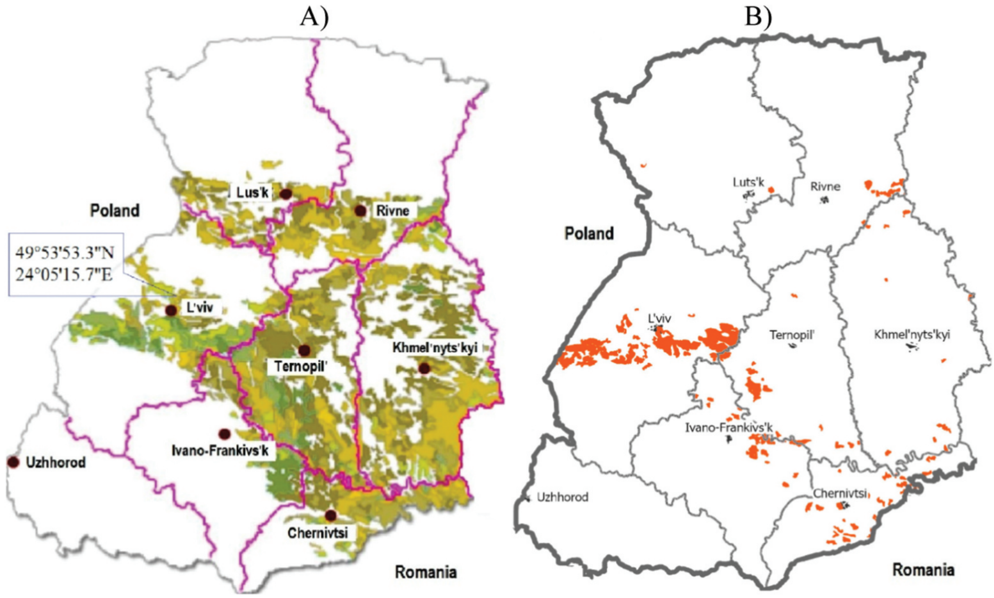

Figure 1. The Western Forest-Steppe of Ukraine, distribution of Podzolised and Podzolised gleyed soils according to the national soil classification (A). Within this group, Luvic Greyzemic Phaeozems (Aric) according to WRB (B) (https://geomap.Land.kiev.ua/soil.Html).

and physicochemical characteristics were determined using standard procedures [25] as previously published [26].

Total C, N, and S were determined using an Elementar Vario Macro Cube instrument in the Department of Soil Science and Agricultural Chemistry at the Warsaw University of Life Sciences. Total P, K, Na, Ca, Mg, Fe, Mn, Sr, Ba, Zr, Li, and Ti were determined by inductively coupled plasma-optical spectrometry (Perkin Elmer Avio 200) after sample digestion in a 5:3:1 volume ratio mixture of HF, HNO_3, and $HClO_4$. Statistical analysis of the obtained data was carried out using STATISTICA 12 [27].

Winter barley was sown after fertilisation with diammonium phosphate $(NH_4)_2HPO_4$ (DAP) + NH_4NO_3 + KCl: $N_{10}P_{26}K_{26}$) and ploughed to a depth of 20–22 cm. In spring (BBCH 21–25), nitrogen application was topped up to N_{120} using commercial urea and ammonium nitrate. Nitrapyrin as N-Lok™ which inhibits aminomonooxygenase and nitrite oxidoreductase enzymes was used as a nitrogen stabiliser at a dose of 1.7 l/ha. P and K rates were set at 60 kg/ha in the treatment variants and the control was unfertilised (Table 1).

Results and discussion

The soil used in the experimental plot was a silty clay loam with a sharp increase in clay content from 10% in the ploughed layer to 20% below. The organic carbon content in the ploughed layer was 1.09%, low for Luvic Greyzemic Phaeozems (aric); the reaction was slightly acidic but less so at greater depth, likewise hydrolytic acidity; total exchangeable bases ranged from 11.3 to 24.2 cmol/kg corresponding to medium to high levels, with a base saturation exceeding 80%. The total content of C, N, P, Ca, and Mg in the ploughed

Table 1. Fertilisation scheme in the 2020–2021 field experiment and total bioavailable element content in the arable layer (0–20 cm).

Variant code	Fertiliser scheme	Elements, g/kg						
		C	N	P	Ca	S	Mg	K
1.	**Without fertilizers – $N_0P_0K_0$**	**9.8**	**1.3**	**0.5**	**3.1**	**0.19**	**0.6**	**15.2**
2.	Background ($N_{23}P_{60}K_{60}$) + N_{97}(urea) - N_{120}	11.7	1.4	0.6	4.3	0.20	0.5	15.8
3.	Background + N_{97}(urea + N-Lok^{TM} - N_{120}	11.9	1.5	0.6	3.7	0.21	0.6	15.5
4.	Background + N_{97}(amm. nitr. - BBCH 21–25) - N_{120}	11.6	1.5	0.6	3.7	0.20	0.6	15.6
5.	Background + N_{97}(amm. nitr. + N-Lok^{TM} - BBCH 21–25) - N_{120}	11.9	1.5	0.6	3.8	0.22	0.8	16.3

layer (0–20 cm) under winter barley (average data for 2020–2021) was published in an earlier paper [14]. We now extend the study to include total sulphur and potassium, which prove to be crucial bioavailable nutrients for yield formation. The total content of C, N, P, Ca, S, Mg, and K in the ploughed layer was significantly less in the unfertilised control, except for Mg. Figures 2–4 depict the changes of total soil C, N, and P over spring and summer.

Fertilisation with N_{97} urea in spring, following $N_{23}P_{60}K_{60}$ (DAP) before autumn ploughing, increased the total content of C, N, P, Ca, S, and K. Spring application of nitrapyrin restricted microbial hydrolysis of urea, thereby contributing to an increase in the total content of C, N, and S. Replacing urea with ammonium nitrate at the same N_{97} rate did not significantly alter the total content of bioavailable elements but stabilising nitrates limited nitrate leaching and N_2O emissions [28] and probably contributed to the stabilisation of the highest total C, N, P, S, Mg, and K levels.

Considering how the total content of bioavailable elements in the soil varied according to the weather, 2021 provided significantly better growing conditions (Table 2).

In 2020, the total soil N decreased from spring tillering (BBCH 21–25) to full ripening (BBCH 92) at varying rates under different fertiliser regimes. Analysis of variance showed that all variants exhibited significantly more carbon than the unfertilised control; the least significant difference (LSD) absolute was at the level of 1043.99 mg/kg. By harvest, treatment 2 with N_{97} urea application in spring following DAP before ploughing resulted in the largest total C reduction of 6.96% (Figure 2). Spring application of N-Lok™ inhibitor limited carbon loss to 3.35% . But, replacing urea with ammonium nitrate

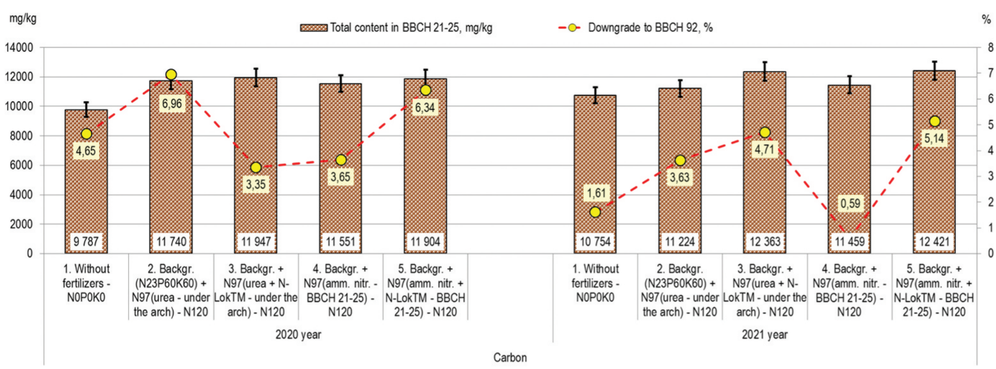

Figure 2. Loss of total carbon under winter barley during spring and summer under different fertiliser treatments (least significant difference (LSD) absolute 1043.99mg/kg; LSD relative (%) 9.07).

Table 2. Precipitation and temperature during the spring and summer vegetation months: https://meteopost.com/weather/climate/.

Month	2020 year	2021 year	Climatic mean
Precipitation, mm			
January	33	50	40
February	81	118	43
March	36	51	44
April	7	39	51
May	148	51	75
June	140	94	93
July	81	47	102
Spring-summer precipitation	**526**	**450**	**448**
Air temperature,℃			
April	8.7	5.9	7.4
May	10.8	12.7	12.9
June	18.4	18.4	16.3
July	18.8	21.7	17.5
Average air temperature	**14.2**	**14. 7**	**16.9**

reversed the effect of the inhibitor. The combination of ammonium nitrate and inhibitor had a similar effect in the more favourable 2021 season, but the combination of urea and inhibitor had the opposite effect under the warmer but drier conditions of 2021.

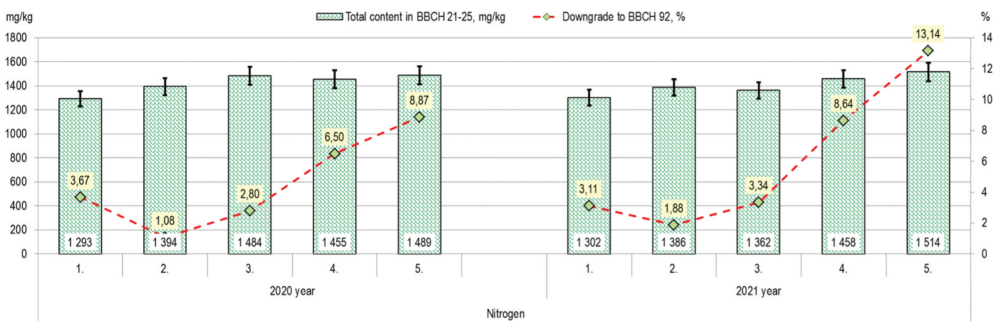

Figure 3. Loss of total nitrogen content under winter barley during spring and summer under different fertiliser treatments (least significant difference (LSD) absolute 101.88mg/kg; LSD relative (%) 7.21).

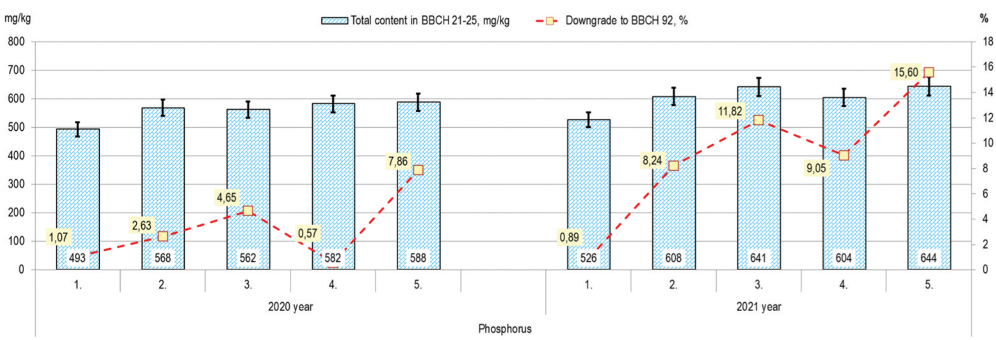

Figure 4. Loss of total phosphorus content under winter barley during spring and summer under different fertiliser treatments ($F_{calculated}$ 2.77 < $F_{05(theoretical)}$ 5.79).

Figure 3 shows a clear pattern of changes in total N content depending on fertilisation and nitrate stabilisation: the most significant reduction in total N content was associated with the application of N_{97} ammonium nitrate in spring following DAP before ploughing – a decrease of 8.87% in 2020 and 13.14% in 2021. Nitrification enzyme inhibition did not prevent unproductive nitrogen losses, which were higher in the more favourable growing season. But, significantly lower total N losses (1.08% to 3.34%) were recorded when urea was used instead of ammonium nitrate, even though the nitrification inhibitor showed no positive effect in the presence of ammonium-based fertiliser.

Loss of N was accompanied by loss of P (Figure 4). The least P losses were recorded in the unfertilised control (0.89–1.07%); greater losses were observed in the more favourable 2021 season. Meanwhile, nitrification inhibition significantly increased phosphate removal thanks to the higher crop yield.

Total soil calcium was significantly reduced in the variant receiving N_{97} ammonium nitrate along with N-Lok™ in spring, following DAP before ploughing (Figure 5). It is noteworthy that nitrification inhibition was accompanied by a significant reduction in calcium content in the unfavourable 2020 season when urea was used. Replacing urea with ammonium nitrate caused a similarly high calcium loss (25.82%) in the favourable 2021 season.

The relatively warm and moist conditions in 2021 led to the greatest total sulphur removal with the spring application of N_{97} ammonium nitrate and N-Lok™ following DAP before ploughing (20.19% reduction, Figure 6). The urea + inhibitor resulted in a sulphur loss of only 11.72%.

The spring application of N_{97} urea combined with N-Lok™ following DAP before ploughing, led to similar total magnesium losses in both study years (7.35–7.43%) (Figure 7). The ammonium nitrate + nitrate stabiliser treatment resulted in the greatest magnesium loss in the wet and cold 2020 season (9.02%) and a slightly lower loss in the more favourable 2021 season (7.39%). Lower total potassium losses were observed in the control and urea treatments (Figure 8). The spring application of N_{97} ammonium nitrate with N-Lok™ following DAP before ploughing significantly increased total potassium loss (7.62–8.20%).

Figure 9 presents the grain yields of winter barley under different treatments. The rates and forms of mineral fertilisers and application of nitrification inhibitors had

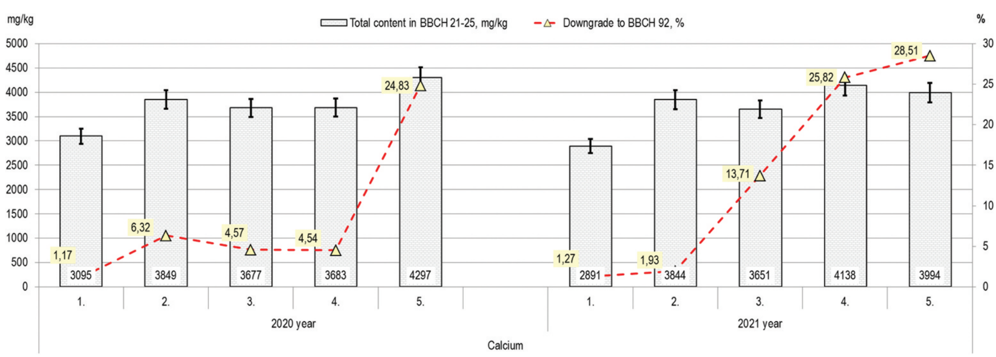

Figure 5. Calcium loss under winter barley during spring and summer under different fertiliser treatments (least significant difference (LSD) absolute 474.69 mg/kg; LSD relative (%)12.79).

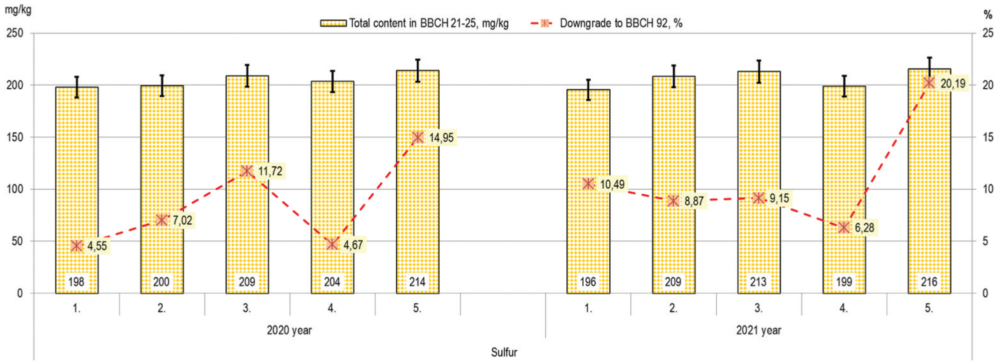

Figure 6. Total sulphur loss under winter barley during spring and summer under different fertiliser treatments (least significant difference (LSD) absolute 9.27mg/kg; LSD relative (%) 4.5).

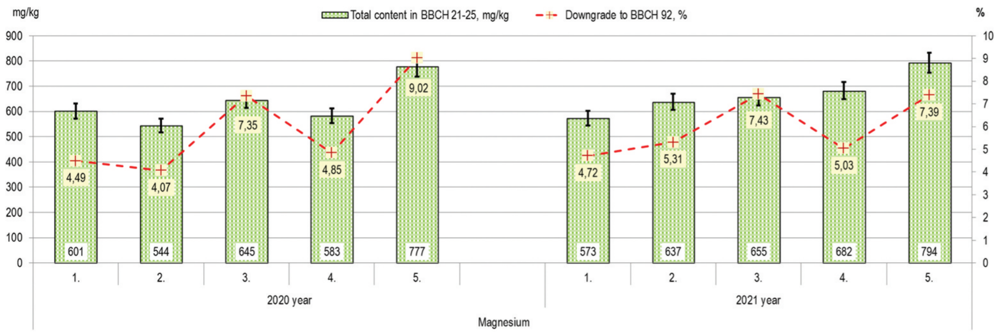

Figure 7. Loss of total magnesium under spring barley during spring and summer under different fertiliser treatments (least significant difference (LSD) absolute 113.85mg/kg; LSD relative (%)17.54).

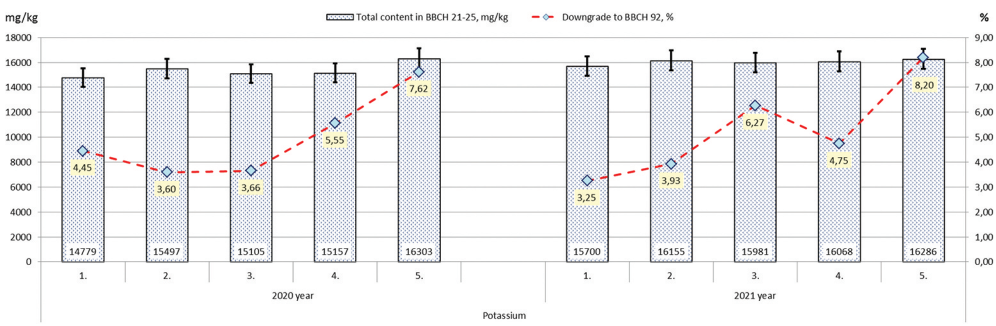

Figure 8. Loss of total potassium under spring barley during spring and summer under different fertiliser treatments ($F_{calculated}$ 1.07 < $F_{05(theoretical)}$ 5.79).

a significant impact on grain yield. Consequently, higher bioavailable element removal due to higher yield resulted in greater soil losses of essential functional components: the patterns of total element loss in the 0–20 cm soil layer were closely related to grain yield.

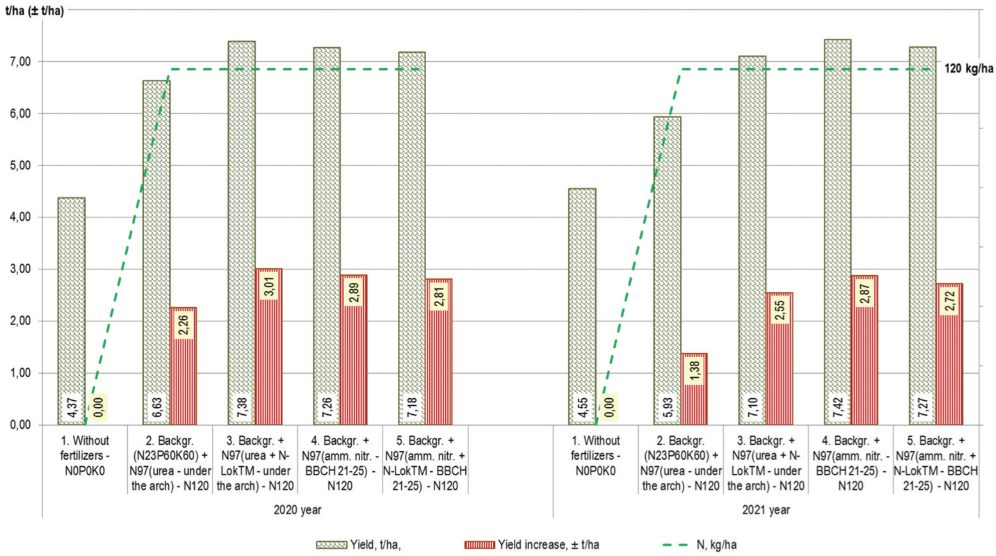

Figure 9. Changes in winter barley grain yield under different fertiliser and nitrate stabilisation treatments.

Table 3. Correlations of total bioavailable element content in Luvic Greyzemic Phaeozems (Aric) in Western Ukraine, r ±.

	N	S	P	K	Ca	Mg
C	0.69	0.44	0.80	0.55	0.26	0.53
N	X	0.63	0.67	0.50	0.67	0.48
S	–	X	0.67	0.61	0.46	0.34
P	–	–	X	0.71	0.39	0.47
K	–	–	–	X	0.51	0.55
Ca	–	–	–	–	X	0.16

Table 3 presents the strength of correlations between total C, N, P, Ca, S, Mg, and K content: the concentrations of nearly all analysed elements are significantly correlated. A particularly strong correlation was observed between total phosphorus and total carbon and potassium, as well as moderate to strong correlations between total nitrogen and all examined bioavailable elements. The natural optimal stoichiometry of chemical elements in Luvic Greyzemic Phaeozems (Aric) is the foundation for production and ecosystem services. Moreover, the total content of certain bioavailable microelements is strongly and positively correlated with the total nitrogen content (Table 4). A high

Table 4. Correlation between total and mobile element forms and total nitrogen content (N = 24) in Luvic Greyzemic Phaeozems (Aric) in Western Ukraine, r ±.

Elements											
Fe	Mn	Zr	Sr	Cu	Ba	Li	Ti	Na	Zn	Pb	Ni
Total forms											
0.57	0.44	0.28	0.33	–	0.37	0.45	0.07	0.09	–	–	–
Mobile bioavailable forms											
0.21	0.59	0.10	0.26	0.27	0.18	0.15	0.31	–	0.76	0.49	0.16

nitrogen supply in field crops was associated with higher total Fe content, total and mobile Mn, total Li, and mobile Zn and Pb. The bioavailable elements Sr, Cu, and Ti exhibited weak but positive correlations with total nitrogen content.

Table 5 presents the strength of correlations between the studied factors: the total content of C, N, Ca, and P had the strongest influence on the winter barley grain yield and its increase under the effect of fertilisers and the nitrification inhibitor.

Correlation between the reduction in total N, P, and K and yield increase was weak; for Ca and Mg, moderate; for C, insignificant. Since Ca and Mg are present only in small amounts in the applied fertilisers, these fertilisers do not compensate for their uptake by crops, and supplementation should be considered to maintain a positive and functional stoichiometry of chemical elements.

Figures 10–13 present graphical correlation models of total C, N, P, Ca, S, Mg, and K concentrations, their changes, and their relationship with grain yield and yield increases:

A higher total carbon content leads to higher barley yield (r = 0.87, Figure 10). Similarly, the yield increase is strongly correlated (r = 0.88) with total nitrogen content in the active tillering phase of the crop (Figure 11A).

The substantial reserves of bioavailable elements in Luvic Greyzemic Phaeozems, along with their drawdown in the course of yield formation under intensive fertilisation, indicate a highly productive agroecosystem. The impact of nitrapyrin was indirectly

Table 5. Correlations of total C, N, P, Ca, S, Mg, and K content with winter barley yield.

Interrelated factors	Downgrade of total content from BBCH 21–25 to BBCH 92, mg/kg	Yield, t/ha	Yield increase, ± t/ha
Carbon			
Total content in BBCH 21–25, mg/kg	0.42	0.87	0.85
Downgrade to BBCH 92, %	–	0.14	0.17
Downgrade to BBCH 92, mg/kg	X	0.25	0.28
Nitrogen			
Total content in BBCH 21–25, mg/kg	0.69	0.87	0.88
Downgrade to BBCH 92, %	–	0.45	0.43
Downgrade to BBCH 92, mg/kg	X	0.47	0.45
Phosphorus			
Total content in BBCH 21–25, mg/kg	0.86	0.75	0.70
Downgrade to BBCH 92, %	–	0.53	0.47
Downgrade to BBCH 92, mg/kg	X	0.52	0.46
Calcium			
Total content in BBCH 21–25, mg/kg	0.75	0.83	0.82
Downgrade to BBCH 92, %	–	0.62	0.59
Downgrade to BBCH 92, mg/kg[1]	X	0.61	0.58
Sulphur			
Total content in BBCH 21–25, mg/kg	0.74	0.60	0.59
Downgrade to BBCH 92, %	–	0.31	0.28
Downgrade to BBCH 92, mg/kg	X	0.33	0.31
Magnesium			
Total content in BBCH 21–25, mg/kg	0.91	0.48	0.46
Downgrade to BBCH 92, %	–	0.53	0.52
Downgrade to BBCH 92, mg/kg	–	0.52	0.51
Potassium			
Total content in BBCH 21–25, mg/kg	0.59	0.37	0.31
Downgrade to BBCH 92, %	–	0.49	0.48
Downgrade to BBCH 92, mg/kg	X	0.50	0.48

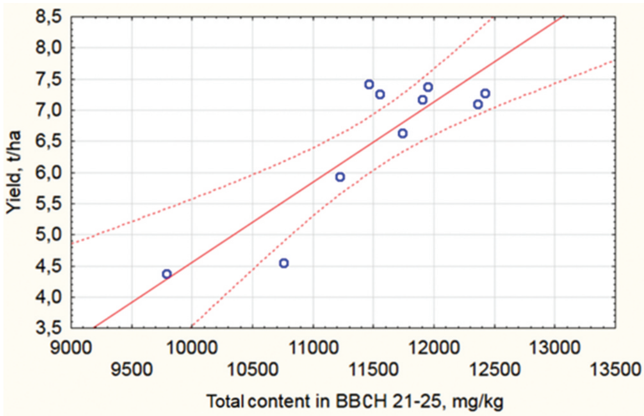

Figure 10. Dependence of winter barley grain yield on total soil carbon during active tillering (r = 0.87).

positive and its use is recommended, as in our previous studies [28]. The drawdown of the total nitrogen aligns with the trend of increasing grain yield (r = 0.47, Figure 11B). Grain yield is also strongly correlated with total phosphorus reserves during active tillering (r = 0.75, Figure 12) and moderately correlated with the decline in total phosphorus stocks up to grain maturity (r = 0.52). Additionally, grain yield is strongly correlated with total calcium reserves in the active tillering phase (r = 0.83, Figure 13A) and moderately correlated with total magnesium depletion up to the maturity phase (r = 0.53, Figure 13B).

This nutrient drawdown raises concern about the depletion of bioavailable elements that are removed from the ecosystem by the harvest. The C:N:P ratio and other bioavailable elements in soil are indicators of sustainable land use [29] and key criteria for optimal plant growth and productivity [5,30] but reserves must be maintained by adequate application of manure and fertilisers, as well as minimising losses by leaching [31,32].

Our experiments and analysis demonstrate that the total content of C, N, P, Ca, S, Mg, and K in soil decreased to varying degrees under different fertilisation systems between the spring active tillering phase and crop ripening. Both the weather and fertilisation practice influenced the drawdown of bioavailable elements but the main driver, particularly with respect to nitrogen uptake, was the high grain yield – as we reported earlier [14].

Conclusions

Phaeozems are productive soils, as demonstrated by the winter barley yield of 4.37–4.55 t/ha on the unfertilised control to 6.63–7.42 t/ha under $N_{120}P_{60}K_{60}$ fertilisation in 2021–2. They provide good drainage and water-holding capacity and optimum stoichiometry of nutrient elements, but they lack the inherent-free calcium and magnesium carbonate and extraordinary humus content of their chernozem cousins, so we hold a watching brief.

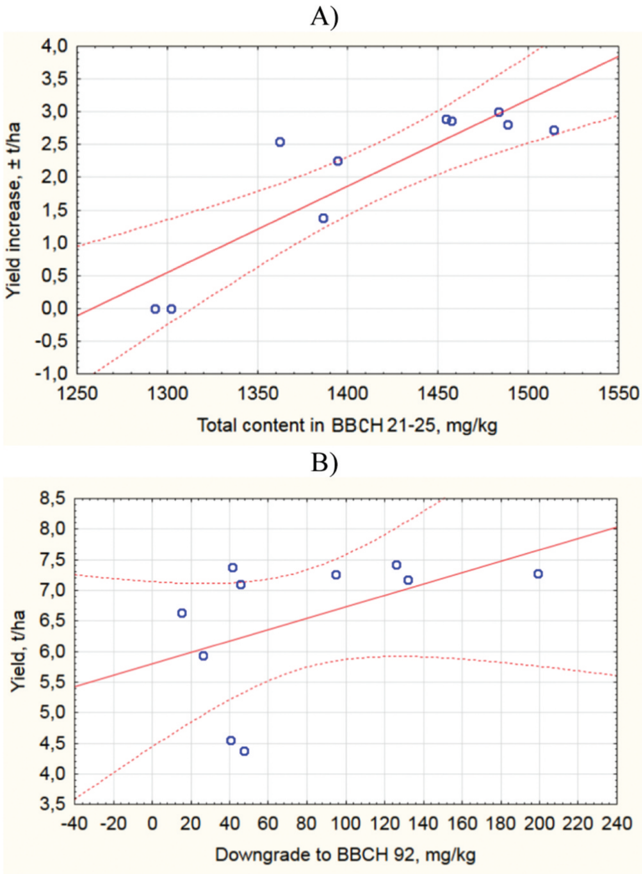

Figure 11. Dependence of yield increase on total nitrogen content in soil during the active tillering phase (A) ($r = 0.88$) and dependence of grain yield on the decrease in total nitrogen content in soil until the maturity phase (B) ($r = 0.47$).

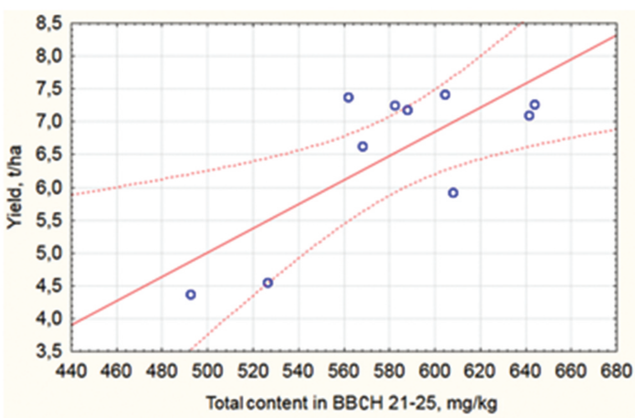

Figure 12. Dependence of grain yield on total phosphorus content in soil during the active tillering phase ($r = 0.75$).

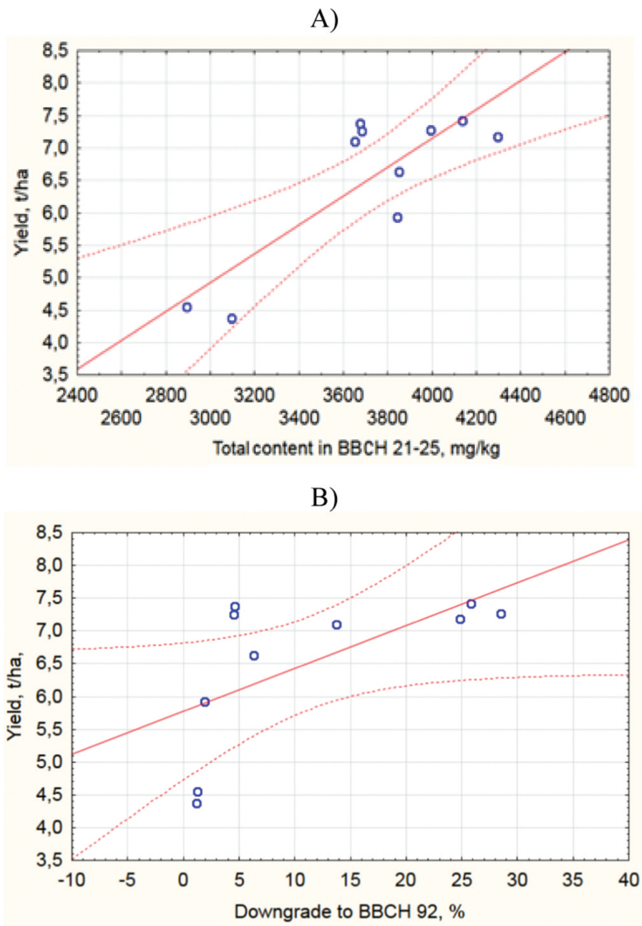

Figure 13. Dependence of grain yield on total soil calcium content (A – *r* = 0.83) during the active tillering phase, and magnesium uptake up until harvest (B – *r* = 0.62).

The total content of C, N, P, Ca, S, Mg, and K, and its vegetation changes in the arable (0–20 cm) layer of Luvic Greyzemic Phaeozems (Aric) in Western Ukraine were investigated within a long-term field experiment on rotational crops. There was a strong interdependence of the drawdown of biophilic elements, soil organic carbon, and grain yield.

Application of high rates of NPK with mineral fertilisers and stabilising nitrate formation with the N-Lok^{TM} inhibitor does not threaten excessive depletion of Luvic Greyzemic Phaeozems (Aric) in Western Ukraine but requires supplementation with sulphur-containing, calcium and magnesium fertilisers, and micronutrients, the content of which is closely correlated with the content of macroelements.

Note

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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