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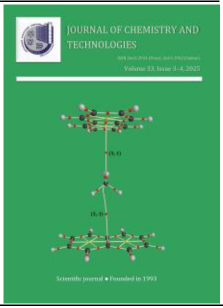

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A NEW APPROACH TO PROCESSING POLYMINERAL POTASSIUM ORE FROM THE CARPATHIAN REGION

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Abstract

Existing technologies for processing polymineral potash ores of the Carpathian region do not ensure the production of high-quality, highly concentrated fertilizers and are also characterized by a low degree of potassium extraction (less than 60 %). Shenitization of ore in dumps allows achieving a higher degree of conversion of sparingly soluble langbeinite and kieserite into readily soluble minerals but it is a long-term process. Therefore, it is proposed to convert sparingly soluble minerals into readily soluble ones from the residue after ore dissolution using reversible salt suspensions. Based on experimental studies, a new technology for the production of chlorine-free potash fertilizers has been developed which includes the conversion of pre-prepared ore with a reversible salt suspension. This technological method and grinding of the residue before conversion can increase the extraction of potassium into the product up to 82 %. The main products of the proposed technology are table salt, potash fertilizers, magnesium chloride (or bischofite). The residue, which contains insoluble components, in particular, polyhalite can be processed into a long-acting, chloride-free, granular potassium fertilizer with trace elements.

Keywords: Potash raw materials; processing methods; langbeinite; kainite; polymineral ore; sulfate potash fertilizers; production.

НОВИЙ ПІДХІД ДО ПЕРЕРОБЛЕННЯ ПОЛІМІНЕРАЛЬНОЇ КАЛІЄВОЇ РУДИ КАРПАТСЬКОГО РЕГІОНУ

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Анотація

Технології переробки полімінеральних калійних руд Карпатського регіону, які існували раніше, не забезпечували виробництво високоякісних, висококонцентрованих добрив, а також характеризувалися низьким ступенем вилучення калію (менше 60 %). Шенітизація руди на відвалах дозволяє досягти вищого ступеня перетворення важкорозчинних лангбейніту та кізериту в легкорозчинні мінерали, але це довготривалий процес. Тому в даній роботі пропонується перетворювати важкорозчинні мінерали із залишків на легкорозчинні після розчинення руди за допомогою оборотних сольових суспензій. На основі експериментальних досліджень розроблена нова технологія виробництва безхлорних калійних добрив, яка включає перетворення заздалегідь підготовленої руди за допомогою оборотної сольової суспензії. Цей технологічний метод та подрібнення залишків перед перетворенням можуть збільшити вилучення калію в продукт до 82 %. Основними продуктами запропонованої технології є кухонна сіль, калійні добрива, хлорид магнію (або бішофіт). Залишки, що містять нерозчинні компоненти, зокрема полігаліт, можуть бути перероблені на безхлоридні калійні добрива з мікроелементами пролонгованої дії.

Ключові слова: калійна сировина; методи переробки; лангбейніт; каїніт; полімінеральна руда; сульфатні калійні добрива; виробництво.

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Introduction

Recently, the demand for chloride-free potash fertilizers has increased in the world which include potassium sulfate, nitrate, phosphate, potassium magnesia, potassium-magnesium concentrate [1]. They improve the quality characteristics of grown products, contribute to increasing plant resistance to diseases, drought and frost and ensure the stability of fruits during storage and transportation.

The natural raw materials for the production of chloride-free potash fertilizers are polymineral ores containing langbeinite, kainite and kainite-langbeinite rocks, the reserves of which in the world are estimated at 8–10 %. [2; 3].

Ukraine has a unique Precarpathian deposit of polymineral ores which can serve as a raw material base for the production of high-quality chloride-free potash fertilizers (Fig. 1). The most well-known amnong the deposits that have been put into operation were Stebnytske (Lviv region) and Kalush-Holynske (Ivano-Frankivsk region) deposits. Boryslavske, Markovo-Rozsilnyanske, Nezhukhivske and Trostyanetske deposits deserve attention and are promising for processing [4].

Thanks to these deposits, Ukraine has the potential to produce a significant amount of chloride-free potash fertilizers, which are in demand in modern agriculture. Polymineral ores of the Carpathian Basin have a complex and unstable chemical composition with a wide range of minerals. The average mineral composition of polymineral ores of the Carpathian Basin is presented in Table 1.

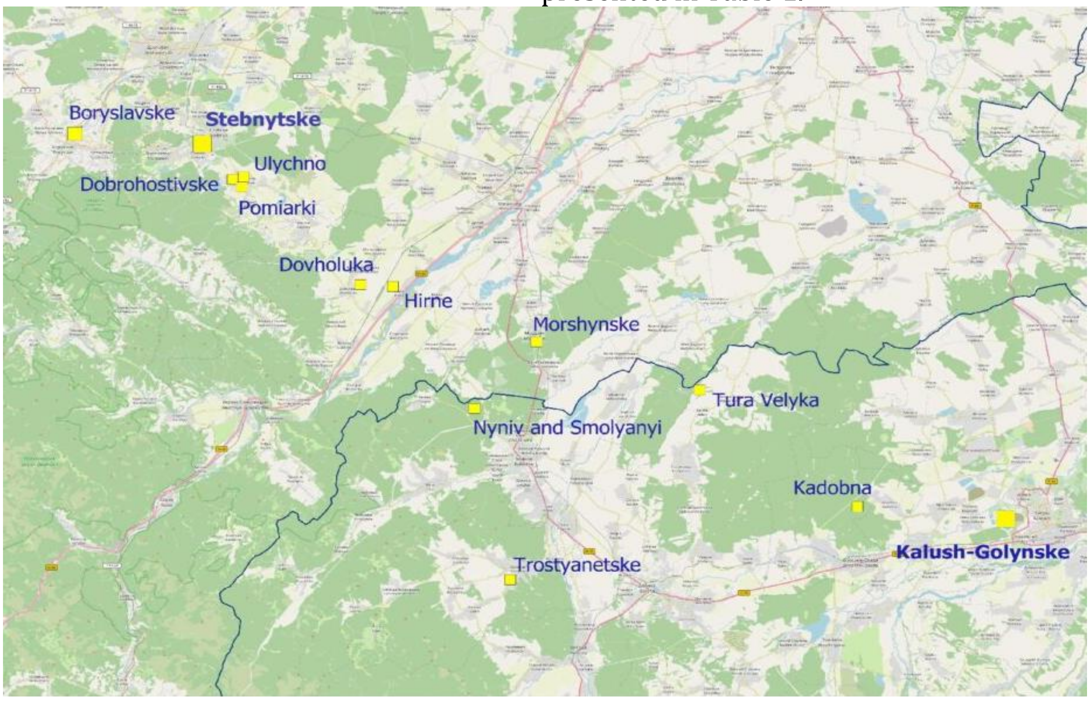

Fig. 1. Map of polymineral potassium ore deposits in the Carpathian region [2]

They can conventionally be divided into readily soluble minerals (halite, sylvite, carnallite, schoenite, leonite, kainite, bischofite, epsomite), sparingly soluble (langbeinite, kieserite) and insoluble ones (gypsum, anhydrite, polyhalite, syngenite).

Table 1

Mineral composition of polymineral potash ores of the Carpathian Basin (wt.%) [4]

Minerals	Name of the deposit									
	Kalush-Holynske				Stebnytske	Boryslavske	Markovo-Rosilnyanske		Nezhuhivske	Trostianetske
	Holinske	Kaluske	Dombrovo	Pilo			Rosilnyanske	Markivske		
Kainite	26.17	23.18	17.07	13.41	15.98	21.99	18.56	9.21	26.97	2.59
Langbeinite	3.20	2.45	16.04	5.53	10.95	11.86	13.41	24.20	6.99	24.16
Sylvine	8.80	9.01	4.36	9.76	4.44	2.18	3.70	2.58	5.14	3.44
Polyhalite	4.40	4.86	7.24	5.67	6.33	10.82	7.74	8.40	4.16	9.50
Halite	38.60	39.45	27.67	35.31	35.78	22.76	28.71	29.63	33.47	30.10
Schoenite	0.80	0.52	2.39	0.85	0.30	0.67	0.68	2.33	0.01	2.83

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Continuation of Table 1

Leonite	-	-	-	-	-	1.23	0.20	0.79	0.22	-
Kieserite	3.10	2.84	6.82	7.08	4.00	3.34	7.12	2.70	10.72	6.73
Epsomite	0.20	0.02	0.42	-	3.50	1.36	2.77	4.51	-	2.63
Carnallite	1.59	0.76	0.17	0.43	0.20	0.07	-	-	0.11	-
Glaserite	-	-	0.21	-	-	0.42	-	-	-	-
Astrakhanite	-	-	0.42	-	-	0.14	-	-	-	-
Thenardite	-	-	0.54	-	-	-	-	-	-	-
Mirabilite	-	-	0.10	-	-	-	-	-	-	-
Anhydrite	1.20	1.73	0.34	2.14	-	0.87	0.68	0.83	1.20	0.30
Gypsum	0.02	-	0.09	-	-	0.02	-	-	-	-
Leveite	-	-	-	-	-	0.02	-	-	-	-
Clay impurities	10.60	13.09	14.84	17.34	16.84	18.97	14.00	13.71	8.97	15.45

Given the presence of such a large numerous of minerals, as well as a large amount of insoluble residue (up to 20 %), the processing of polymineral ores of the Carpathian region is a complex and multi-stage process.

Technological processes for processing polymineral ores into potash fertilizers are based on data on the solubility of salts in the five-component system Na+, K+, Mg^{2+}||||Cl^{-}, SO_4^{2-}, H_2O. Analysis of the solubility polytherms of salts in this system shows that it is practically impossible to separate useful potassium-magnesium minerals from halite with an acceptable technological yield by one-stage dissolution of the ore in water or salt solutions.

Currently, three main methods have been developed for processing polymineral potassium ore of this composition: flotation, halurgical and combined [5–10].

The flotation method requires expensive flotation reagents and is based on different wettability of different minerals. The disadvantage of the flotation method is the relatively low selectivity of the process due to the need to separate two or more potassium minerals of different mineralogical and chemical nature by flotation, the presence of contaminating impurities of chlorides and clay minerals in the final product; the low degree of extraction of potassium and magnesium from the ore into the concentrate (less than 50 %). The method was implemented at the Stebnytsky Potash Plant and partially at Kalush Plant where it proved ineffective due to the high content of insoluble residue in local ore, secondary flotation and low quality of the obtained sodium chloride.

The halurgical method, which is based on different degrees and rates of dissolution of individual minerals at various temperatures, followed by crystallization of products from saturated and evaporated solutions, was implemented at the Kalush Potash Plant. The drawback of the halurgical method is the presence of sodium chloride as an impurity at all technological stages, which requires additional evaporation of the schoenite solution; low extraction of potassium and other useful components from the ore, the multistage nature and energy consumption technology.

The use of mineral acids for dissolving sparingly soluble minerals has not been applied in industry due to the high cost and complexity of manufacturing technological equipment. The use of organic solvents is also ineffective in large-scale production due to significant solvent losses and environmental risks due to processing. The electrostatic separation method is known and which has been applied and is used in Germany. The essence of the method lies in the ability of particles of different minerals to accumulate electric charges of varying magnitude. The method is effective only for ores with a small number of components and a low content of clay impurities [9]. The gravity method has also not found application due to the small difference in the density of the salts that need to be separated.

The halurgical-flotation (combined) scheme for processing polymineral potassium ore implemented at the Kalush Potash Plant turned out to be ineffective due to the complexity of the halurgical processing of the flotation-enriched insoluble residues together with the products of the halurgical cycle caused by secondary flotation of crystallized products. This led to the suspension of the flotation stage, resulting in the accumulation of approximately 25 million m^3 of halite–langbeinite waste in the tailings storage facilities [11; 12].

The accumulation of vast amounts of waste is not only a loss of valuable raw materials and degradation of agricultural lands but also a serious environmental threat. Therefore, it is necessary to implement new technologies that will allow not only complete processing of freshly mined ore but also efficiently utilize the already accumulated waste. Such an approach will ensure waste-free production [13].

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A possible way to increase the extraction of potassium from polymineral ores of the Carpathian region is to convert langbeinite and kieserite into a readily soluble form - schoenite and epsomite by storing the moistened ore in heaps followed by ore hydration. [14].

Materials and Methods

Analysis of the processes of conversion of chloride and sulfate minerals and crystallization of salts from saturated salt solutions was carried out using equilibrium diagrams of the system Na^+, K^+, Mg^{2+}||||Cl^-, SO_4^{2-}, H_2O. The quantitative compositions of liquid and solid phases in the experiments were determined by standard methods of chemical analysis, in particular K^+ – by gravimetric tetraphenylborate, Mg^{2+} and Ca^{2+} – by complexometric, Cl^- – by mercurometric, SO_4^{2-} – by gravimetric methods. The concentration of Na^+ was determined by the difference in equivalents of anions and cations. According to the results of chemical analyses, the salt and mineral compositions of the systems were calculated using methods known in halurgy. Statistical processing of experimental data and calculations of material balances were carried out using the computer program Microsoft Excel.

The reliability of the results was ensured by using standardized chemical analysis methods for each ion and confirmed by calculating the material balances of the processes.

The results of studies on the influence of technological parameters on the conversion of poorly soluble minerals into easily soluble ones and studies on the kinetics of conversion processes are presented in [7; 14; 15].

Experiments have shown that the preliminary treatment of the ore to convert potassium langbeinite into a soluble state takes a certain amount of time and can last up to 25–30 days depending on the degree of grinding of the initial ore [12].

The hydration of langbeinite is an exothermic process as a result of which the temperature of the ore mass increases by 2–3 °C.

In order to increase the soluble part of the ore in the processing process, the polymineral langbeinite-containing ore is subjected to grinding, classification and hydration.

The following reactions occur during the hydration of polymineral ore:

1. Hydration reaction of langbeinite with the formation of schoenite and epsomite:

$$K_2SO_4\times 2MgSO_4+13H_2O \rightarrow K_2SO_4\times MgSO_4\times 6H_2O+MgSO_4\times 7H_2O \quad (1.1)$$

2. The conversion reaction of epsomite and sylvine to form schoenite:

$$2KCl+2MgSO_4\times 7H_2O \rightarrow K_2SO_4\times MgSO_4\times 6H_2O+MgCl_2+8H_2O \quad (1.2)$$

3. The conversion reaction of kieserite and sylvine to form schoenite:

$$2KCl+2MgSO_4\times H_2O \rightarrow K_2SO_4\times MgSO_4\times 6H_2O+MgCl_2 \quad (1.3)$$

$$MgSO_4\times H_2O+6H_2O \rightarrow MgSO_4\times 7H_2O \quad (1.4)$$

4. Kainite hydration reaction to form schoenite:

$$KCl\times MgSO_4\times 3H_2O \rightarrow K_2SO_4\times MgSO_4\times 6H_2O+MgCl_2 \quad (1.5)$$

The reaction products are readily soluble salts of schoenite and epsomite, the solubility of which is 200 times higher than that of langbeinite [14]. The project was implemented at the Stebnytskyi Potash Plant. The ore was prepared in an open warehouse No. 1, equipped with grab loaders for shoveling ore during the first 7 days. The shenitization process was completed in 3 closed shenitization warehouses according to the following scheme. The pre-prepared ore was classified by class 2 mm. Class +2…–5 mm, as the largest, must pass through three warehouses, so it was moistened in a mixing machine and was in warehouse No. 2 for 13–15 days. After the end of holding term, the ore was reloaded to the second warehouse by scraper cranes and conveyors. At the same time, it was mixed with half of the fine ore (fraction -2 mm), moistened in a mixing machine and stored in warehouse No. 3. After 8-10 days, the ore was transferred to the third warehouse No. 4 in the same way where it was mixed with the second half of the fine fraction and moistened in a mixing machine. In storage No. 4, the ore remained for 6–7 days before being sent for dissolution. As can be seen, the shenitization process is long-term, requires special technological equipment and significant capital investments in the construction of covered warehouses. Since the projected indicators were not confirmed, in particular, an increase in potassium extraction to 70–80 % was not achieved, the plant was not reconstructed, and the project was closed [12].

The issue of increasing the extraction of potassium into the product from polymineral ores of the Carpathian region remains unresolved currently.

In our opinion, the process of transferring potassium-containing minerals into the liquid phase should be carried out selectively and the bulk of halite should be removed to the residue at the

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beginning of the process. To ensure the complete utilization of raw materials, it is necessary first to dissolve all readily soluble minerals, while only the residue after preliminary dissolution should be subjected to conversion. We have proposed carrying out wet grinding of the ore in rod mills and removing the clay-salt residue after preliminary dissolution, which will significantly reduce the time the ore stays in the heaps and thereby accelerate the conversion process.

NIOCHIM State Enterprise has proposed to convert previously prepared ore of the -1 mm classification into readily soluble kainite. The conversion is carried out using carnallite and carnallite solution from the stage of evaporation of excess schoenite solution.

Compared to shenitization, this process has its advantages:

- prevents the formation of crystal hydrates, which crystallize on the surface during shenitization and bind it into a monolithic mass, which requires periodic mixing;
- it is possible to form piles immediately without the need to mix the ore;
- kainitized ore practically does not cake and remains porous and dry.

The proposed method allows processing not the entire ore but only the residue after dissolving readily soluble minerals. This will lead to a reduction in the reactive mass by half and significantly improve the conversion conditions.

The conversion of langbeinite to kainite occurs according to the overall reaction:

$$K_2SO_4 \times 2MgSO_4 + 7H_2O + MgCl_2 \rightarrow 2(KCl \times MgSO_4 \times 3H_2O) + MgSO_4 \times H_2O \quad (1.6)$$

Also in the conversion process, kainite is synthesized from sylvine and kieserite (or epsomite):

$$MgSO_4 \times H_2O + KCl + 2H_2O \rightarrow KCl \times MgSO_4 \times 3H_2O \quad (1.7)$$

Under certain conditions, the conversion of langbeinite to kainite will occur through its interaction with carnallite:

$$KCl \times MgCl_2 \times 6H_2O + K_2SO_4 \times 2MgSO_4 \rightarrow 3(KCl \times MgSO_4 \times 3H_2O) \quad (1.8)$$

As a result of the above reactions (1.6–1.8), the poorly soluble langbeinite ore can be converted into a readily soluble one. The langbeinite hydration reaction 1.1 belongs to the class of topochemical reactions, the rate of which depends on the available reaction surface [15]. Therefore, multiple wetting solution feeds are required to ensure access of moisture to the reaction surface. The amount of salt solution required to achieve a conversion degree of langbeinite-containing ore of 80% depends on the mineral composition of the initial ore and can vary within 12–18 % by weight in terms of pure water. Reducing the supply of the wetting solution leads to a decrease in the degree of langbeinite conversion and increasing it leads to caking of the converted ore (crystals are formed that bind the ore particles into a monolithic mass that must be constantly stirred). Studies have shown [15-18] that the completeness and speed of conversion are influenced by the following factors: the degree of ore grinding, the height of storage in the heap, time, temperature and amount of wetting liquid, the method of wetting.

The maximum impact on the conversion is the specific consumption of the salt solution. As its quantity increases, the degree of conversion rises at the initial stage and hardly changes once a 30 % excess is reached. However, from a practical point of view, it is important to obtain a moistened mass in the formed piles which can be conveniently stored and processed. Taking this into account, it is desirable to add the solution in an amount of 116–120 % of the stoichiometric requiremnt, and as mentioned above, in several doses. Readily soluble salts and impurities of clay particles negatively affect the degree of conversion. The presence of clay impurities adversely impacts the transformation of langbeinite into kainite. An increase in the clay content from 5 % to 20 % leads to a critical drop in the degree of conversion (from 46.9 % to 0.31 %). This occurs because clay forms a barrier that blocks the reagents' access to the langbeinite surface, slowing the chemical reaction. Therefore, to increase the conversion efficiency, it is necessary to pre-clean the ore from clay and soluble salts. Considering the significant difference in the mineral composition of unconverted polymineral ore and converted ore (Table 2), the technological scheme of processing also needs to be changed.

Table 2

Average mineral composition of converted and unconverted ores [14]

Conversion time, hours	Mineral composition of the solid phase, wt. %						
	Anhydrite	Polyhalite	Langbeinite	Kainite	Carnallite	Halite	Sylvine
0	0.45	3.97	44.74	2.48	15.28	21.16	1.64
96	0.63	5.60	6.73	39.84	1.68	25.78	8.18

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Results and Discussion

Based on the conducted research and critical analysis of the technologies available in the Carpathian region [19-21], a technological scheme for the processing of polymineral potassium ores using the conversion process was developed, which is presented in Fig. 2.

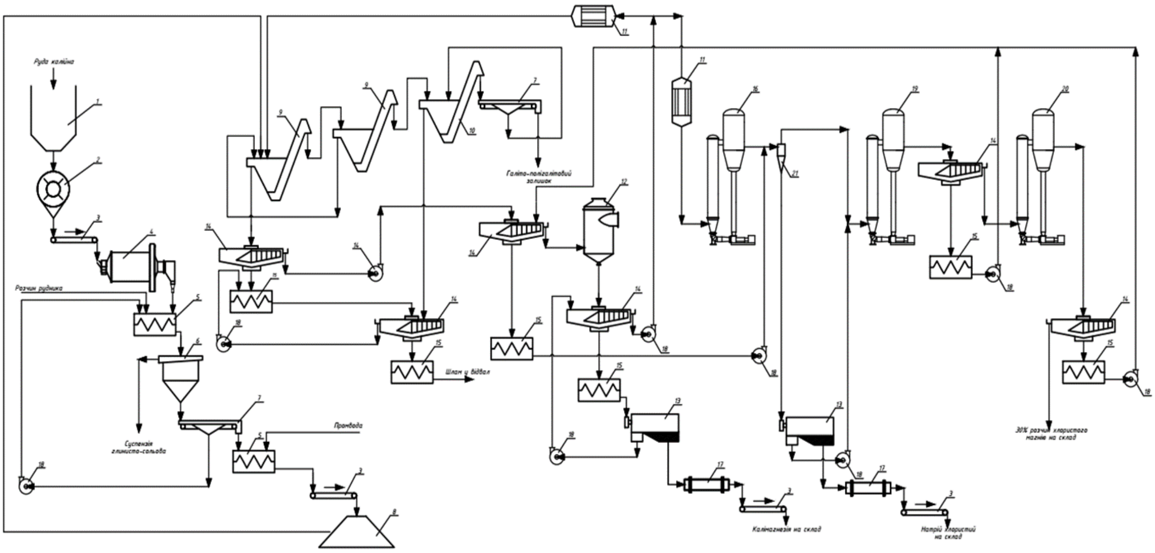

Fig. 2. Technological scheme of processing of polymineral potassium ore of the Carpathian region: 1 – bunker; 2 – crusher; 3 – belt conveyor; 4 – rod mill; 5 – mixer; 6 – Brandes settling tank; 7 – belt vacuum filter; 8 – ore heaps; 9 – solvents with an elevator; 10 – recuperator; 11 – shell-and-tube heat exchanger; 12 - crystallizer; 13 – filter centrifuge; 14 - radial settling tank; 15 – tank with agitator; 16 – 4-case evaporation plant; 17 – drying drum; 18 – centrifugal pump; 19 – 3-case evaporation plant; 20 – 2-case evaporation plant; 21 – hydrocyclone

The potash ore crushed to a size of 5 mm is fed by a conveyor system to a wet grinding rod mill, where it is ground to a size of 0.5–1.0 mm using a saline solution from the ore dissolution stage.

Clay impurities are washed out of the potash ore fractions in a frame mixer using saline solutions, after which the resulting suspension is fed to Brandes settling tanks. After separation of clay impurities, the salt precipitate is fed to belt filters for filtration, from where the resulting precipitate is stored in banks for conversion. Before storage, the precipitate is mixed with potassium-magnesium sulfate-chloride salts, which are formed at the stage of evaporation of excess shenite solution.

For proper operation of the conversion unit, all storages facilities are filled to their working volumes to ensure uninterrupted conversion processes and uniformity of the ore.

After the time allotted for conversion (4 days), the ore is fed for dissolution using a scraper crane and a conveyor system. The process of dissolving the converted ore takes place in two consecutive screw dissolvers in a countercurrent scheme. The movement of the mixture along each apparatus and its mixing is carried out using a screw mixer. The required temperature regime is maintained by preheating the dissolving solution in a heater using hot steam at a pressure of 0.3 MPa. The insoluble residue from the recuperator is fed for filtration, after which the filtered sediment (langbeinite-polyhalite concentrate) can be used as a potassium-magnesium fertilizer of prolonged action. The dissolving solution is a mixture of schoenite mother liquor and a washing solution from the stage of washing clay-salt sludge. The composition of the dissolving solution must be saturated with sodium ions and unsaturated with potassium ions. The halite residue and clay sludge formed after dissolution can be used to fill mine voids while the clarified solution is fed to the crystallization stage of schoenite.

The thickened schoenite suspension after the crystallizers is fed for dehydration and drying and is shipped to the warehouse as a finished product. [22] The separated schoenite mother liquor after the thickener is used to dissolve the converted ore.

The discharge of the clarified solution thickener is sequentially sent to vacuum evaporation units for evaporation of excess schoenite solution to obtain the finished product: sodium chloride and bischofite (or 32 % magnesium chloride solution, which can be used to obtain metallic magnesium, magnesia binders or magnesium oxide).

Using the results of the laboratory studies, a consolidated material balance was calculated (Table 3).

The proposed method of processing polymineral potassium ore is more efficient than those considered in the works [23-25].

Table 3

Summary material balance of polymineral ore processing in the Carpathian region

Component name	Mass, т/год	Chemical composition, mass %							
		K^+	Mg^{2+}	Ca^{2+}	Na^+	Cl^-	SO_4^{2-}	H_2O	H.З.
income in the material balance									
Ore	133.50	11.60	7.26	1.25	15.50	29.18	38.77	11.41	18.52
Brine solution	95.76	2.05	2.00	0.00	5.18	12.98	3.68	69.88	-
Water	64.35	-	-	-	-	-	-	74.64	-
Total	293.61	13.65	9.26	1.25	20.68	42.16	42.45	81.29	18.52
expenses in the material balance									
Shoenite	40.60	9.16	2.68	0.00	0.33	1.06	21.19	6.18	-
Sodium chloride	9.97	0.00	0.00	0.00	3.92	6.05	0.00	0.00	-
Magnesium chloride solution	28.94	0.52	1.97	0.14	0.08	5.57	0.85	19.80	-
Halite residue	57.10	2.27	2.80	0.72	10.38	13.76	18.61	6.98	1.58
Clay-salt mud (solid phase)	31.35	0.49	0.39	0.39	2.89	6.88	0.23	3.14	16.94
Clay-salt mud (liquid phase)	61.31	1.21	1.42	0.00	3.08	8.84	1.57	45.19	-
Water evaporated	64.34	-	-	-	-	-	-	64.34	-
Total	293.61	13.65	9.26	1.25	20.68	42.16	42.45	81.29	18.52

Conclusions

1. Analysis of literary sources and practical research confirm that the most effective way to process polymetallic potash ore is to convert poorly soluble minerals into easily soluble ones. In this case, the pre-prepared ore (size less than 0.5 mm) was treated with water or salt solution in bunkers for 5 days.

2. Experimental studies have confirmed that the conversion of polymetallic potash ore allows the degree of extraction of useful components (potassium and magnesium ions) from the ore into the product to be increased to 90 %, compared to 50–60 % using the existing method.

3. The most intensive conversion of poorly soluble ore minerals occurred in mixers at a material moisture content of 120 % of stoichiometry, at a temperature of 25–30 °C and a duration of 300 min.

4. The optimal temperature regime for dissolving the processed ore was determined to be a process duration of 30 min and a phase ratio of T : P = 1.5 in the first solvent at 65–75 °C; in the second solvent at 75–85 °C.

5. A basic technological scheme for processing polymetallic potash ore was developed, and hardware and technological solutions were proposed for obtaining three products: table salt, potash fertilizer, and magnesium chloride. New technological solutions allow stabilizing the process of obtaining potassium-magnesium fertilizers from polymetallic potash raw materials with variable mineral composition.

To convert poorly soluble ore minerals into easily soluble ones, it is recommended to use reversible salt solutions formed during the halurgical processing of ore, or concentrated solutions from the Dombrovsky quarry and tailings ponds. This will ensure more efficient use of raw materials and reduce the negative impact on the environment.

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