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Posted Date: 19 March 2026

doi: 10.20944/preprints202603.1574.v1

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Development of a Method of Secondary Use of Slags of Non-Ferrous Metallurgy for Obtaining Mineral Fertilizers

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Abstract

In this study, extracts of metallurgical slags of the former lead plant in Shymkent and Zhezkent Mining and Processing Plant are used as a liquid mineral fertilizer for growing corn. Slag extraction was carried out by the method of chemical leaching with potassium sulfate and ammonia solution in hydrogen peroxide medium. Macro- and microelement analysis of extracts from slag was carried out. Among the obtained extracts, the slag extract of the second slag store of the former lead plant is the least toxic and the richest in macro- and microelements (27.605 Ca^{2+}; 5.959 Mg^{2+}; 423.751 Cu^{2+}; 86.649 Zn^{2+}; 5.567 $Fe^{2+,3+}$; 22.652 Mn mg/L). The studied solution was diluted in the ratio of extract: distilled water 1:10 for the extract based on potassium sulfate and 1:200 for the extract based on ammonia and used to evaluate the initial development of seeds and yield of corn. Germination of seed corn and its development after 90 days did not differ from the control variant. The concentration of potentially toxic elements in the dry mass of the plant does not exceed the permissible concentration. The results showed the potential of safe application of this fertilizer in agriculture and rational utilization of industrial waste.

Keywords: metallurgical slag; mineral fertilizer; chemical leaching; waste utilization; corn

1. Introduction

Processing of metal-containing mineral waste [1] is a global challenge, as the metallurgical industry is a significant source of environmental pollution by highly toxic metals (Pb, As, Cd) and gases (H_2S, SO_2, etc.) [2]. Large volumes of solid waste are generated at former and operating metallurgical enterprises, among which slags form extensive slag heaps [3]. One of the largest slag dumps in Europe and the world, located in Poland, the volume of waste, even with regular processing, reaches more than 600 million tons [4]. Currently, in Kazakhstan, the total volume of waste from various branches of non-ferrous metallurgy reaches more than 5 billion tons, occupying over 13 thousand hectares of land [5]. The volume of accumulated metallurgical waste in the tailing dump of the Zhezkent Mining and Processing Plant (MPP) is over 1 billion tons [4], while the former lead plant in Shymkent contains approximately 1.9 million tons [6,7].

The quantity of slag produced depends on the amount of ferrous or non-ferrous metal produced. Non-ferrous metallurgy is associated with high levels of slag formation [8,9]. Table 1 presents the volumes of slag generated per ton of metal produced.

Table 1. Volumes of metallurgical slags from non-ferrous and ferrous metallurgy.

Produced metal	Volume of generated slag, t	Reference
Iron (steel)	0.12	[10]
Iron (cast iron)	0.22-0.37	[11]
Lead	0.6-0.7	[12]

Aluminum	0.2-0.5	[13]
Ferrochrome	1.1-3.5	[14]
Zinc	> 2	[15]
Copper	2.2	[9,16,17]
Magnesium	> 6	[18]
Nickel	6-16	[19]

The main chemical components of slags (wt.%) from Zn and Pb production are FeO_{total} 0.88–59.6; SiO_2 2.04–57.1; CaO 0.18–32.23; MgO 0.61–15.9; ZnO 0.03–47.3; PbO 0.002–6.4 [20,21]. Table 2 shows the concentrations of chemical elements potentially toxic to soils and plants, contained in metallurgical slags [3].

Table 2. Comparative content of potentially toxic chemical elements in metallurgical slags, mg/kg.

Element	Ferrous metallurgy slag	Non-ferrous metallurgy slag	Maximum allowable concentration (MAC) in soil [22,23]
Cd	≤128	≤14000	5
Pb	0.2-126	≤319190	32
As	≤244	75865	2
Cr	0.1-32700	≤7510	6
Co	0.03-210	0.97-24104	5
Zn	0.15-11000	13-379694	23
Cu	0.13-540	5-353580	3
Fe	0.02-61.8	0,67-62,0	2000-15000

According to Table 2, non-ferrous metallurgy slags may have a significantly greater negative environmental impact than ferrous metallurgy slags. To mitigate these impacts and ensure waste-free production, slags can be used as a secondary raw material source for metal recovery through hydrometallurgical methods and bioleaching [20,24]. The hydrometallurgical method is based on leaching slags with solutions of acids, salts, or alkalis, resulting in the transfer of metals into solution in the form of Me^{n+} ions (Reactions 1, 2):

$$ZnFe_2O_4 + 4H_2SO_4 = ZnSO_4 + Fe_2(SO_4)_3 + 4H_2O \qquad (1)$$

$$Ca[Zn(OH)_3]_2 \cdot H_2O + 3H_2SO_4 = CaSO_4 + 2ZnSO_4 + 8H_2O \qquad (2)$$

During bioleaching, metals are transferred into solution by microorganisms under acidic conditions at pH 1.5–3.0 (reactions 3, 4) [25]:

$$4FeS_2 + 14O_2 + 4H_2O \rightarrow 4FeSO_4 + 4H_2SO_4 \qquad (3)$$

$$4FeSO_4 + O_2 + 2H_2SO_4 \rightarrow 2Fe_2(SO_4)_3 + 2H_2O \qquad (4)$$

In both approaches, metals are subsequently extracted from solution by solvent extraction, electrolysis or precipitation.

In addition, metallurgical slags are used as fillers in building materials (for road construction, concrete, cement, etc.) due to their favorable physical and mechanical properties (density, hardness, melting point) [20,26].

Metallurgical slags represent a practical and affordable solution to wastewater treatment. They have been used to treat acid mine drainage in the Witwatersrand basin and the Mpumalanga coalfields in South Africa, and as a substrate in vertical flow-through wetlands for municipal wastewater treatment in La Motte d'Aigues, France [27].

Studies of the positive effects of physicochemical modification of various types of slags on crop yields and soil fertility are presented in Table 3.

Table 3. Studies on the use of metallurgical wastes to improve crop yields and soil fertility.

Metallurgical slag	*Effects of application in agriculture*
Steel slag	Application at 20 g/kg of soil enhanced the growth parameters of *Capsicum annuum L.* by 2 or more times, with sulfur (S) content in fruits exceeding that of the positive control by fourfold. Concentrations of other macro- and microelements (N, P, K, Ca, Mg, Zn, Fe, Si) in fruits remained comparable to those in the positive control [28]
Dried sludge from wet gas cleaning in a blast furnace shop and converter slag	The application increased the field yield of *Avena sativa L.* by more than 30%, with plant height increased by an average of 18% [29]
Linz-Donawitz converter slag	The addition increased soil organic carbon by 14%, readily mineralizable carbon by 42%, microbial biomass carbon by 30%, available phosphorus by 33%, exchangeable Ca^{2+} by 47%, and exchangeable Mg^{2+} by 65%. It also enhanced the rate of photosynthesis in Oryza sativa L. by 21.1 and 18%, and increased the contents of N, P and Si in straw by 20.1 and 22.2%, 17 and 18.4%, and 29.9 and 30.5% in Japonica and Indica rice varieties, respectively. Grain yield increased by 15.2 and 13.6%, straw biomass by 19.9 and 22%, and root biomass by 17.2 and 19.4% in the two varieties [30]
Steel slag	The simultaneous application of steel slag (2% by weight) and vermicompost (4% by weight) increased soil electrical conductivity by 34% compared to the control, microbial growth rate by 119%, while Cu bioavailability in contaminated soils decreased by 72%. In addition, the biomass of *Lolium Perenne L.* under the combined application of vermicompost and steel slag exceeded that of the variant with vermicompost alone by 15% [31]
Electric arc furnace (EAF) slag	The introduction of low-slag EAF additives into the soil, a combination of EAF slag and NPK, improved gas exchange parameters, with the net rate of photosynthesis being 30% higher under the combined use of NPK fertilizer and slag compared to NPK alone. It also enhanced the activity of nitrate reductase in the bean plant *Phaseolus vulgaris L.* [32]
Steel slag	The use of water-cooled slag or steel slag fertilizer over two years of testing increased the yield of table grapes *Vitis vinifera L.* by 13.5% compared to the control [33]
Blast furnace gas cleaning sludge	The introduction of highly dispersed blast furnace gas cleaning sludge (at a dosage of 0.5 to 2 t/ha) stimulated the photosynthetic activity of *Brassica napus L.* plants, increasing the average root length by 50% and stem length by 15%. Maximum seed germination was also recorded, being 7% higher than the control values [34]
Lead slag	The introduction of a 20% dose of lead slag extract increased the biomass of *Cucumis sativus L.* seedlings by 11% compared to the control [35]
Magnesium slag	Application of magnesium slag fertilizer increased lodging resistance, enhanced late-stage growth, and shortened the growing season of agricultural crops, including *Zea diploperenni L.*, *Raphanus sativus L.* [36]

Based on the studies presented in Table 3, it can be concluded that metallurgical slags possess several agronomically valuable properties, including the content of macro- and microelements in their chemical composition, improvement of soil moisture and structure, stimulation of plant growth, and improved stress resistance. Macro- and microelements such as Ca, Mg, Cu, Fe, Mn, Si and Zn are of key importance for plant growth and development [37–39]. Further study of the possibility of using slag in agriculture contributes to both environmentally friendly disposal and a reduction in the amount of industrial waste, as well as to an increase in soil fertility and the sustainability of crop production. Currently, two main methods are used for applying metallurgical slags as fertilizer: the application of solutions and the application of liquid or solid mixtures. When processing slags into liquid fertilizers, metals are converted into soluble forms, which are more easily absorbed by plants [20].

This paper examines the production of solutions through chemical leaching (extraction) of metallurgical slags and their use as alternative liquid fertilizers. The bioactivity of the resulting extracts was assessed using bioassays (various organs) of the maize plant *Zea mays L.* This high-biomass species is commonly used in ecotoxicological studies due to its sensitivity to chemical pollutants and its ability to accumulate heavy metals [40–43]. Bioassays of the developed fertilizer allow the evaluation of the bioavailability of metal compounds present in *Zea mays L.* plants.

2. Results

2.1. Characteristics of the Granulometric Composition of Metallurgical Slags

According to literature data [44–46], slag particle size has a significant impact on the efficiency of leaching processes, since it is related to both the contact surface area and the distribution pattern of mineral phases. Thus, a decrease in fraction size to d_{80}< 10 μm leads to a 7% increase in Cu^{2+} recovery during slag flotation [44]. A similar relationship was also found for electric arc slags [45], where a change in fraction size in the range from 4 to 10 mm resulted in a decrease in the concentrations of Ba^{2+}, Ca^{2+}, and Sr^{2+} in aqueous extracts. According to [46], a clear correlation is observed between the size of steelmaking slag fractions and their chemical and phase composition: large particles are predominantly enriched in Fe and Si due to the presence of the minerals magnetite (Fe_3O_4) and fayalite (Fe_2SiO_4). Whereas smaller fractions contain elevated concentrations of Mg^{2+} and Al^{3+}, which is due to the presence of amorphous and glassy phases, including aluminosilicates and magnesium-containing compounds. The grinding process produces a significant amount of dust-like and finely dispersed particles, which can also influence the intensity of leaching along with the phase and chemical composition of the material.

Thus, particle size analysis allows us to draw preliminary conclusions about the mineralogical and chemical composition of the slag, which is important for selecting effective processing methods. The particle size distribution of the slags studied prior grinding is shown in Table 5.

Table 5. Results of particle size analysis of slags.

Fraction size, mm	Content, %			
	Slag from former lead plant, storage facility 1	**Slag from former lead plant, storage facility 2**	**Slag from former lead plant, storage facility 3**	**Slag from Zhezkent MPP**
>5	83.,32	42.18	41.35	89.30
5-2	11.31	22.31	23.95	0.27
2-1	1.66	18.06	16.72	0.60
1-0,75	1.63	9.93	10.05	0.25
0,75-0,5	0.68	4.24	4.35	0.75
0,5 - 0,25	0.61	2.05	2.15	4.07
<0,25	0.80	1.25	1.45	4.77
Total	100.01	100.02	100.02	100.01

According to Figure 1 and Table 4, the slags from slag dumps 2 and 3 of the former lead smelter are finer (fraction size <5 mm—57.82% and 58.65%, respectively) and visually characterized by a solid structure and sharp particle edges, indicating their mechanical strength. Among the four samples, the slag from slag dump 1 of the former lead smelter is the most fragmented into fractions < 2 mm (4.66%). The slags from the Zhezkent MPP consist of large particles with a diameter of 5–15 cm and easily disintegrate with under minimal mechanical impact, which may indicate their low density and high porosity content. This material also differs from other slags in its loose internal structure and light brown color.

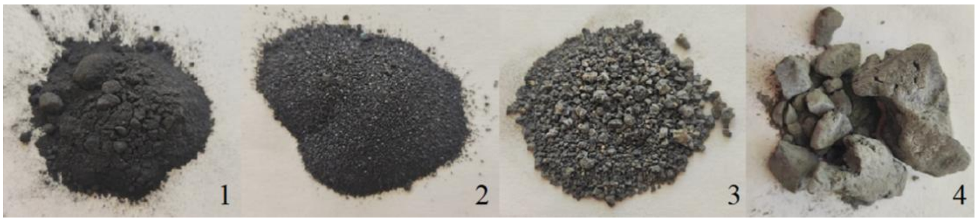

Figure 2. Metallurgical slags: 1) slag storage 1 of the former lead plant, 2) slag storage 2 of the former lead plant, 3) slag storage 3 of the former lead plant, 4) slag storage of the Zhezkent MPP.

The studied non-ferrous metallurgical slags may contain significant amounts of macro- and microelement chemical compounds valuable for plants. Thus, according to literature [5], the slags of the Zhezkazgan MPP are characterized by a high content of copper minerals, including chalcopyrite ($CuFeS_2$, 30 - 50%) of the mineral composition, covellite (CuS 5 - 20%), chalcocite (Cu_2S, 20%), and bornite (Cu_5FeS_4, 4 - 25%). In addition, they contain galena (PbS), sphalerite (ZnS), and smaller amounts of pyrite (FeS_2), hematite (Fe_2O_3), magnetite (Fe_3O_4), arsenopyrite (FeAsS), rutile (TiO_2) and inclusions of native gold (Au). Also, the slags of the former lead plant, according to data [7], contain up to 22.2% SiO_2, 46% metal oxides and 17.8% C, as well as significant amounts of Zn (9.08%) and Pb (4.22%). In [12], it is indicated that lead slags consist of 80% glassy matrix CaO–FeO–SiO_2. However, during long-term storage, slags may undergo chemical changes under enviromental influence such as solar radiation, precipitation, temperature fluctuations. Therefore, macroelements (Ca, Mg), microelements (Cu, Zn, Fe, Mn) and potentially toxic elements (As, Cd, Cr, Pb) were analyzed in the studied slags to confirm the feasibility of their chemical leaching for the purpose of obtaining liquid fertilizers. The results of the elemental analysis of the slags are presented in Table 6 [47].

Table 6. Elemental analysis of metallurgical slags before chemical leaching, mg/kg.

Element	Slag from former lead plant, storage facility 1	Slag from former lead plant, storage facility 2	Slag from former lead plant, storage facility 2	Slag from Zhezkent MPP
K	10600±1400	7500±980	4000±540	6100±830
Ca	93800±12200	92800±12000	40500±5400	19400±2500
Mg	17800±2300	15400±2000	13900±1900	18700±2500
Cu	5100±670	7200±940	3700±500	1800±250
Zn	< d.l.	< d.l.	87400±15600	4400±650
Fe	256000±33000	259000±34000	261000±34700	150900±19800
Mn	4400±5800	4900±640	3800±510	400±50
Na	10700±1400	9200±1200	5100±670	2600±360
Pb	860±110	460±60	17500±2300	2200±290
Cr	< d.l.	< d.l.	65±11	10±1
Cd	< d.l.	1,4±0,2	37±6	19±3
As	< d.l.	8±1	110±19	79±12

* Note: "< d.l. " – below the detection limit.

As can be seen from Table 6, the elemental composition of slags collected from three slag dumps of the former lead smelter and from the Zhezkent Mining and Processing Plant demonstrates significant differences in elemental content. All slag samples showed the highest concentration of Fe, ranging from 150,900 to 261,000 mg/kg. Significant amounts of Ca, Mg, and K were found in the slag from slag dump 1, indicating its potential agronomic value as a fertilizer, provided that Pb is excluded or immobilized. Heavy metals Pb (17,500 and 2,200 mg/kg), Cd (37 and 19 mg/kg), and As (79 and 110 mg/kg), indicating a potential environmental hazard, were found in the slags from slag dump 3 and the Zhezkent Mining and Processing Plant. To carry out chemical leaching and use the obtained extracts as fertilizer, according to the results of elemental analysis, a sample with the lowest concentration of potentially toxic elements (PTE) was selected, taken from slag storage facility 2 of the former lead plant.

The next stage of this study involved selective extraction of valuable elements from metallurgical slags. Chemical leaching, which involves the use of acids, salts, and alkalis as leaching agents, was chosen as an energy-saving and environmentally friendly technology for slag processing. Two leaching agents were tested. The first reagent, 15% K_2SO_4, reacts with metal oxides contained in the slag to form KOH, which acts as a source of K, as well as metal sulfates, which are sources of trace elements (Reaction 5). The second reagent, 25% NH_4OH, is more widely used in hydrometallurgical processes for metal extraction and serves as a source of the nutrient nitrogen N (reactions 6–9) [48]. Chemical leaching in both cases was carried out in the presence of H_2O_2 as an oxidizing agent. H_2O_2 plays a key role in the acid leaching process, as it promotes iron oxidation and its efficient removal from the system. The presence of H_2O_2 enhances the oxidative environment, thereby increasing the solubility of metal components and improving the overall leaching efficiency [49].

$$FeO + K_2SO_4 + H_2O_2 = Fe_2O_3 + H_2SO_4 + KOH \tag{5}$$

$$2CuFeS_2 + 16NH_3 + 8{,}5O_2 + (n + 2)H_2O= 2Cu(NH_3)_4SO_4 + 2(NH_4)_2SO_4 + +Fe_2O_3{\cdot}nH_2O \tag{6}$$

$$Cu_2AsS_4 +16NH_3+8.75O_2+2.5H_2O=3Cu(NH_3)_4SO_4+(NH_4)_2SO_4+(NH_4)_2HAsSO_4 \tag{7}$$

$$CuS+4NH_3+2O_2=Cu(NH_3)_4SO_4 \tag{8}$$

$$Cu_2S+6NH_3+(NH_4)_2SO_4+2.5O_2=2Cu(NH_3)_4SO_4+H_2O \tag{9}$$

Table 7. Elemental analysis of metallurgical slag extracts, mg/L.

Element	K_2SO_4	NH_4OH
Ca	27.605±0.199	9.398±0.084
Mg	5.959±0.064	6.088±0.046
Cu	91.365±4.327	423.751±5.750
Zn	86.649±1.094	34.352±0.882
Fe	5.567±0.025	5.028±0.013
Mn	22.652±0.163	0.185±0.001
Pb	3.343±0.108	<d.l.
Cr	<d.l.	<d.l.
Cd	0.393±0.050	0.269±0.001
As	<d.l.	<d.l.

According to Table 7, leaching with K_2SO_4 solution extracts more Ca, Zn, Mn, and Pb than with NH_4OH. A key advantage of ammonia leaching is that Cu forms soluble complex ions $[Cu(NH_3)_4]^{2+}$. Leaching with NH_4OH solution extracts four times more Cu than K_2SO_4. The resulting extracts have similar concentrations of Mg, Fe, and Cd, while Cr and As remain below the detection limit. Possibly due to the presence of poorly soluble oxides and silicates (e.g., fayalite, magnetite), the low recovery at high element concentrations in the slag is confirmed.

The degree of leaching may be related to the composition of the vitreous and crystalline phases, slag cooling conditions (rapid quenching or slow cooling), and the content of residual sulfur or other fluxes. A common factor complicating the recovery of metals from slag by acid solvent extraction is silica precipitation [50]:

$$2FeO{\cdot}SiO_2+2H_2SO_4 \rightarrow 2FeSO_4+H_4SiO_4\downarrow \tag{10}$$

Thus, the obtained results indicate the possibility of selective element extraction using the selected reagents. Extraction efficiency depends on the chemical composition of the slag and the nature of the reagent.

The resulting metallurgical slag extracts were tested as mineral fertilizers for yield in *Zea Mays L.* maize plants. To assess fertility, the agrochemical composition of the applied soil was analyzed. The results of soil texture analysis are presented in Table 8.

Table 8. Texture of the surface soil layer.

Fraction size, mm	>5	5-2	2-1	1-0,75	0,75-0,5	0,5 - 0,25	<0,25
Mass fraction, %	12,03	9,29	9,24	14,30	24,62	22,97	7,56

As shown in Table 8, according to Kachinsky's classification, the soil under study is classified as loose sand in terms of mechanical composition [51]. Loose sandy soils are characterized by low strength and weak structural stability, which limits their water-holding capacity and increases erosion [52]. Nutrients are rapidly leaching, limiting plant yields and the effectiveness of fertilizers [53].

The results of the agrochemical analysis of the soil before planting, on which the pot experiments were conducted, are presented in Table 9.

Table 9. Chemical characteristics of the soil.

Parametr	Measured value	Optimum value [54]
pH in water	8,5	6-7
Organic matter, %	1,2573 ± 0,5157	>5
N, g/kg	11,9 ±0,03	30-40
K, g/kg	0,0386 ± 0,00015	20-30
P, g/kg	0,2286±0,0086	3-5
Ca, g/kg	0,4853 ±0,0051	2,5-8
Mg, g/kg	0,0531 ±0,0001	1,5-6
Cu, mg/kg	0,8±0,05	5-25
Zn, mg/kg	8,4±0,09	20-70
Fe, mg/kg	396,5±7,5	30-250
Mn, mg/kg	3,0±0,1	20-150
Pb, mg/kg	2,3±0,027	-
Cr, mg/kg	12,1±0,01	-
Cd, mg/kg	<п.о.	-
As, mg/kg	<п.о.	-

Note: «-»-no data available.

The presented agrochemical parameters indicate low contents of organic matter as well as macro- and microelements. According to [55], deep loamy and loam-clay soils are most favorable for growing corn, providing more stable yields. To improve seed viability in loose sandy soils, corn seeds were subjected to pre-sowing treatment with fertilizer solutions to enhance the early availability of macro- and microelements from slags.

The laboratory germination results (Figure 2) showed good germination and full germination of corn seeds in all three experimental conditions: 18 seeds germinated on the 4th day, i.e., the germination energy was 81%, and 22 seeds germinated on the 7th day (germination rate was 100%). This indicates that fertilizer solutions do not negatively affect the initial germination rates of seeds and provide additional nutrients at an early stage, which is particularly important for loose sandy soils prone to rapid nutrient leaching.

Figure 2. Laboratory germination assessment of corn seeds using different extractants: 1 - control (distilled water), 2 - 1:200 extract with NH_4OH solution, 3 - 1:10 extract with K_2SO_4 solution.

After 7 days, the germinated seeds were transplanted into soil. Morphometric growth parameters 90 days after planting are presented in Table 10.

Table 10. Biometric growth parameters of *Zea mays L.* plants.

Biometric parameter	Seed treated with K_2SO_4–based extract	Seed treated with NH_4OH – based extract	Control (no fertilizer extract treatment)
Plant height, cm	158,5±10,14	167,5±7,31	165±9,72
Stem length, cm	149,5 ±15,53	155,5±13,82	154,7±14,35
Root length, cm	25±1,68	22±1,43	24±1,54
Stem diameter, cm	1,56±0,02	1,62±0,01	1,59±0,02
Number of leaves, pcs	13±1	9±1	13±1
Number of cobs, pcs	1	1	1
Cob diameter, cm	4,9±0,15	5,1±0,09	4,9±0,16
Cob length, cm	29±0,71	28±0,49	28±0,65
Fresh plant biomass, g	379,39±29,61	338,93±27,15	381,0791±31,67
Dry aboveground biomass, g	115,53±14,67	73,6151±8,96	100,3553±8,61
Fresh root biomass, g	10,89±0,83	12,4506±0,71	8,9676±0,75
Dry root biomass, g	10,85±0,21	11,0562±0,84	8,1235±0,54

Plants grown with an ammonia extract demonstrated the highest values for stem height, diameter, stem length, ear diameter, and root wet and dry biomass. This is due to the fact that the plant received N early in growth, when it is most needed by corn. N supports root development and healthy seedling growth. The use of a K_2SO_4-based extract promoted long ear formation and the accumulation of plant dry biomass [57]. A study [60] demonstrated a direct relationship between the concentration of applied K and ear length. The potassium extract, along with K, contained Ca, Mg, and Cu compounds, which promoted better absorption of other elements [58].

Fertilizer use affects the elemental composition of plant organs. Furthermore, the widespread use of NPK fertilizers leads to increased yields. However, the use of micronutrient fertilizers is associated with certain difficulties, as plants require low concentrations of micronutrients. Therefore, micronutrients are most often added directly to basic fertilizers during their production. The results of elemental analysis of corn plant organs are shown in Table 11.

Table 11. PTE concentrations in *Zea mays L.* plant organs (dry weight, mg/kg).

Roots	Solution	Cr	Pb	Cu	Zn
	K_2SO_4	1,515± 0,0174	6,554 ±0,387	12,490±0,470	53,146±0,852
	NH_4OH	0,9617± 0,017	6,847±0,501	6,090±0,522	45,348±2,909
	H_2O	3,029±0,149	10,382±0,350	9,262±0.172	70,218±2,716
Stems	K_2SO_4	< п.о.	6,520± 0,710	10,328±1.8	41,971±1,619
	NH_4OH	< п.о.	< п.о.	1,423± 0,1317	80,627±2,599
	H_2O	< п.о.	< п.о.	0,446±0,0422	104,281± 4.978
Leaves	K_2SO_4	2,319±0,054	< п.о.	4,341± 0,067	44,324±1,116
	NH_4OH	< п.о.	3,179±0,595	3,078±0,328	53,979±2,827
	H_2O	< п.о.	3,359±0,053	2,491±0,126	34,112±1,190

Cobs	K_2SO_4	< п.о.	< п.о.	1,220±0,221	45,438±0,857
	NH_4OH	< п.о.	< п.о.	2,739±0,239	55,661±2,233
	H_2O	< п.о.	< п.о.	3,506±0,199	46,382±5,331
MAC in plants, mg/kg [22]		5-30	30-300	5-30	100-400

For elemental analysis, toxicants with low MAC values in plant tissues according to Kabata-Pendias (Table 15) were selected, to assess an assessment of the potential phytotoxicity of the studied fertilizer.

As shown in Table 11, the concentrations of the studied elements in corn organs does not exceed the established MACs values. When soaking seeds in an extract containing K_2SO_4 with elevated levels of K, Ca, Mg, Cu, Zn, Fe, and Mn, plants accumulated Cu and Pb in the roots, stems, and leaves, and Cr in the leaves. Soaking seeds in an extract containing NH_4OH promoted the accumulation of Zn in the stems of mature plants.

3. Discussion

In accordance with the recommendations of Kabata-Pendias and Pendias [22] (Table 12), the translocation coefficient (TC) of elements is calculated to assess the degree of accumulation of chemical elements in plant organs (Table 13).

Table 12. The degree of accumulation of pollutants depending on the TC.

TC value	Degree of uptake
KT < 0,01	no uptake
0,01 ≤ KT≤0,1	weak uptake
0,1 ≤ *KT≤ 1,0*	moderate uptake
1,0 ≤ *KT*	intensive uptake

Table 13. Translocation coefficients of the plant *Zea mays L.*

Element	average concentration in stems / average concentration in roots			average concentration in leaves / average concentration in roots			average concentration in cobs / average concentration in roots		
	K_2SO_4	NH_4OH	H_2O	K_2SO_4	NH_4OH	H_2O	K_2SO_4	NH_4OH	H_2O
Cr	1,531*	—	—	—	—	—	—	—	—
Pb	0,995	—	—	0,485	0,464	0,324	—	—	—
Cu	0,827	0,233	0,048**	0,347	0,505	0,269	0,097**	0,449	0,378
Zn	0,789	1,778*	1,485*	0,834	1,190*	0,486	0,855	1,227*	0,661

Note: «—» - concentration below detection limit. *- intense accumulation. ** - weak accumulation.

TC confirmed the general distribution pattern for Cr, Pb, and Cu in plants: roots > stems > leaves. When seeds were treated with K_2SO_4, intensive Cr accumulation was observed in the stems relative to the roots. Cr translocation is likely related to the presence of mobile ions in the K_2SO_4 extract. The TC of Pb in stem cells was significantly lower than in leaves, reflecting the limited mobility of Pb. When seeds were treated with NH_4OH, intensive accumulation was observed for Zn, which is related to the synergistic interaction between Zn and N.

4. Materials and Methods

4.1. Sampling and Sample Preparation of Metallurgical Slags

Slag samples were collected from three slag dumps of the former Shymkent lead smelter and from one slag dump of the Zhezkent MPP in October 2024 (Figure 1). Spot samples were collected according to a route pattern at a depth of 0–10 cm. The combined sample weight from each slag dump was 8–10 kg. After collection, the slag was mixed on a clean surface using a spatula, then air-dried

doi:10.20944/preprints202603.1574.v1

and stored in a dry, ventilated area. The air-dried samples were subjected to particle size distribution and elemental analysis.

4.2. Slag Granulometric Analysis

The slag granulometric composition of the slag was determined using the sieve method with a sieve analyzer (MITR ZDS-200W, China). A 200 g sample of the average slag mass was placed on the top sieve and sifted through sieves with aperture diameters of >5; 5-2; 2-1; 1-0.75; 0.75-0.5; 0.5-0.25; and <0.25 mm for 10 minutes. After sifting, each fraction was weighed on an analytical balance with an accuracy of ±0.0001 g. The mass fraction of each fraction was calculated using the following formula (1):

$$\omega = \frac{m}{M} * 100\% \tag{1}$$

where ω is the mass fraction (%); m is the fraction mass (g); M is the total slag sample mass (g).

4.3. Elemental Analysis of Slags

Elemental analysis of solid slags was performed using atomic emission spectroscopy with an Agilent 7700x mass spectrometer (Agilent Technologies, USA) and an iCAP 6300 Duo inductively coupled plasma atomic emission spectrometer (Thermo Scientific, USA), following the method described in article by Sabitova A. et al. [44].

4.4. Chemical Leaching of Slags and Elemental Analysis of Leachates

Chemical leaching was performed using a 15% K_2SO_4 solution and a 25% NH_4OH solution following the study by Katarzyna M. et al. [35]. To prepare a 15% K_2SO_4 solution, 150 g of salt was dissolved in 0.85 L of distilled water. To obtain 10% H_2O_2, 1 L of a 37% H_2O_2 solution was diluted in 2.7 L of distilled water. A 25% NH4OH solution was used without further dilution.

A 50 g slag sample with a particle size of <1.0 mm obtained after preliminary grinding was mixed with 100 mL of leaching agent in a 10% H_2O_2 medium at a 1:1 volume ratio in 500 mL conical flasks (in triplicate). The flasks were placed on a laboratory shaker (Ikeme Lab, China) for 1 hour and subsequently centrifuged using a centrifuge (ELMI SkyLine CM-6M, Latvia). After separation of the solid residue, the resulting extract was diluted with distilled water at a 1:10 volume ratio and analyzed for elemental composition using an inductively coupled plasma mass spectrometer (ICP-MS, Varian 820, Australia) [44].

4.5. Soil Sampling and Sample Preparation

Soil sampling was carried out in a residential area of Semey, Republic of Kazakhstan. Sampling was performed using the quadrat method over a total area of 16 m^2. Within each quadrat, soil samples were collected from five points at a depth of 0–20 cm (surface horizon) and combined to obtain a composite sample weighing approximately 100 g. The samples were air – dried, homogenized, and sieved. The mechanical (particle-size) composition of the soil was determined by the dry sieving method using a set of laboratory sieves with mesh sizes ranging from 5.0 to 0.25 mm, arranged sequentially from coarse to fine fractions.

Soil agrochemical properties, including hygroscopic moisture content, actual pH, organic matter content, total nitrogen (Kjeldahl method), and elemental composition, were determined using standard physicochemical methods. The 1-2 mm air-dried soil fraction was used for all analyses.

For pH determination, a soil suspension was prepared in distilled water at a soil-to-solution mass ratio of 1:2.5. The suspension was shaken for 60 min using a laboratory shaker (Ikeme Lab, China) and allowed to settle for 60 min. It was then stirred again for 10 s, after which the pH was measured using a pH meter (INESA ZDJ-4A, China) equipped with a glass electrode.

The organic matter content was determined by the gravimetric loss-on-ignition method. Soil samples were calcined in a muffle furnace (SNOL LSF01, Lithuania) at 525-550^{o}C until constant mass was achieved. The organic matter content was calculated according to Equation (2).

$$\omega = \frac{m1-m2}{m1} *100\ \% \tag{2}$$

where m_1 is the dry sample mass, g; m_2 is the sample mass after ignition, g.

For elemental analysis, acid digestion of dry soil was performed. A 2.00 g sample of soil was placed in a round-bottomed flask, 20 ml of 3% HNO_3 solution was added, and the mixture was shaken on a reciprocating shaker (Ikeme Lab, China) for 60 minutes. After settling, the resulting soil suspension was filtered through blue ribbon filter paper. The filtrate was then diluted with distilled water at a 1:50 volume ratio and analyzed using a ICP-MS (VARIAN 820, Australia). Formula (3) was used to calculate the concentration of chemical elements in the sample:

$$C = \frac{a \times 50}{2 \times 1000} \tag{3}$$

where C is the element concentration, mg/g; a is the element concentration value displayed by the instrument, mg/L; 50 is the extract volume, mL; 2 is the soil sample weight, g; 1000 is the conversion factor for converting volume from mL to L.

4.6. Determining the Laboratory Germination of Zea mays L. Seeds.

To determine the bioactivity of metallurgical waste extracts, they were diluted with distilled water. Filter paper in plastic containers was then sprayed with 10.5 mL of the solution using a spray bottle. The following conditions were used: 1 – control (H_2O), 2 – with a 1:200 solution (NH_4OH), 3 – with a 1:10 solution (K_2SO_4). The elemental composition (mg/L) of the extracts after dilution is shown in Table 4.

Table 4. Composition of metallurgical waste extracts.

Element	Ca	Mg	Cu	Zn	Fe	Mn	Pb	Cr	Cd	As
K_2SO_4	2,509	0,542	8,306	7,877	0,506	2,059	0,304	<п.о.	0,0357	<п.о.
NH_4OH	0,047	0,030	2,108	0,171	0,025	0,0009	<п.о.	<п.о.	0,001	<п.о.

Corn seeds (22 seeds per 17 x 26 cm container) were placed between layers of moistened filter paper rolled into roll. The hermetically sealed containers with seeds were germinated in a light-tight growbox at a temperature of 20–25°C. The containers were ventilated once daily for 30 minutes and moistened as needed with distilled water [45]. Germination energy was determined on the 4th day, and germination rate on the 7th day, according to formulas (4) and (5):

$$E = \frac{\text{number of germination seeds}}{total\ number\ of\ seeds\ in\ the\ sample} *100\% \tag{4}$$

$$G = \frac{\text{viable seeds}}{total\ number\ of\ seeds\ in\ the\ sample} *100\% \tag{5}$$

where E is the seed germination energy, %; G is the laboratory germination rate, %.

4.7. Elemental Analysis of Zea mays L. Plant Organs.

To study the subsequent development of the plants, small-plot field pot experiments were conducted. Plant seedlings obtained from the laboratory germination experiment were transplanted into open field conditions and grown for 90 days, with regularly irrigation and mechanical weed removal. Field experiment variants: 1) seeds treated with an NH_4OH-based extract; 2) seeds treated with a K_2SO_4-based extract; 3) seeds without extract treatment (control). At the end of the experiment, plant height, stem diameter, number of leaves and cobs, and fresh and dry biomass of the aboveground parts and roots were determined.

Samples of leaves, stems, roots, and cobs were washed and dried in a drying oven (SNOL 58/350, Lithuania) at 105°C to constant weight, after which they were ground to a homogeneous state in a coffee machine. For dry ashing, a 5-g portion of the dried sample was transferred to porcelain crucibles and calcined in a muffle furnace (SNOL LSF01, Lithuania) at 450-500°C for 5-8 hours. Ash extraction was performed in concentrated HNO_3 (65%), at a solid phase (g) to solution (mL) ratio of 1:10. The reaction was carried out in an ADS Multi Acid Digestion System (Spectromart, Russia) at 120°C for 2 h. The extraction solutions were passed through blue ribbon filters and their volume was adjusted to 50 ml with distilled water. The concentrations of Ca, Mg, Cu, Zn, Fe, Mn, Cr, Cd, Pb, As, and Cd in the resulting extracts were determined using a ICP-MS (VARIAN, Australia) [44].

The translocation coefficient (TC), defined as the ability of PTE to be transferred from the roots to the aboveground parts of agricultural crops, was calculated using formula (6):

$$\text{TC} = \frac{C_1}{C_2} \tag{6}$$

where c_1 is the average concentration of PTE in the aboveground parts of the plant, mg/kg; c_2 is the average concentration of PTE in the root, mg/kg.

5. Conclusions

Elemental analysis revealed the lowest PTE content in metallurgical slags from the former Shymkent lead smelter and the Zhezkent mining and processing plant. Chemical leachates from these metallurgical wastes with potassium sulfate contained more than threefold higher concentration of Ca, Zn, and Mn contents compared to the ammonia extract. In contrast, the ammoniaextractant demonstrated more than fourfold highter Cu extraction efficiency.

The bioactivity of the obtained extracts was assessed using bioassays (seeds and various organs) of the *Zea mays L.* corn plant. A 100% laboratory germination rate was established after pre-sowing treatment by soaking in diluted extracts of 1:10 $K_2SO_4 : H_2O$ and 1:200 $NH_4OH : H_2O$. Seed treatment with the ammonia extract had the greatest positive effect on the root system, stems, and ears of the mature plant. Elemental analysis of plant organs revealed the accumulation of Cu after seed treatment with the K_2SO_4 extract and Zn after using the NH_4OH extract, as well as the absence of toxic metal concentrations. The experiment demonstrates the potential of using metallurgical slag extracts in the production of liquid mineral fertilizers.

Author Contributions: "Conceptualization, methodology, Zh. Kassymova.; validation, Zh. S. Kassymova, B.B. Bayakhmetova and A.N. Sabitova.; formal analysis, A.N. Sabitova; investigation, R.M. Mukiyanova; resources, A.N. Sabitova; data curation, R.M. Mukiyanova; writing—original draft preparation, R.M. Mukiyanova; writing—review and editing, Zh.S.Kassymova; supervision, A.N. Sabitova.; project administration, A.N. Sabitova; funding acquisition, A.N. Sabitova All authors have read and agreed to the published version of the manuscript." Please turn to the CRediT taxonomy for the term explanation. Authorship must be limited to those who have contributed substantially to the work reported.

Funding: "This research was funded by Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan, grant number BR24993178 "Development of innovative technology for processing secondary resources—waste from metallurgical industries, by-products of oil refining and oil production".

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

MPP	Mining and Processing Plant
ICP-MS	Inductively coupled plasma mass spectrometer
MAC	Maximum allowable concentration
TC	Translocation coefficient

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