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Profitable Processing of Lithium-Bearing Low-Grade Ore for Obtaining Lithium Compounds and Cement

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Abstract

To enhance the efficiency of the production of lithium compounds from unconcentrated spodumene ore, the possibility of its integrated processing using the calcareous procedure of high-temperature treatment is demonstrated.

Key words: spodumene ore, treatment, calcareous method, leaching, aluminate solution, decomposition, lithium compounds, sludge, cement, economical efficiency

INTRODUCTION

To obtain lithium compounds, spodumene ore had been used in Russia till the 90es of the past century. After concentrating, lithium concentrate was obtained and processed according to a classical calcareous technology [1]. However, with the discovery of lithium-bearing brine deposits in the USA, Chile, Argentina and other countries and establishment of industrial enterprises on obtaining lithium carbonate, processing of aluminosilicate lithium raw material became economically non-profitable in comparison with the technology of obtaining cheap lithium carbonate from brines and salars [1, 2]. Lithium plants of Russia passed to the imported raw material – cheap lithium carbonate, however, soon the cost of Li_2CO_3 sharply increased, and the problem concerning the renewal of the home raw material basis became urgent. On the one hand, the need in lithium products can be provided by the own brine deposits [2], but it is necessary to develop them. On the other hand, in Russia there are many ore deposits and dumps from concentrating rare metal raw material; they are low-grade with respect to lithium content [3] but they can be used if there were cheap methods of processing them.

With the establishment of the Siberian Branch of the Russian Academy of Sciences, researchers from the Institute of Physicochemical Foundations of Mineral Raw Processing (at present Institute of Solid State Chemistry and Mechanochemistry (ISSCM), SB RAS) and Institute of Silicate Chemistry, AS USSR, carried out joint investigations on obtaining lithium products and cement from unconcentrated spodumene ore of the Zavitinskoye (Chita Region) and Polmostundrovkoye (Murmansk Region) deposits. Investigations were supervised by I. S. Lileev, winner of the State Prize of the USSR, creator of the calcareous technology of treating lithium concentrates (Li_2O content 4–6 %) which was taken as the basis for the first lithium plant in the USSR – the Krasnoyarsk Chemical and Metallurgical Plant JSC (KCMP).

To simplify the calcareous method of spodumene ore treatment, avoiding the stage of its concentrating, the following changes were introduced into the process: 1) the consumption of chalkstone in the mixture was decreased (from 3 to 2 moles of CaO per 1 mol of SiO_2) for its agglomeration with the product of treatment of lithium raw material; 2) soda was added into the mixture in order to bind sodium and potassium into soluble aluminates; 3) evaporation of the solutions of alkaline metal (Li, Na, K) hydroxides was replaced by a simpler operation involving precipitation of double aluminium–lithium hydroxide $LiOH \cdot 2Al(OH)_3 \cdot 5H_2O$ (DHAL-OH) from aluminate solutions containing lithium hydroxide; 4) the possibility to obtain cement from the waste material of lithium production (sludge) was provided.

These changes are directed, on the one hand, at a decrease in material fluxes at the operation of obtaining lithium products and complex extraction of lithium and aluminium; on the other hand, they provide the possibility of subsequent use of the wastes from lithium production to obtain cement. Results of the investigations, scaled and experimental-industrial tests of the technology, as well as the promising directions of the use of double hydroxide of aluminium and lithium were generalized in [4]. The results of further development of the technology, mainly dealing with the directions of the use of double lithium and aluminium compounds, and the studies of the new aspects of the technology development for the purpose of improving the method of processing unconcentrated spodumene ore, were published in [5].

At present, the technology of processing aluminosilicate raw material with the low Li_2O content can become necessary. In connection with an increase in the intensity of building during the recent years, the problem of obtaining cement has become urgent. As the authors of [4, 5] demonstrated, the wastes from lithium works containing the major components of cement clinker (dicalcium silicate, calcium aluminates and aluminoferrites), after the corresponding correction, may serve as the efficient raw material source for obtaining both slurry cement and Portland cement [4–6].

The goal of the present study was to develop and optimize the calcareous-soda technology in the two-stage mode and determination of the efficiency of its realization through the establishment of the joint production of lithium ($LiOH \cdot H_2O$), aluminium (anhydrous lithium aluminates) products and cement using unconcentrated spodumene ore from the Zavitinskoye deposit.

EXPERIMENTAL

To determine the chemical composition of solid and liquid phases, we used atomic absorption spectroscopy [8], chelatometry, and gravimetric analysis [9]. Carbon dioxide was determined using the gas volumetric method of analysis [10]. Phase and mineralogical composition of cakes and sludge, as well as the components of the cement clinker were determined by means of X-ray phase and crystal optical methods using a DRON-4 diffractometer and microscope PLAM-L-211, respectively.

In optimizing the two-stage scheme of processing unconcentrated spodumene raw material, we used mine-run ore from the Zavitinskoye deposit (Table 1).

TABLE 1

Averaged data on the chemical composition of ore and products of its processing

Material	Mass fraction, %							
	Li_2O	Na_2O	K_2O	CaO	Al_2O_3	Fe_2O_3	SiO_2	$H_2O + CO_2$
Mine-run ore of the Zavitinskoye deposit	0.95	2.05	1.11	0.3	16.85	2.6	73.3	2.8
Cake after agglomeration at 1200 °C	0.56	2.65	0.9	53.4	7.75	1.3	30.6	–
Sludge after leaching of cake with NaOH solution	0.08	n/d	n/d	49.4	4.36	1.1	28.8	25.0

Note. n/d – not determined.

RESULTS AND DISCUSSION

Agglomeration of spodumene ore with chalkstone and soda was carried out at the molar ratio in the mixture $CaO : SiO_2 = 2 : 1$, $(\Sigma Na_2O + K_2O) : Al_2O_3 = 1.0$ at a temperature of (1200±20) °C. After cooling the cakes inclined to self-dispersal [4], the powder was treated with the solutions of sodium hydroxide (25 g/L). The time of lithium leaching fromt eh cake into solution was usually 1.5–2.0 h at a temperature of 75–90 °C.

After alkaline treatment, the solutions are characterized by the following composition, g/L: Li_2O 0.94 (LiOH 1.5), Al_2O_3 8.24, Na_2O 21.8.

The obtained aluminate solutions were subjected to decomposition using carbon dioxide [11]. Aluminate ions $Al_2(OH)_7^-$ interact with lithium ions and form within a short time (τ = 1–2 h) carbonate-containing form of double aluminium–lithium hydroxide (DHAL-CO_3) according to reaction

$$2Al_2(OH)_7^- + 2Li^+ + CO_2 + mH_2O \rightarrow Li_2CO_3 \cdot 4Al(OH)_3 \cdot mH_2O \quad (m = 9–13)$$

Lithium precipitation from aluminate solutions can be performed also by mixing them for 24–30 h with seeds and formation of DHAL-OH [12]. The obtained compound absorbs CO_2 from the air and is transformed into the carbonate form [10]. In this situation, Li_2O content in DHAL-CO_3 and DHAL-OH and the properties of the compounds obtained are close to each other [10–12].

The use of carbonization to precipitate lithium allows one to accelerate the process of formation of DHAL-CO_3 and improves the rheological characteristics of the precipitate that is well washed with water almost without losses of lithium during washing. Carbonization of aluminate solutions can be carried out with the help of exhaust gas from agglomeration furnaces with carbon dioxide content 10–15 %.

The obtained product has the following chemical composition, mass %: Li_2O 5.8, Al_2O_3 48.8, $(CO_2 + H_2O)$ 45.4; molar ratio $Al_2O_3 : Li_2O = 2 : 6$.

Lithium hydroxide monohydrate was obtained by caustification of DHAL-CO_3 with limewater [13] according to reaction

$$Li_2CO_3 \cdot 4Al(OH)_3 \cdot 11H_2O + 6Ca(OH)_2 = 2LiOH + 2[3CaO \cdot Al_2O_3 \cdot 6H_2O] + CO_2 + 10H_2O$$

The process of $3CaO \cdot Al_2O_3 \cdot 6H_2O$ formation in alkaline media was described previously by the authors of [14]. After washing and separation of the precipitate, the solution of LiOH was evaporated to decrease its volume by a factor of 2–3 to crystallize $LiOH \cdot H_2O$.

In more recent studies [2], the methods of obtaining lithium hydroxide monohydrate without the formation of voluminous precipitates $3CaO \cdot Al_2O_3 \cdot 6H_2O$ were developed.

Salt-acid treatment of DHAL-CO_3 allows one to perform separate obtaining of lithium salts and $Al(OH)_3$. By calcination of $Al(OH)_3$, lithium-containing alumina is formed; it contains Li_2O 0.2–0.3 %. Any lithium salt – LiCl, LiF, Li_2CO_3 – can be obtained from lithium-containing solutions using known methods [1, 2].

On the basis of investigations, a method of obtaining lithium carbonate from DHAL-CO_3 was developed (Fig. 1) [5]. Using membrane electrolysis, lithium hydroxide monohydrate was obtained through conversion LiCl → LiOH and Li_2CO_3 → LiOH. Electrochemical conversion allows one to obtain high-pure lithium hydroxide monohydrate efficiently [2, 5].

Another practically important product that can be obtained from DHAL-CO_2 is lithium concentrate, which is a mixture of anhydrous lithium aluminate and Al_2O_3 formed during calcinations of DHAL-CO_3 at T = 800 °C:

$$2[Li_2CO_3 \cdot (4 + x)Al(OH)_3 \cdot mH_2O] \rightarrow Li_2O \cdot Al_2O_3 + Li_2O \cdot 3Al_2O_3 + xAl_2O_3 + pH_2O \quad (x = 1–3)$$

During decomposition of aluminate solutions by means of carbonization, $Al(OH)_3$ is also coprecipitated [11]. Because of this, after calcinations of the mixture of indicated compounds we observe the formation of well crystallized lithium monoaluminate (this may be metastable $Li_2O \cdot 3Al_2O_3$) with aluminium oxide. The calcined product is lithium concentrate with Li_2O content 10–12 %.

Lithium concentrate, similarly to lithium-containing alumina, can be used to intensify electrolysis of aluminium, in the production of glass and ceramics [4], and also in obtaining materials for novel technologies, for example for synthesis of catalysts, conducting materials *etc.*) [5].

During cake leaching and separation of the aluminate solution containing LiOH, sludge is formed in the amounts exceeding those of the

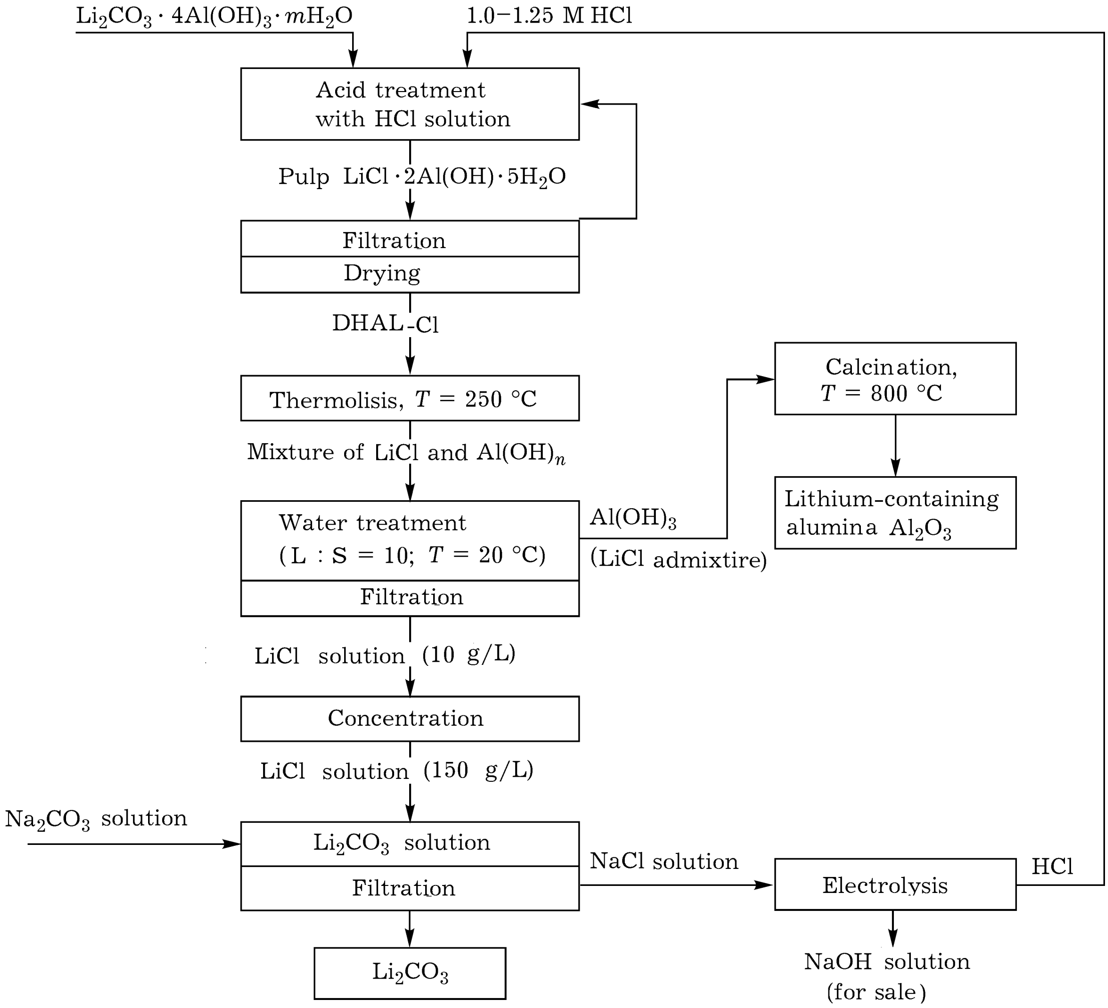

Fig. 1. Scheme of the production of lithium carbonate and lithium-containing alumina from DHAL-CO_3.

initial ore by a factor of about two and a half. In this connection, the problem of its utilization arises.

One of the methods of utilization of sludge formed during lithium processing (see Table 1) is to make sludge-cement from it [15]. For this purpose, it is necessary to remove moisture and bound water from sludge preliminarily by thermal treatment of the material at a temperature of 300–600 °C. Sludge binders are obtained by joint milling of dried sludge and lime. Addition of lime (CaO) enhances binding properties and promotes the formation of highly basic silicates and aluminates of calcium. Calcareous-sludge cement slowly sets and slowly hardens forming the cement stone with high strength (90–110 kg/cm^2) [15].

Wastes from lithium production can also be used for the production of Portland cement because its main characteristics, namely the silicate module ($n = SiO_2 : (Al_2O_3 + Fe_2O_3)$) and alumina module ($p = Al_2O_3 : Fe_2O_3$) correspond to the conditions of the formation of cement clinker [15].

Another condition for its formation may be the coefficient of mixture saturation with lime (CS); addition of lime into the sludge is to be made taking into account the conditions of the formation of tricalcium aluminate [6].

This condition is realized with the two-stage preparation of cement from the wastes of lithium production. The first stage includes lime and soda dosing for the formation of the aluminates of alkaline metals (Li, Na, K) and $2CaO \cdot SiO_2$, which allows simultaneous extraction of lithium and aluminium. At the second stage, lime dosing for the formation of $3CaO \cdot SiO_2$ is performed.

During the production of Portland cement from the wastes of lithium production, a two-component mixture (sludge + chalkstone) is used without correction. The fraction of the added chalkstone is 15–25 % of the sludge mass. Agglomeration temperature increases from 1300 to 1400 °C; the degree of CaO assimilation during annealing reaches 99.5 %, CS = 0.95.

According to the data of X-ray phase and crystal optical analyses, Portland cement clinker obtained on the basis of the sludge of the ore from Zavitinskoye deposit, during annealing contains the mixture $3CaO \cdot SiO_2$ and $3CaO \cdot Al_2O_3$, calcium aluminates ($nCaO \cdot Al_2O_3$) and a small amount of lithium aluminate (according to the data of chemical analysis, Li_2O

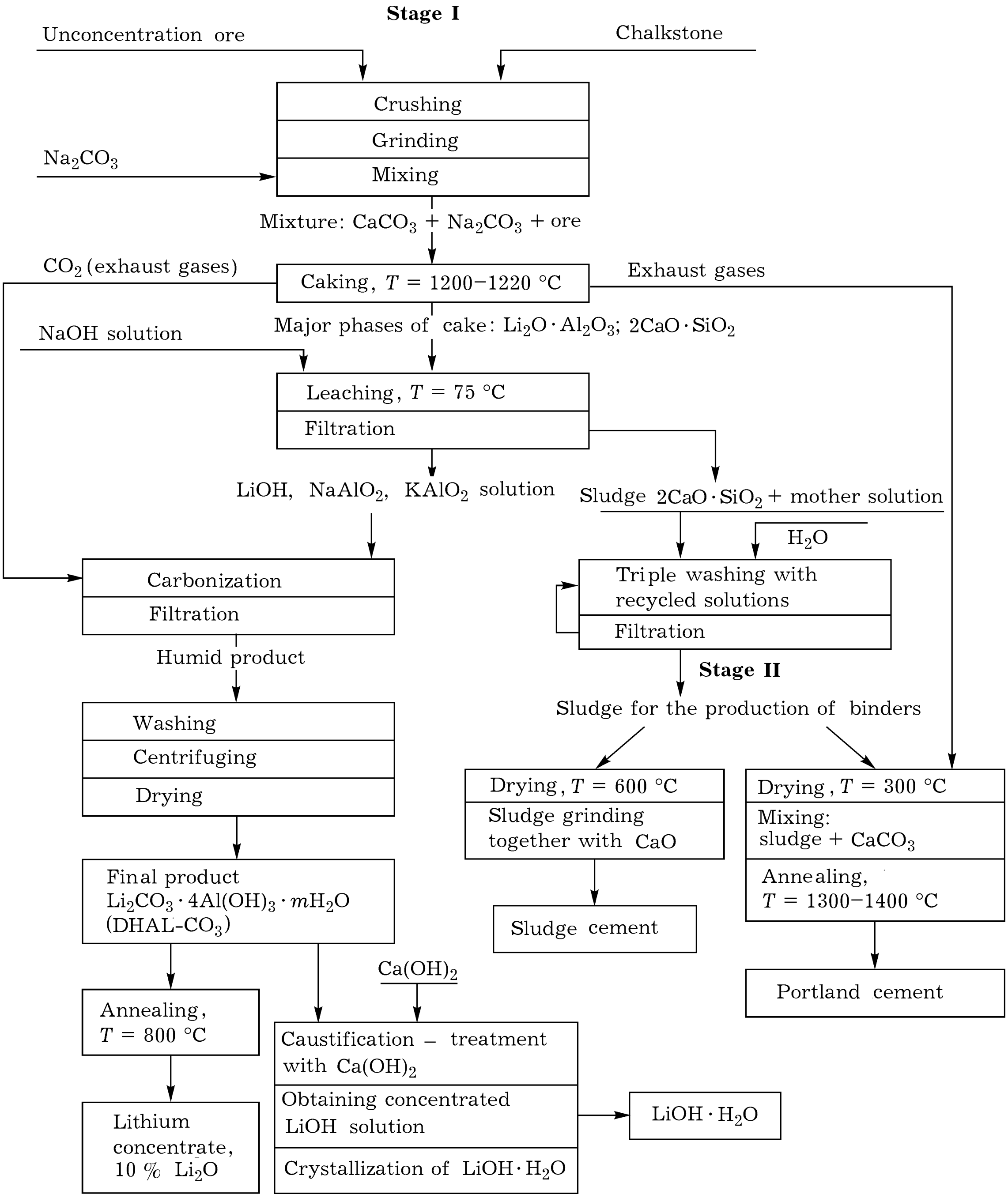

Fig. 2. Technological scheme of the integrated processing of unconcentrated spodumene ore leading to lithium hydroxide monohydrate, lithium concentrate and cement of different kinds (for simplicity of the scheme, obtaining $LiOH \cdot H_2O$ according to the procedure described in [13] is shown).

TABLE 2

Chemical composition of spodumene ore

Ore	Mass fraction, %						Molar ratio
	Li_2O	Na_2O	K_2O	Al_2O_3	SiO_2	$(FeO + Fe_2O_3)$	$Al_2O_3 : Li_2O$
Regions enriched with spodumene	1.35	2.16	1.19	16.30	72.20	2.6	3.7
Mine-run	0.95	2.05	1.11	16.85	73.30	2.6	5.2

Note. Concentrations of admixture of other ore components are 3–4 %.

content is 0.02–0.03 %). Due to this, lithium-containing cement with improved characteristics can be made from it [17].

Thus prepared Portland cement corresponds in its physical characteristics to the requirements of GOST (State Standard) for Portland cement of 300 grade. It was established that the conditions of cake leaching do not affect the quality of cement. In this connection, with the optimization of mixture composition and caking process, the characteristics of Portland cement made of the wastes of lithium production can be improved (to the level corresponding to grade 400 and above) [15].

On the basis of investigations performed, a scheme of integrated processing of unconcentrated spodumene ore was developed, leading to DHAL-CO_3 and the products of its processing, as well as various kinds of cement (Fig. 2).

Experimental-industrial tests using representative samples of ore from the Zavitinskoye deposit, with the total amount of about 20 t, demonstrated reproducibility of examination performed, and an increase in total yield of lithium in the final product at a level of 85–88 % [7] (under laboratory conditions it was 90–95 %). Taking into account losses during ore concentrating, the yield of lithium from spodumene concentrate is 65 % [1].

Results of the experimental-industrial test were laid into the basis of calculation of economical efficiency of ore processing according to the proposed technology. The data on the composition of ore from the regions enriched with spodumene and mine-run ore without their combined concentrating (gravitational and by means of floatation). The quantitative calculation data on the products obtained from 1 t of ore are presented in Table 3.

The choice of ore from the Zavitinskoye deposit for estimations of the economical efficiency was due to several factors: first, deficit of cement in the Far East and Siberian Federal Territories; second, the infrastructure available at the deposit (Zabaikalskiy Mining-and-Processing Integrated Works (MPIW)), which allows one to decrease the capital investment for the construction of cement plant; third, the possibility to obtain lithium products from DHAL-CO_3 at the KCMP, in particular lithium hydroxide monohydrate. Anhydrous lithium aluminates (lithium concentrate) can be obtained directly at the cement plant by calcinations of DHAL-CO_3, and used as an additive that decreases energy consumption for the production of aluminium [16, 18, 19], and also to obtain metal lithium [20]. This circumstance is important because the majority of large aluminium plants of Russia are situated at the territory of East Siberia (Krasnoyarsk, Bratsk, Irkutsk, Sayan *etc.*).

Calculation of the economical efficiency was made for the two versions of the organization of production on the basis of processing the mine-run ore of the Zavitinskoye deposit.

Version 1. Ore mining, preparation of DHAL-CO_3 and sludge for the production of

TABLE 3

Quantitative data on the products obtained after processing the ore from the Zavitinskoye deposit

Ore	Li_2O content, kg/t of ore	Products, kg/t of ore			
		$LiOH \cdot H_2O$	Lithium concentrate	Sludge cement	Portland cement
Regions enriched with spodumene	13.5	34	119	2620	2880
Mine-run	9.5	24	84	2700	3000

TABLE 4

Characteristics of the economical efficiency of processing unconcentrated spodumene or from the Zavitinskoye deposit (Li_2O 0.95 %)

Parameter	Version I	Version I
Annual amount of manufactured products, thousand tons:		
carbonate form of double aluminium-lithium hydroxide DHAL-CO_3 (Li_2O 6.0 %)	57.0	57.0
lithium hydroxide monohydrate (Li_2O 37.5 %)	9.6	–
lithium concentrate (Li_2O 10 %)	–	34.2
sludge after cake leaching (Li_2O 0.02–0.03 %)	720	720
cement	1160	1160
Annual cost of production for sale, mln roubles	5550	5160
Total capital investment, mln roubles	2754	2778
Including:		
ore mining	1100	1100
production of DHAL-CO_3	176	176
production of lithium concentrate	–	24
production of cement	1267	1267
Annual operational expenses, mln roubles	1319	1430
Net profit during the period of project implementation, mln roubles	32 605	26 665
Pure discounted income, mln roubles	7854	6106
Internal norm of yield, %	77.9	65.0
Pey-back period, years	2.8	3.0

Notes. 1. Capital investment is shown without taking into account the infrastructure available at the Zabaikalskiy MPIW. 2. Realization of the products is calculated for the prices lower than the market price by 20–25 %.

cement at the deposit, transportation of DHAL-CO_3 to KCMP to obtain lithium hydroxide monohydrate.

Version 2. Ore mining, preparation of DHAL-CO_3 and sludge, production of anhydrous lithium aluminates and cement directly at the deposit.

Annual amounts of the raw material to be processed for both versions – mine-run ore from the Zavitinskoye deposit and chalkstone – are 400 and 1460 thousand tons, respectively (calculated for CaO, the amount of chalkstone is 820 thousand tons).

Estimation of the economical efficiency of the production was carried out using the standard procedure of profit approach based on the comparison of discounted fluxes of profits and expenses during the 15-year period of project implementation [21]. Prices of production and raw materials at the beginning of 2008 were used in calculation.

Results of the estimation of economical efficiency of the project for the versions under consideration are listed in Table 4. One can see that both versions of the integrated processing of the initial ore with obtaining lithium products and sludge for the production of cement turned out to be highly efficient. Higher efficiency of version 1, in spite of the necessity to transport DHAL-CO_3 for processing to KCMP is explained by the higher cost of lithium hydroxide monohydrate in comparison with the cost of lithium concentrate obtained at the deposit for its further use in glass and aluminium production [4, part II].

One can see in the data shown in Table 5 that the integrated processing of lithium-bearing base ores decreases the cost of obtaining both lithium hydroxide monohydrate and cement. Calculated net cost of lithium hydroxide monohydrate is US 1020 dollars for 1 t (for the dollar rate of 26 roubles), while its price in China at the end of 2007 was US 3700 dollars, and in the US 4500–5000 dollars for 1 t. The net-cost of cement production according to the proposed technology will not exceed

TABLE 5

Calculated net cost of obtaining commercial products from integreted processing mine-run spodumene ore of Zavitinskoye deposit, roubles/t

Product	Version I	Version II
Lithium hydroxide monohydrate	26 500	–
Lithium concentrate	–	4800
Cement	1100	1100

1100 roubles/t, its market price being at present 5000 roubles/t.

CONCLUSION

Integrated processing of aluminosilicate ore with low lithium content without their preliminary concentrating allows one to expand the raw material basis for obtaining not only strategically important lithium products but also cement in broad demand. In addition, the two-stage scheme of production arrangement allows one to extract also aluminium from the raw material, thus obtaining lithium concentrates that are needed not only in the production of glass, ceramics and aluminium but also in the production of new materials and metal lithium. Within the classical calcareous method of processing spodumene raw material, aluminium remains in sludge.

The high economical characteristics of the integrated processing of lithium-bearing ore allow substantial decrease in the net cost of lithium products and cement. The determining economical factor for the integrated processing of low-grade spodumene ore is the production of cement.

A decrease in Li_2O content in unconcentrated spodumene ore (to less than 0.95 %) is accompanied by a decrease in the amount of lithium production and can affect the pay-back period. Capital investment for the construction of the cement plant at the industrial territory of the Zabaikalskiy MPIW with the available communications to supply energy, gas, water, and transportation infrastructure can be much lower than the calculation data.

Results of investigations on the integrated technology of processing unconcentrated spodumene ore and estimation of its economical efficiency provide evidence of the broad possibilities of its application for processing other kinds of raw material (wastes from OMPE of ores from reserve and newly explored deposits).

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