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ZHAYREM OVERBURDEN ACID LEACHING STUDY

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Abstract: *Introduction*. Most deposits of manganese ores in Kazakhstan are mined in an open way. This creates large waste dumps that are not disposed of and have a negative impact on the environment. Since these wastes contain various valuable components, they can be used as an alternative raw material source in the production of various inorganic materials. However, there is no practice of involving overburden waste in production in Kazakhstan. *The purpose of this work* is to study the process of leaching overburden waste from the Zhayrem deposit with various mineral acids and the possibility of using the resulting solutions as liquid mineral fertilizers enriched with trace elements. *Results and discussion*. The process of leaching overburden waste from the Zhayrem manganese ore deposit with various mineral acids was investigated. It has been shown that the optimal reagents of the leaching process are nitric and orthophosphoric acids. It was revealed that leaching of overburden waste with 10% solutions of these acids at Т: Ж = 1:25 and a temperature of 50 ° C leads to the formation of productive solutions that can be recommended for use as a basis for the production of liquid phosphorus or nitrogen fertilizers containing the trace element manganese.

Key words: manganese ores, overburden waste, acid leaching, productive solution.

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1. Introduction

Kazakhstan is home to over 100 manganese deposits and occurrences, grouped into 19 major deposits. Approximately 60% of the reserves contain 10–20% manganese, 32% contain 20–30%, and 11% have manganese concentrations above 30%. The total reserves, including explored deposits, amount to about 700 million tons. About 98% of these explored reserves are located in Central Kazakhstan, with approximately 70% comprising iron-manganese ores [1–2].

One of the largest industrial enterprises in Kazakhstan engaged in manganese ore mining is the JSC "Zhairem Mining and Processing Plant", commissioned in 1972 to develop iron-manganese and barite-polymetallic deposits in the Atasui ore district [3–4]. Manganese ore is extracted exclusively through open-pit mining, which has led to the accumulation of large volumes of overburden rocks. These overburden materials are currently underutilized and disposed of in waste dumps [3].

Overburden rocks refer to geological materials overlying ore bodies and removed during surface mining operations to access valuable minerals [5]. The mineral composition of overburden and host rocks in polymetallic deposits is generally diverse and predominantly composed of non-metallic minerals such as shales, quartzites, clays, limestones, and granites, with silicon compounds as the main components. Consequently, one of the main directions for their reuse is the production of binding agents and construction materials [6–8].

Although overburden utilization is largely absent at most deposits in Kazakhstan, including the Zhairem deposit, investigating the potential for converting this anthropogenic raw material into new inorganic products is a relevant scientific and practical challenge. Its successful implementation may foster the development of new industries, diversify the country's chemical product range, and improve the environmental conditions around mining operations.

Manganese is a vital micronutrient in phosphate fertilizers. It plays a role in redox reactions, forms part of many enzymes, facilitates carbon dioxide reduction in photosynthesis, aids phosphorus translocation in plants, and affects carbohydrate and protein metabolism [9]. Conventional manganese fertilizers typically use individual chemical compounds such as manganese sulfate [9-10], which increases costs and limits accessibility for agricultural producers.

Therefore, it is scientifically and practically relevant to explore the potential of using metallurgical waste, particularly overburden waste from the Zhairem deposit, as a raw material source of micronutrients for producing mineral fertilizers.

This study aims to investigate the acid leaching process of Zhairem overburden using various mineral acids and assess the feasibility of using the resulting leachates as micronutrient-enriched liquid phosphate fertilizers.

2. Experimental Section part

The study focused on crushed overburden materials from the Zhairem deposit, characterized by a brownish color with earthy tones and particle sizes up to 5 mm.

The chemical composition of the raw overburden and solid residues after acid leaching was analyzed using energy-dispersive X-ray fluorescence (EDXRF) with a Rigaku NEX CG spectrometer.

The phase composition was determined by X-ray diffraction (XRD) using a DRON-3 diffractometer with CuKα radiation and a β-filter. Parameters: U = 35 kV, I = 20 mA, scan mode: θ–2θ, scan speed: 2°/min. Phase identification was based on the ICDD PDF2 database using the equal-weight and artificial mixture methods to determine the semi-quantitative phase proportions.

Sulfuric (H_2SO_4), phosphoric (H_3PO_4), and nitric (HNO_3) acids at 40 wt.% concentration were used as leaching agents, with a solid-to-liquid (S:L) ratio of 1:25. Each leaching experiment used 2–2.5 g of overburden and 50 mL of acid. The mixture was stirred for 30 minutes on a shaker, filtered, washed to neutral pH (6–7), dried at 105°C for 1 hour, and then analyzed using XRF and EDXRF methods. Filtrate composition was determined using titrimetric and photocolorimetric methods. Experiments were conducted at 25°C and 50°C.

3. Results and discussion

The relative solubility (%) of the overburden in different acids was determined gravimetrically (Table 1). The chemical and mineral compositions before and after leaching are shown in Tables 2 and 3.

Table 1 — Dependence of overburden solubility on acid type and temperature

Temperature (°C)	Leaching Agent		
	H_3PO_4	HNO_3	H_2SO_4
	Solubility (%)		
20	59.50	62.50	38.20
50	61.56	60.20	28.05

Table 2 – Elemental Composition of Solid Residues After Acid Leaching of Zhairem Overburden Waste Using Mineral Acids at Different Temperatures (Based on EDXRF Data)

Content of Main Components, wt.%	Composition of Initial Overburden Waste	Leaching Agent					
		H_3PO_4		HNO_3		H_2SO_4	
		Temperature (°C)					
		20	50	20	50	20	50
MgO	4.71	2.81	0.00	2.94	2.93	3.63	0.00
Al_2O_3	5.35	6.13	5.78	7.09	7.50	5.73	4.58
SiO_2	23.90	39.20	39.80	41.80	48.90	31.30	27.70
P_2O_5	0.10	3.25	9.04	0.07	0.00	0.07	0.00
SO_3	1.01	0.81	0.89	0.77	0.61	1.60	22.10
K_2O	0.57	0.75	0.84	0.86	1.00	0.52	0.56
CaO	29.90	0.32	0.30	0.37	0.30	14.90	12.60
TiO_2	0.34	0.41	0.43	0.38	0.50	0.62	0.39

MnO	24.50	32.60	29.20	32.20	27.10	30.60	21.60
Fe_2O_3	6.52	11.70	11.60	11.70	9.82	9.24	8.60
BaO	1.23	0.75	0.95	1.06	0.42	0.55	0.79
PbO	0.18	0.15	0.13	0.16	0.12	0.17	0.19

Maximum solubility (62.5%) was observed with 40% HNO_3 at room temperature. Increasing the temperature to 50°C slightly decreased solubility to 60.2%. H_3PO_4 showed similar behavior (59.5–61.56%), while H_2SO_4 demonstrated significantly lower solubility, decreasing from 38.2% at 20°C to 28.05% at 50°C **(**Table 1**).**

The low solubility in sulfuric acid is attributed to the formation of poorly soluble $CaSO_4$ as per reaction:

$$CaCO_3 + H_2SO_4 \rightarrow CaSO_4\downarrow + CO_2\uparrow + H_2O \quad (1)$$

XRF and XRD data confirmed $CaSO_4$ precipitation on particle surfaces, hindering further leaching. XRD also revealed the presence of $CaSO_4 \cdot 0.5H_2O$ and $CaSO_4 \cdot 2H_2O$ in the solid residue (Table 3).

Table 3 – Semi-Quantitative X-Ray Phase Analysis Results of Zhairem Overburden Rocks and Solid Residues After Acid Leaching at 20°C

Phase Name |	**Chemical Formula**	**Pre-Leaching Waste Content, wt.%**	**Content, wt.%**		
			H_3PO_4	HNO_3	H_2SO_4
Calcite	$CaCO_3$	29.5	0.0	0.0	46.0
Kaolinite	$Al_2(Si_2O_5)(OH)_4$	19.8	0.0	0.0	0.0
Braunite	$(Mn_2O_3)_3MnSiO_3$	14.6	35.0	48.0	21.0
Quartz	SiO_2	10.7	33.0	34.0	23.0
Hematite	Fe_2O_3	9.6	6.0	6.0	2.0
Barite	$BaSO_4$	8.5	6.0	3.0	3.0
Albite	$Na(AlSi_3O_8)$	3.9	10.0	8.0	2.0
Muscovite	$KAl_2(AlSi_3O_{10})(OH)_2$	1.8	0.0	0.0	0.0
Orthoclase	$KAlSi_3O_8$	1.7	0.0	0.0	0.0
Birnessite	$Na_{0.35}Mn_2O_4 \bullet 1.5H_2O$	0.0	3.0	0.0	3.0
Lipscombite	$Fe_{2.95}(PO_4)_2(OH)_2$	0.0	7.0	0.0	0.0

Reactions with phosphoric and nitric acids proceeded with significant calcium removal due to the following:

$$CaCO_3 + 2H_3PO_4 \rightarrow Ca(H_2PO_4)_2 + CO_2\uparrow + H_2O \quad (2)$$
$$CaCO_3 + 2HNO_3 \rightarrow Ca(NO_3)_2 + CO_2\uparrow + H_2O \quad (3)$$

At elevated temperatures and with decreased H_3PO_4 availability, secondary phosphate minerals such as insoluble aluminum and iron phosphates form:

$$2(Al,Fe)(H_2PO_4)_3 \rightarrow (Al,Fe)_2(HPO_4)_3 + 3H_3PO_4 \quad (4)$$

$$(Al,Fe)(H_2PO_4)_3 \rightarrow (Al,Fe)PO_4 + 2H_3PO_4 \quad (5)$$

This is confirmed by the XRD data (Tables 3–4), which show the presence of the lipscombite phase $Fe_{2.95}(PO_4)_2(OH)_2$ in the solid residue after leaching with phosphoric acid. The XRD findings are further supported by the EDXRF results (Table 2).

Table 4 – Semi-Quantitative X-Ray Phase Analysis Results of Solid Residues After Leaching of Overburden Waste at 50°C with Different Acids

Mineral |	**Chemical Formula**	**Content, wt.%**			
		Initial Sample	**Residue After H_3PO_4**	**Residue After HNO_3**	**Residue After H_2SO_4**
Calcite	$CaCO_3$	29.5	0.0	0.0	35.0
Kaolinite	$Al_2(Si_2O_5)(OH)_4$	19.8	0.0	0.0	0.0
Braunite	$(Ca_{0.01}Mg_{0.13}Mn_{0.86})(Fe_{0.03}Mn_{0.97})_6SiO_{12}$	14.6	31.0	30.0	15.0
Quartz	SiO_2	10.7	45.0	52.0	16.0
Hematite	Fe_2O_3	9.6	7.0	7.0	5.0
Barite	$BaSO_4$	8.5	8.0	7.0	5.0
Albite	$Na(AlSi_3O_8)$	3,9	5.0	4.0	3.0
Muscovite	$KAl_2(AlSi_3O_{10})(OH)_2$	1.8	0.0	0.0	0.0
Orthoclase	$KAlSi_3O_8$	1.7	0.0	0.0	0.0
Lipscombite	$Fe_{2.95}(POAl_4)_2(OH)_2$	0.0	4.0	0.0	0.0
Bassanite	$Ca(SO_4)(H_2O)_{0.5}$	0.0	0.0	0.0	17.0
Gypsum	$Ca(SO_4)(H_2O)_2$	0.0	0.0	0.0	4.0

Based on the results, the leachates obtained after overburden treatment with phosphoric and nitric acids at 50°C show particular promise for further investigation as potential liquid fertilizers. The salt composition of these productive solutions was analyzed using titrimetric methods (for Ca^{2+} and Mg^{2+}) and photocolorimetric methods (for Mn^{2+}, Fe^{3+}, and Al^{3+}). The results are presented in Table 5.

Table 5 – Salt Composition of Productive Leachates After Acid Leaching of Zhairem Overburden Waste at S:L = 1:25 and 50°C

Acid concentration (%)	**Content of Main Components, g/L**					
	Phosphoric Acid Leachate					
	$Ca(H_2PO_4)_2$	$Mg(H_2PO_4)_2$	$Mn(H_2PO_4)_2$	$Al(H_2PO_4)_3$	$Fe(H_2PO_4)_3$	$H_3PO_4^{сво}$
40	51.6	10.6	19.6	8.2	4.4	421.2
10	50.7	10.6	14.5	6.1	2.1	30.6
Nitric Acid Leachate						
	$Ca(NO_3)_2$	$Mg(NO_3)_2$	$Mn(NO_3)_2$	$Al(NO_3)_3$	$Fe(NO_3)_3$	$HNO_3^{своб}$
40	36.2	7.2	14.2	4.6	3.0	450.0
10	36.2	5.4	12.2	4.2	1.8	59.0

The results indicate that the use of 40% H_3PO_4 and HNO_3 as leaching agents leads to a high concentration of residual free acid in the productive leachates (Table 5), rendering them unsuitable for use as liquid fertilizers. Consequently, additional leaching experiments were conducted using 10% acid solutions under the same conditions (S:L = 1:25, temperature 50°C). It was found that the concentrations of key components such as $Ca(H_2PO_4)_2$ and $Ca(NO_3)_2$ remained nearly unchanged (Table 5), although a slight decrease in the concentrations of manganese-, aluminum-, and iron-containing compounds was observed.

At the same time, the content of free H_3PO_4 in the leachate was reduced by nearly an order of magnitude, while free HNO_3 decreased by a factor of 7.5 (Table 5).

By partially neutralizing the residual acidity with ammonia to a pH of 5.5–6.0, it is possible to obtain complex NP fertilizers enriched with the micronutrient manganese. Under these conditions, aluminum and iron precipitate as hydroxides, while calcium, magnesium, and manganese remain in solution, as the precipitation pH of their hydroxides lies in a more alkaline range.

Thus, the study demonstrates that one of the viable technological approaches for recycling overburden waste from the Zhairem mining and metallurgical complex is the development of a process for producing liquid complex fertilizers based on phosphoric or nitric acid leachates.

4. Conclusion

The acid leaching process of overburden waste from the Zhairem manganese ore deposit was investigated. It was found that the use of sulfuric acid reduces the solubility of the overburden to 28–38% due to the formation of poorly soluble calcium sulfate on the surface of solid particles, as confirmed by EDXRF and XRD analyses. This surface layer hinders further interaction with the acid reagent.

The solubility of the waste in 40% orthophosphoric and nitric acids was found to be comparable, ranging from 59.50 to 61.56% for H_3PO_4 and from 60.20 to 62.50% for HNO_3. However, these leachates contained a significant amount of residual free acid.

It was also established that during leaching with phosphoric acid, increasing the process temperature causes part of the readily soluble iron and manganese dihydrogen phosphates to transform into poorly soluble hydro-phosphates, which remain in the solid phase.

Leaching with 10% phosphoric and nitric acids at an S:L ratio of 1:25 and a temperature of 50°C produced leachates that can be recommended as a basis for the production of liquid phosphate or nitrate fertilizers enriched with the micronutrient manganese.

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ЖӘЙРЕМ КЕН ОРНЫНЫҢ ЖЫНЫС ҚАЛДЫҚТАРЫН ҚЫШҚЫЛМЕН СІЛТІЛЕУ ПРОЦЕСІН ЗЕРТТЕУ

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Түйіндеме. *Кіріспе.* Қазақстандағы марганец кен орындарының көпшілігі ашық әдіспен өндіріледі. Бұл айтарлықтай көлемде жыныс қалдықтарының үйінділерін түзеді, олар қайта өңделмей, қоршаған ортаға теріс әсер етеді. Аталған қалдықтарда әртүрлі құнды компоненттер болғандықтан, оларды бейорганикалық материалдар өндірісінде баламалы шикізат көзі ретінде пайдалануға болады. Алайда, Қазақстанда бұл қалдықтарды өндіріске енгізу тәжірибесі жоқ. *Осы жұмыстың мақсаты* — Жәйрем кен орнының жыныс қалдықтарын әртүрлі минералды қышқылдармен сілтілеу процесін зерттеу және алынған ерітінділерді микроэлементтермен байытылған сұйық минералды тыңайтқыштар ретінде пайдалану мүмкіндігін қарастыру. *Нәтижелер мен талқылау.* Жәйрем марганец кен орнының жыныс қалдықтарын әртүрлі минералды қышқылдармен сілтілеу процесі зерттелді. Сілтілеу процесі үшін ең қолайлы реагенттер — азот және ортофосфор қышқылдары екені анықталды. Бұл қышқылдардың 10%-дық ерітінділерімен, Қ:С=1:25 қатынаста және 50°C температурада сілтілеу нәтижесінде, өнімді ерітінділер алынады. Бұл ерітінділерді микроэлемент — марганецті қамтитын сұйық фосфорлы немесе азотты тыңайтқыштар өндірісінің негізі ретінде пайдалануға болады.

Түйін сөздер: марганец кендері, жер үсті қалдықтары, қышқылды сілтілеу, өнімді ерітінді

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ИССЛЕДОВАНИЕ ПРОЦЕССА КИСЛОТНОГО ВЫЩЕЛАЧИВАНИЯ ВСКРЫШНЫХ ПОРОД МЕСТОРОЖДЕНИЯ ЖАЙРЕМ

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Резюме. *Введение.* Большинство месторождений марганцевых руд Казахстана отрабатывают открытым способом. Это создает большие отвалы вскрышных отходов, которые не утилизируются и оказывают негативное влияние на окружающую среду. Так как эти отходы содержат различные ценные компоненты, то они могут быть использованы как альтернативный сырьевой источник в производстве различных неорганических материалов. Однако практика вовлечения отходов вскрыши в производство в Казахстане отсутствует. *Целью данной работы* является исследование процесса выщелачивания вскрышных отходов месторождения Жайрем различными минеральными кислотами и возможности использования полученных растворов в качестве жидких минеральных удобрений, обогащенных микроэлементами. *Результаты и обсуждение.* Исследован процесс выщелачивания вскрышных отходов месторождения марганцевых руд Жайрем различными минеральными кислотами. Показано, что оптимальными реагентами процесса выщелачивания являются азотная и ортофосфорная кислоты. Выявлено, что выщелачивание отходов вскрыши 10%-ными растворами этих кислот при Т:Ж=1:25 и температуре 50ºС приводит к образованию продуктивных растворов, которые можно рекомендовать к использованию в качестве основы для производства жидких фосфорных или азотных удобрений, содержащих микроэлемент марганец.

Ключевые слова: марганцевые руды, вскрышные отходы, кислотное выщелачивание, продуктивный раствор.

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