ISSN 2411-3336; e-ISSN 2541-9404

JOURNAL OF MINING INSTITUTE
Zapiski Gornogo instituta
Journal homepage: pmi.spmi.ru

Research article
Geotechnical Engineering and Engineering Geology

Production of calcium carbonate with tailored particle size characteristics as a factor in efficient recycling of gypsum-containing technogenic raw materials

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***How to cite this article*:** Suchkov D.V., Litvinova T.E., Lebedev A.P. Production of calcium carbonate with tailored particle size characteristics as a factor in efficient recycling of gypsum-containing technogenic raw materials. Journal of Mining Institute. 2026. Vol. 281. Iss. 2, p. 90-104.

Abstract
This study focuses on developing a method for utilizing phosphogypsum, a large-tonnage waste product from phosphate raw material processing, whose disposal sites are sources of complex negative impacts on the environment. As a promising approach to the utilization of gypsum-containing waste, the study considers the interaction of phosphogypsum with a sodium carbonate solution (carbonate conversion), which yields calcium carbonate, a marketable product referred to as phosphochalk. Existing technologies typically produce a fine-grained material, which hinders filtration and restricts the potential applications of phosphochalk. This study aims to determine the carbonate conversion parameters that enable the production of phosphochalk with tailored particle size characteristics while maintaining a high degree of feedstock conversion. A series of single-factor experiments was carried out to evaluate the influence of reaction conditions – temperature, Na_2CO_3 concentration in the carbonate solution, liquid-to-solid (L:S) ratio, and phase contact time – on the conversion of $CaSO_4 \cdot 2H_2O$. The results showed that raising the conversion temperature to 90-100 °C and increasing the L:S ratio to 10 promote intensive nucleation of $CaCO_3$ crystals and the formation of large agglomerates. A Na_2CO_3 concentration of 250 g/dm^3 and a phase contact time of up to 3 h together yield phosphochalk with a particle size of $D90$ = 45 μm at a conversion degree of 98.8 %. In terms of its characteristics, the resulting product is suitable for use in the metallurgical industry as a raw material for flux production, which demonstrates its competitiveness in the secondary raw materials market. The recommendations developed in this work enable the design of a low-waste technology for processing technogenic raw materials, consistent with the principles of sustainable development and helping to reduce the environmental burden.

Keywords
gypsum-containing waste; calcium carbonate; carbonate conversion; waste utilization; pulp filtration rate; phosphogypsum; phosphochalk

Funding
None.

Received: 31.03.2026 ***Accepted: 25.06.2026*** ***Online: 18.09.2026*** ***Published: 30.10.2026***

Introduction

The problem of managing large-tonnage waste from the mineral resources sector, including the chemical (processing) industry, has remained acute over the past decades. During the processing of phosphate raw materials via the sulfuric acid (wet-process) route, over 150 million t of phosphogypsum (PG) are generated annually in Russia alone – a by-product consisting predominantly of calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$) or hemihydrate ($CaSO_4 \cdot 0.5H_2O$) [1]. The Russian Federation is home to 12 enterprises employing sulfuric acid technology for the production of wet-process phosphoric acid, which represents one of the main sources of PG. At the waste disposal facilities (WDF) of these enterprises (gypsum ponds), approximately 10-12 million t of PG have been accumulated. At currently operating plants, this amount increases by 0.8 million t annually. The majority of WDF are

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located in the European part of Russia; in the Northwestern Federal District alone, over 150 million t of waste have already been placed in three gypsum ponds (Fig.1). Among the largest WDF are:

• The "Combined Phosphogypsum Slurry Storage" (N 35-00029-X-00592-250914, covering an area of 500 ha, with a design capacity of 120 million t) of the Cherepovets chemical cluster of Apatit JSC;

• The "Secondary Materials and Waste Landfill (Phosphogypsum Dump)" (N 64-00066-X-00592-250914, covering an area of 159 ha, with a design capacity of 121.5 million t) of the Balakovo branch of Apatit JSC.

Gypsum ponds often act as sources of serious environmental pressure through intense dust emissions [2] and the formation of technogenic hydrochemical flows due to pollutant infiltration through the dump body into soil and underspoil wastewater [3-5], which may require additional treatment [6]. Similar patterns of technogenic hydrochemical flow formation have been identified in studies of the impact of potash production waste [7] and mineral fertilizer plants [8] on surface water and groundwater.

Current approaches to PG utilization include its use in the construction industry, agriculture, and the recovery of rare earth elements [9, 10]. However, the presence of water-soluble impurities complicates its use [11], and technologies aimed at extracting valuable components generally do not result in a significant reduction of stored volumes [12]. All of this confirms the need to find effective measures for reducing the impact of objects of accumulated environmental damage on the environment. Within the paradigm of sustainable development, the most effective way to minimize damage is not the isolation of waste but its maximum reintegration into economic turnover as secondary material resources [13, 14]. For PG, carbonate conversion represents such a method [15, 16]. The conversion of phosphogypsum in carbonate media represents the interaction of $CaSO_4 \cdot nH_2O$ with carbonate-containing solutions (sol.) of M_2CO_3, where M^+ is an NH_4^+, Na^+, or K^+ ion, according to the general reaction

$$CaSO_4 \cdot 2H_2O + M_2CO_3 = CaCO_3 + M_2SO_4 + 2H_2O.$$

The conversion products are chemically precipitated calcium carbonate (phosphochalk, PC) and the corresponding sulfate M_2SO_4 [17, 18]. It should be noted that this process may be accompanied by the simultaneous utilization of CO_2-containing flue gases when used as a source of carbonate ions [19, 20].

However, the practical value of this method directly depends on the properties of the resulting phosphochalk and, above all, on its particle size. Known technical solutions in the field of PG carbonate conversion share a common drawback – the resulting PC is a finely dispersed product with a particle size generally not exceeding 20-25 μm [21]. The presence of such a precipitate complicates

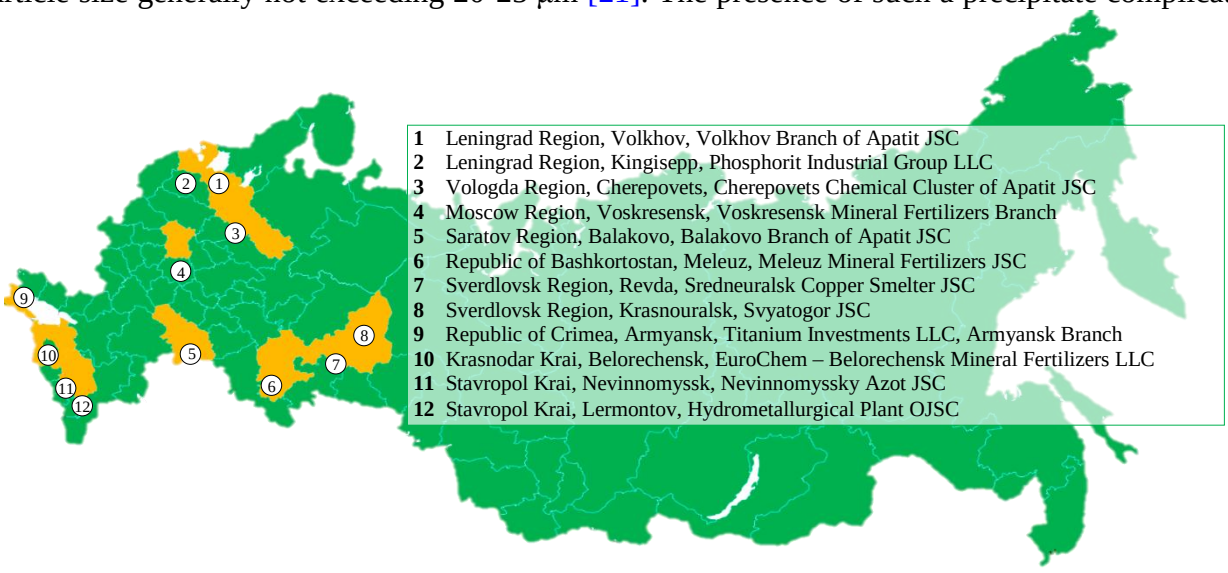


Fig.1. Location of PG waste-generating facilities in the Russian Federation

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pulp filtration, which increases energy costs for dewatering and limits the potential applications of the finished product, since in a number of industries, including metallurgy and building materials production, coarser carbonate raw materials are required [22]. Thus, solving the technological problem of controlling the granulometric composition of phosphochalk is a necessary condition for realizing the environmental potential of the carbonate conversion method. Obtaining a product with tailored particle size characteristics ensures its market demand, closes the processing cycle of technogenic raw materials, and, as a consequence, reduces the volume of waste sent for long-term disposal.

The aim of this work is to determine the parameters of phosphogypsum carbonate conversion that ensure the production of phosphochalk with tailored particle size characteristics while maintaining a high degree of conversion of the raw material. To achieve this aim, the following tasks were set:

• Justification of the choice of the carbonate reagent and the ranges of variation of technological parameters.

• Experimental evaluation of the influence of carbonate solution concentration, temperature, liquid-to-solid ratio (L:S), and phase contact time on the degree of conversion and granulometric composition of the product.

• Determination of conversion conditions that ensure the formation of large phosphochalk agglomerates with a high degree of conversion, suitable for use in the metallurgical industry.

Methods

Object of study. Experiments were carried out with model samples based on chemically pure $CaSO_4 \cdot 2H_2O$. The choice of model samples as the object of study made it possible to exclude the influence of impurities associated with PG and to focus on studying the patterns of carbonate conversion in its pure form. Studies of the properties of technogenic deposits in PG dumps formed during the processing of phosphate raw materials of various origins demonstrate that it is $CaSO_4 \cdot 2H_2O$ that is the main mineral phase in the body of gypsum ponds. Since transformations of PG in the dump body proceed by the hydration mechanism, regardless of the form (dihydrate or hemihydrate) in which the waste initially enters storage, under the influence of atmospheric precipitation and air humidity, calcium sulfate hemihydrate transforms into the dihydrate form. Consequently, the use of the dihydrate as a model compound is appropriate from the standpoint of the prospects for the utilization of both fresh and stockpiled PG.

Reagents. Na_2CO_3 solution of "chemically pure" grade prepared with distilled water was used as the carbonate reagent. This choice was based on the results of preliminary experimental studies showing that the use of Na_2CO_3 provides the best combination of parameters in terms of precipitate yield, filtration rate, and particle size of the resulting product compared to K_2CO_3 [23].

An alternative reagent – $(NH_4)_2CO_3$ – is known as the basis of the Merseburg process for producing $(NH_4)_2SO_4$. However, in industrial practice, its use is associated with serious technical difficulties. In particular, when PG interacts with an ammonia solution, solidification of the reaction suspension often occurs, caused by too rapid crystallization of ammonium carbonate and/or bicarbonate, which makes further mixing and filtration impossible; solving this problem complicates the process equipment design[1]. In addition, at the high temperatures considered in this work, the thermal instability of $(NH_4)_2CO_3$ leads to its intensive decomposition and ammonia losses [24]. Thus, to solve the fundamental problem of controlling precipitate particle size, the use of Na_2CO_3 is a more reliable and methodologically correct solution. Nevertheless, the study of the ammonium route with a focus on obtaining $(NH_4)_2SO_4$ as a second commercial product (a valuable fertilizer) represents a separate promising research direction.

The concentration of the Na_2CO_3 carbonate solution (CS) was varied in the range from 200 to 300 g/dm^3 in increments of 25 g/dm^3. The density values of the solutions were determined by calculation

[1] Toczek A., Merka W., Gosciniak P., Gosciniak M. Patent N US 10919774 B2. Method for the management of phosphogypsum. Publ. 16.02.2021.

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using linear relationships derived from reference data[2]. The choice of the CS concentration range is due to the results of stoichiometric calculation. At concentrations below 200 g/dm^3, the required excess of carbonate ions relative to $CaSO_4 \cdot 2H_2O$ is not achieved, which leads to incomplete reaction and a decrease in the degree of conversion; the precipitate will contain a significant amount of unreacted gypsum. The upper limit of the range (300 g/dm^3) was chosen based on the solubility of Na_2CO_3.

Equipment and research methods used. The experimental part of the work was carried out at the Scientific Center "Problems of Mineral and Technogenic Resources Processing" and the Research Center "Assessment of Anthropogenic Transformation of Ecosystems" of Empress Catherine II Saint Petersburg Mining University.

Initial conversion conditions: CS concentration, g/dm^3; L:S ratio by mass; temperature, °C; phase contact time, min; stirring speed, rpm. Controlled product properties: precipitate yield, % of $CaSO_4 \cdot 2H_2O$ mass; precipitate moisture, %; filtrate yield (sulfate solution, SS), % of CS volume; pH of SS; pulp filtration rate, m^3/(m^2·h); precipitate particle size, μm; conversion degree, %. The schematic diagram of PG carbonate conversion is shown in Fig.2.

The study involves a series of single-factor experiments, in each of which only one of the initial parameters is varied within a certain basic range. The other factors are fixed at average values. This makes it possible to evaluate the influence of each parameter on the controlled properties of the reaction products in isolation. The basic ranges of key factors affecting conversion were determined during preliminary experiments [23]. In the present study, these ranges were divided into equal intervals to monitor changes in the properties of conversion products.

The experiments were carried out on a H.E.L. Parallel Reactor multi-reactor system (UK). A calculated amount of the carbonate solution was loaded into the reactor and heated to the required temperature (range 40-105 °C, in steps of 10-15 °C), after which a weighed portion of $CaSO_4 \cdot 2H_2O$ was added. The liquid-to-solid ratio by mass was varied from 2 to 10.

The reaction mixture was thermostated with constant stirring (speed 400 rpm) for a specified time (from 30 to 180 min). The stirring speed was chosen based on preliminary experiments as clearly sufficient to remove external diffusion limitations; additional verification of the effect of stirring on the reaction rate confirmed the absence of significant changes when the speed was increased to 600 rpm [24].

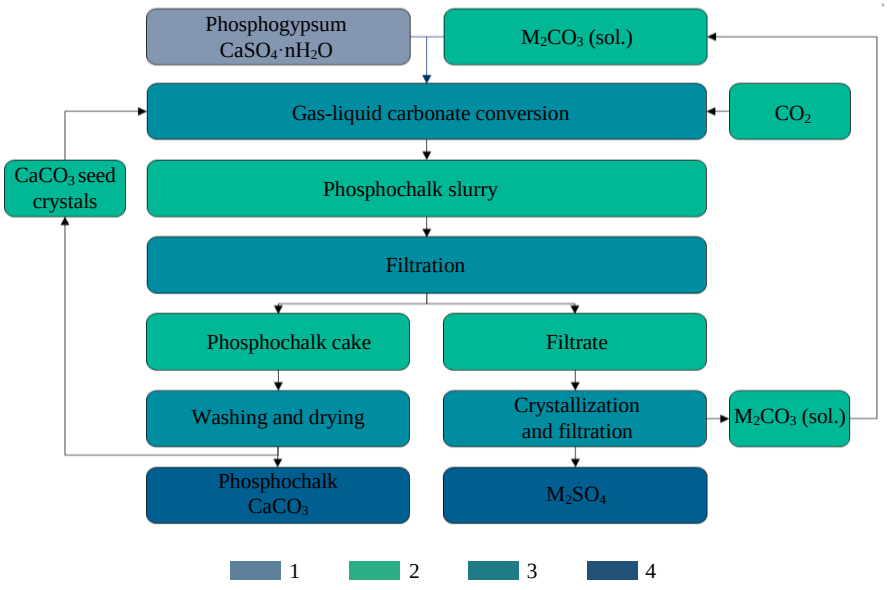


Fig.2. Schematic diagram of the PG carbonate conversion process

1 – secondary material resource; 2 – intermediates; 3 – raw materials; 4 – commercial products

[2] Database. Thermal constants of substances (working version 2) (in Russian). URL: https://www.chem.msu.ru/cgi-bin/tkv.pl?show=welcome.html (accessed 30.03.2026).

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Upon completion of the process, the pulp was subjected to vacuum filtration on a Büchner funnel using a HBM 2×2 vacuum pump (Russia) at a constant pressure of 99 kPa. The volume of the obtained filtrate (sodium sulfate solution) was measured with a graduated cylinder, and the pH value was determined by the potentiometric method using a stationary HANNA HI 2210-02 pH meter (Germany). After filtration, the precipitate was dried at 105 °C in a Binder ED 400 drying oven (Germany).

Determination of filtration rate. The pulp filtration rate was determined by the standard method as the ratio of the filtrate volume to the product of the filter area and filtration time after fixing the passage of a specified volume of washing water through a precipitate layer of known thickness at a constant vacuum pump pressure. To evaluate pulp filterability, a classification was used according to which pulps with a filtration rate of less than 1.5 $m^3/(m^2 \cdot h)$ are classified as difficult-to-filter, and more than 1.5 $m^3/(m^2 \cdot h)$ as easy-to-filter.

Determination of granulometric composition. Before analysis by laser diffraction on a "Microsizer 201C" particle size analyzer (Russia), samples were subjected to ultrasonic treatment for 2 min to break down agglomerates; a 0.1 % sodium hexametaphosphate solution was used as the dispersion medium in accordance with the instrument operating manual. This approach is standard for disaggregation of finely dispersed carbonate materials and is widely used in the mineral industry as a method of physical dispersion of calcite, apatite, and other minerals that does not lead to the formation of new phases [25] due to the extremely low concentration of the solution, small dosage volume, and minimal contact time. Measurement results were presented as cumulative distribution curves with determination of parameters *D*50 and *D*90 – the diameters below which 50 and 90 % of particles are respectively located. For visual assessment of particle morphology and confirmation of laser diffraction data, a Vega 3 LMH scanning electron microscope (SEM) (Czech Republic) was used; before imaging, samples were sputter-coated with a conductive carbon coating using a Q150R E unit.

Determination of conversion degree. The chemical composition of the initial raw material and conversion products was monitored by X-ray fluorescence analysis (XRF) on an Epsilon 3 X-ray fluorescence spectrometer (Netherlands). The degree of $CaSO_4 \cdot 2H_2O$ conversion to $CaCO_3$ (E, %) was assessed by the residual sulfur content in the precipitate (C_S, %). Sulfur was chosen as the marker element because it is present only in the composition of the initial reagent; therefore, a decrease in its mass fraction in the product directly reflects the completeness of the reaction.

The sulfur content was determined by comparing the analytical signal intensity of the test sample with that of a reference sample – chemically pure gypsum with known sulfur content (18.62 %). The calibration curve constructed from calculated data for mixtures of $CaCO_3$ and $CaSO_4 \cdot 2H_2O$ with different component ratios is described by the linear equation

$$E = -5.3694 C_S + 100.$$

Since the points on the calibration graph are calculated rather than experimental, the coefficient of determination R^2 was 1.00. In the practical application of the method, the relative error of sulfur determination by XRF did not exceed 7 %, which was taken into account when interpreting the results.

Kinetic studies. To substantiate the conclusion about the change in the rate-limiting step at elevated temperatures, additional experiments were carried out at temperatures of 80, 90, and 100 °C with the conversion degree recorded over the time interval from 0.2 to 25 min. From the initial sections of the kinetic curves, instantaneous reaction rates were calculated, on the basis of which, using the Arrhenius equation, the apparent activation energy was determined. A detailed presentation of the kinetic patterns is the subject of a separate study; this work provides only generalized data necessary for interpreting the results.

Experimental design and statistical processing. To increase the reliability of the results, each measurement was performed at least three times, and the arithmetic mean was taken as the final value. The obtained data were statistically processed with outliers outside the confidence interval ($p < 0.05$) excluded.

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Results

The experimental results showed that all considered factors have a significant effect on the degree of $CaSO_4 \cdot 2H_2O$ conversion and the resulting phosphochalk granulometric composition; however, the nature of this effect varies. Figure 3 presents changes in the main process indicators depending on the varied parameters. The values obtained at the minimum level of the varied factor were taken as the reference point (conditional zero).

Carbonate solution concentration. At a fixed temperature of 70 °C, an L:S ratio of 5, and a phase contact time of 60 min, an increase in Na_2CO_3 concentration from 200 to 300 g/dm^3 is accompanied by an increase in the conversion degree from 81.2 to 97.9 %. The most intensive increase is observed in the range of 200-250 g/dm^3, where the conversion degree increases by 12 %. A further increase in concentration to 300 g/dm^3 gives an increase of only 0.3 %, which is within the measurement error.

The dependence of product particle size on concentration is extreme in nature – the *D*90 value increases from 19.2 μm at 200 g/dm^3 to 30.5 μm at 275 g/dm^3, but at 300 g/dm^3 it decreases to 26.5 μm. The median diameter *D*50 changes insignificantly – from 6.9 to 7.6 μm with a subsequent decrease to 7.5 μm. The pulp filtration rate reaches a maximum value of 2.39 m^3/(m^2·h) at a concentration of 250 g/dm^3, while at 300 g/dm^3 it decreases to 1.15 m^3/(m^2·h).

Temperature of the process has the most significant effect on product properties. Studies carried out at a Na_2CO_3 concentration of 250 g/dm^3, an L:S ratio of 5, and a phase contact time of 60 min in the temperature range from 40 to 105 °C showed that the conversion degree increases from 93.7 % at 40 °C to 96.5 % at 105 °C. However, the change in granulometric composition is of key importance. At temperatures up to 80 °C, the *D*90 value stabilizes at 24.5-25.8 μm. An increase in temperature to 95 °C leads to an increase in this parameter to 30.1 μm, and at 105 °C – to 38.0 μm.

The median diameter *D*50 changes less significantly – from 7.7 to 7.3 μm, which indicates a predominant increase in the proportion of coarse particles while maintaining the bulk of the product in the medium size range. Particle size distribution curves demonstrate a qualitative change in granulometric composition upon transition through 80 °C; at low temperatures, the distribution is narrow and unimodal, while at 100 °C a pronounced shoulder appears in the coarse size range, and at 105 °C a second maximum forms in the 40-60 μm range. The pulp filtration rate in the studied range increases from 1.03 to 2.23 m^3/(m^2·h).

Liquid-to-solid ratio (by mass) is a factor determining both the completeness of the reaction and the PC particle size. At a fixed Na_2CO_3 concentration of 250 g/dm^3, a temperature of 70 °C, and a phase contact time of 60 min, an increase in the L:S ratio from 2 to 10 is accompanied by an increase in the conversion degree from 46.7 to 98.8 %. When the amount of the liquid phase is insufficient (L:S = 2), the conversion degree does not exceed 46.7 %, and the precipitate is a mixture of unreacted gypsum and calcium carbonate. Increasing the L:S ratio to 3.5 ensures an increase in the conversion degree to 93.5 %, and upon reaching a ratio of 4.5 and above, this value stabilizes at 98.3-98.8 %.

The PC particle size shows a stable tendency to increase throughout the studied range – *D*90 increases from 24.4 μm at L:S = 2 to 35.8 μm at L:S = 5 and reaches 42.6 μm at the maximum L:S ratio of 10. The median diameter *D*50 increases from 6.2 to 7.9 μm. The pulp filtration rate changes non-monotonically: with an increase in the L:S ratio from 2 to 5, a decrease from 2.39 to 1.41 m^3/(m^2·h) is observed, which is associated with an increase in the proportion of fine PC particles as the conversion degree increases. With a further increase in the ratio to 10, the filtration rate increases to 3.17 m^3/(m^2·h), which correlates with an increase in agglomerate size and a decrease in pulp viscosity due to dilution.

Phase contact time has a different effect on the conversion degree and PC particle size. Studies carried out at a Na_2CO_3 concentration of 250 g/dm^3, a temperature of 70 °C, and an L:S ratio of 5 showed that the conversion degree increased slightly with increasing time from 30 to 180 min – from 95.3 to 98.6 %, with the main increase (up to 97.3 %) occurring in the first 60 min. This indicates a high reaction rate already at the initial stage of conversion. The PC particle size turned out to be

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more sensitive to changes in phase contact time. In the first 30-60 min, D90 increased from 30.5 to 33.4 μm, and by 180 min it reached 42.6 μm. The increase in the proportion of particles larger than 30 μm is especially noticeable at phase contact times exceeding 120 min.

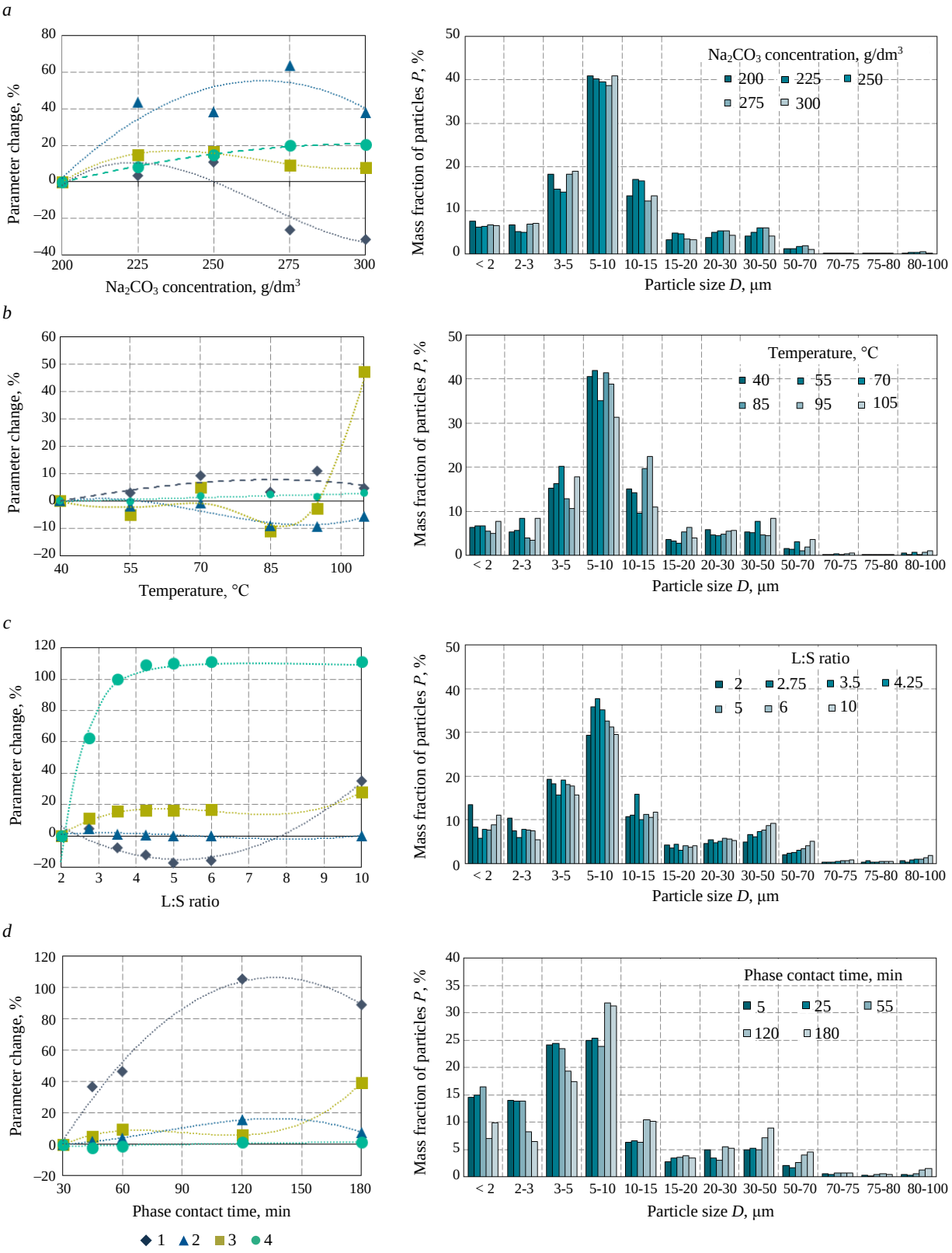


Fig.3. Variation of the main process parameters as a function of the variable factors:
a – effect of Na_2CO_3 concentration; *b* – effect of conversion temperature;
c – effect of liquid-to-solid ratio; *d* – effect of phase contact time

1 – filtration rate; 2 – particle size *D*50; 3 – particle size *D*90; 4 – conversion degree

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The median diameter $D50$ increases from 6.7 to 7.2 μm. The nature of the change in granulometric parameters (growth of $D90$ with negligible change in $D50$) indicates that an increase in phase contact time predominantly promotes agglomeration of primary crystals rather than their growth. The pulp filtration rate increases proportionally to the increase in agglomerate size; at τ = 30 min it is 1.03 $m^3/(m^2 \cdot h)$, at τ = 120 min – 2.23 $m^3/(m^2 \cdot h)$, at τ = 180 min – 2.39 $m^3/(m^2 \cdot h)$.

Kinetic studies carried out at temperatures of 80, 90, and 100 °C showed that the conversion degree reaches 90-93 % already in the first minute of phase contact, indicating a high reaction rate. Based on the analysis of the temperature dependence of the initial reaction rate, the apparent activation energy of the process was calculated to be 89.9 kJ/mol.

Discussion

The experimental results demonstrate that the granulometric composition of chemically precipitated $CaCO_3$ can be controlled by deliberately varying the reaction conditions. Each of the considered factors has a specific effect on both the mechanism of particle formation and the conversion degree.

Polymorphism of $CaCO_3$. In the $CaSO_4 \cdot 2H_2O$–Na_2CO_3–H_2O system, amorphous calcium carbonate or its metastable polymorphic modifications (vaterite, aragonite) may form at the initial stage. Nevertheless, in all experiments, the precipitate was identified as calcite. This is consistent with literature data on the rapid recrystallization of metastable phases under elevated temperatures and high supersaturations, and is confirmed by X-ray diffraction (XRD) analysis performed using a Shimadzu XRD-7000 powder diffractometer (Japan). No metastable phases were detected, which can be attributed to their rapid recrystallization under the experimental conditions. The formation of calcite, which has a rhombohedral crystal habit, promotes the assembly of dense agglomerates observed in SEM images obtained with a Vega 3 LMH scanning electron microscope.

Analysis of the effect of carbonate solution concentration. The observed relationship between the carbonate solution concentration and both the conversion degree and product particle size is consistent with the general understanding of how supersaturation affects crystallization processes. An increase in Na_2CO_3 concentration to 250-275 g/dm^3 increases the driving force of the reaction, thereby contributing to both a higher conversion degree and coarsening of the resulting particles. However, the decrease in $D90$ at a concentration of 300 g/dm^3 (at a nearly unchanged $D50$) suggests that nucleation and agglomeration processes compete under these conditions. At concentrations approaching saturation, the supersaturation with respect to $CaCO_3$ becomes extremely high, shifting the balance toward homogeneous nucleation. The vast number of fine primary particles produced under these conditions hinders the formation of large agglomerates. Thus, at 300 g/dm^3, massive nucleation becomes the dominant mechanism, suppressing agglomeration and adversely affecting the resulting particle size characteristics.

Analysis of the effect of temperature. The conversion temperature exerts the most significant effect on PC particle size. The increase in $D90$ as the temperature rises to 90-100 °C, together with the qualitative change in the granulometric curves (the appearance of a second maximum in the coarse size range without a significant change in $D50$), indicates a shift in the dominant particle formation mechanism. It is reasonable to assume that the high reaction rate at temperatures above 80 °C produces numerous fine primary crystals, which subsequently assemble into larger agglomerates.

The apparent activation energy calculated from the initial sections of the kinetic curves was 89.9 kJ/mol. This is noticeably higher than the values characteristic of diffusion-controlled processes (8-37 kJ/mol) [26, 27]. Together with the absence of any effect of stirring speed on the conversion degree, this suggests that the rate-limiting step of the conversion reaction may be either the chemical reaction itself or internal diffusion within the pores of the forming agglomerates. A definitive answer to this question requires more detailed kinetic experiments.

Analysis of the effect of the liquid-to-solid ratio confirms that the process must be carried out under conditions of CO_3^{2-} excess. At L:S below 3.5, the conversion degree does not exceed 76 %,

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which is due to mass transfer limitations and a decrease in ion mobility in concentrated suspensions. At L:S ratios of 4.5 and above, maximum conversion degrees (98.3-98.8 %) are achieved.

The non-monotonic behavior of the filtration rate with increasing L:S ratio requires separate analysis. The decrease in filtration rate in the L:S range from 2 to 5 (from 2.39 to 1.41 $m^3/(m^2 \cdot h)$) is due to two factors. At L:S = 2, the precipitate contains a significant amount of unreacted gypsum, the coarse crystals of which form a loose, porous structure that makes the pulp easily filterable. However, as L:S increases, the conversion degree also increases, which leads to an increase in the proportion of fine $CaCO_3$ particles in the precipitate, reducing the filtration rate. At L:S = 10, the formation of large $CaCO_3$ agglomerates already occurs, and the pulp viscosity decreases due to dilution. Collectively, these factors improve filtration conditions and significantly increase the filtration rate to 3.17 $m^3/(m^2 \cdot h)$.

Analysis of the effect of phase contact time. Carrying out the conversion for 120 min or longer leads to a noticeable increase in *D*90; however, *D*50 remains practically unchanged. Such an increase in the proportion of coarse fractions while maintaining the primary particle size is characteristic of the predominance of agglomeration processes rather than classical Ostwald ripening (increase in average size and narrowing of distribution). From a practical standpoint, the formation of stable large $CaCO_3$ agglomerates is advantageous because, as noted above, they improve filtration conditions and facilitate subsequent transportation and processing of the product.

Prospects for the use of phosphochalk. The technological process of PG carbonate conversion comprises the main stages outlined in Fig.4. In this case, the carbonate solution M_2CO_3 can be obtained either by absorbing CO_2-containing flue gases in a solution of the hydroxide of the corresponding ion MOH (method 1) or by dissolving solid carbonate M_2CO_3 (method 2).

Achieving a particle size of *D*90 = 45 μm is a key indicator, since it ensures a high pulp filtration rate (3.17 $m^3/(m^2 \cdot h)$). In turn, an easily filterable pulp reduces energy costs for dewatering within the technological process, making conversion more cost-effective. In addition, the utilization of PG by

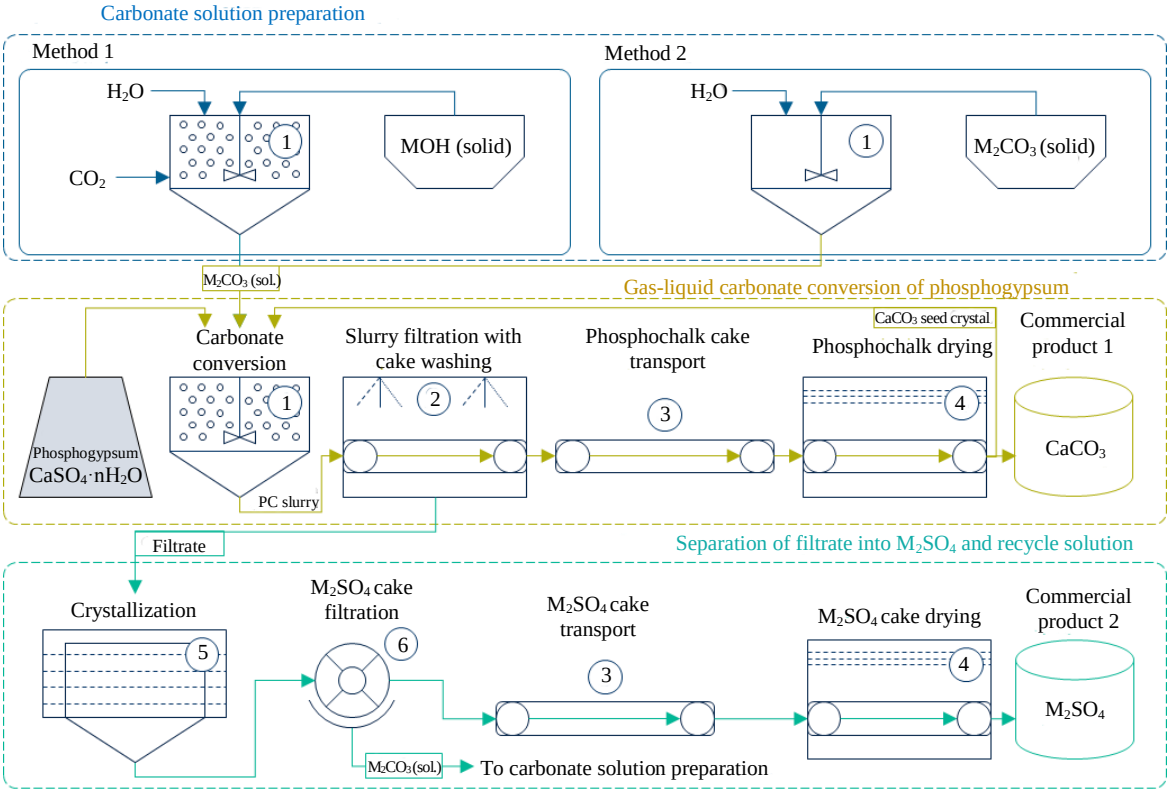


Fig.4. Block flow diagram of the PG carbonate conversion process

1 – mixing reactor; 2 – belt vacuum filter with precipitate washing; 3 – belt conveyor; 4 – belt dryer; 5 – crystallization reactor with cooling; 6 – vacuum filter

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the proposed method makes it possible to involve significant volumes of both freshly generated and previously disposed waste in processing, thereby preventing further environmental pollution within the impact zone of gypsum ponds.

It should be emphasized that the presented results were obtained for a model system. These results can serve as a basis for developing a technology for processing gypsum-containing waste; however, the presence of impurities in phosphogypsum (oxides of aluminum, iron, silicon, and phosphorus, as well as fluorides and rare earth elements) may affect the process kinetics and product properties, which requires additional studies on actual industrial samples [28].

The presence of fluoride ions can affect the course of carbonate conversion due to the interaction of fluorides with calcium carbonate, which can lead to the formation of sparingly soluble compounds on the particle surface, slowing down mass transfer and reducing the conversion degree. At the same time, secondary reaction products (calcium fluoride) can change the filtration properties of the pulp and affect the purity of the final product.

According to literature data, the rheological behavior of sodium carbonate and phosphogypsum suspensions strongly depends on temperature, time, and reagent concentration, and under industrial conditions, achieving a high conversion degree (93-96 %) takes significantly longer (up to 30 min) than in model systems [29]. Thus, the transfer of optimal parameters determined during the study to production conditions requires additional experiments directly with industrial phosphogypsum samples, including assessment of the effect of impurities on kinetics, conversion degree, and product morphology.

The presence of impurities in phosphochalk limits the possibility of using the product in industries requiring high-purity calcium carbonate; however, it is not critical for the metallurgical industry. In agglomerate production and pig iron smelting, fluxes based on calcium carbonate traditionally contain significant amounts of silicon, aluminum, and other oxides, and their content is regulated only within certain limits[3]. The resulting product with a particle size of up to 45 μm does not require additional grinding and can be used directly in agglomeration production, where, according to technical specifications, the fraction below 3 mm must constitute at least 95 %. The use of technogenic PG as a source of feedstock for flux production makes it possible to implement the principles of resource conservation and circular economy. Replacing natural limestone with technogenic phosphochalk reduces the burden on ecosystems associated with quarry development and decreases the volume of PG sent for disposal, together providing a double environmental benefit. This approach is consistent with recent studies demonstrating the prospects for processing various types of technogenic raw materials [30-32].

Assessment of environmental and economic efficiency. The assessment of the proposed technical solution was performed using a combination of technical-economic, market, and environmental-economic analysis methods. The calculations are based on the direct cost-benefit comparison method, which allows determining the commercial effect from processing phosphogypsum into marketable products, as well as the prevented environmental damage method, which reflects the environmental significance of the project.

With a design capacity of 32.4 thousand t of processed PG per year, the output of commercial PC will be 19.1 thousand t, and sodium sulfate – 30.1 thousand t. The design capacity of the plant was determined based on the specific productivity of a standard belt vacuum filter (0.5 t/(m^2·h) in terms of solids) and the possibility of operating three such filters in continuous mode.

When calculating economic efficiency, conservative price values were used: for technical calcium carbonate – 5900 rub./t, for sodium sulfate – 14,900 rub./t (average market price according to analytical reviews for 2023-2024). Revenue from product sales will amount to 112.7 million rub./year from phosphochalk and 448.5 million rub./year from sodium sulfate, totaling 561.2 million rub./year.

[3] Production of pig iron, steel and ferroalloys: information and technical reference book on best available technologies. ITS 26-2017. Moscow: NDT Bureau, 2017, p. 478 (in Russian).

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Journal of Mining Institute. 2026. Vol. 281. Iss. 2. P. 90-104
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Capital investments are estimated on the basis of aggregated cost standards for chemical industry equipment and include costs for the purchase of reactor equipment, filters, dryers, crystallizers, aspiration systems, as well as costs for transportation, installation, and commissioning (totaling about 40 % of equipment cost).

The economic effect associated with savings on waste disposal payments is 0.64 million rub./year. With average annual operating costs of 125.04 million rub./year (costs for electricity, water, labor, insurance contributions, depreciation, and other expenses), the annual economic effect will be 416 million rub. The project payback schedule and sensitivity analysis are presented in Fig.5.

It should be noted that, within the framework of a pre-project assessment, it is impossible to accurately determine the contractual price of the reagent under the terms of a long-term wholesale agreement with a specific enterprise. At large chemical complexes, such as Apatit JSC (Cherepovets), there is in-house infrastructure for receiving and storing large-tonnage reagents, as well as excess carbon dioxide – a by-product of ammonia production. At the same time, the technology for capturing CO_2 from flue gases has already been implemented at enterprises (for example, the chemical absorption method using monoethanolamine solutions) and does not require additional capital investments.

This technology also includes a stage of preliminary purification of flue gases from impurities, including SO_x and NO_x, which is a standard measure for protecting the absorption solution from

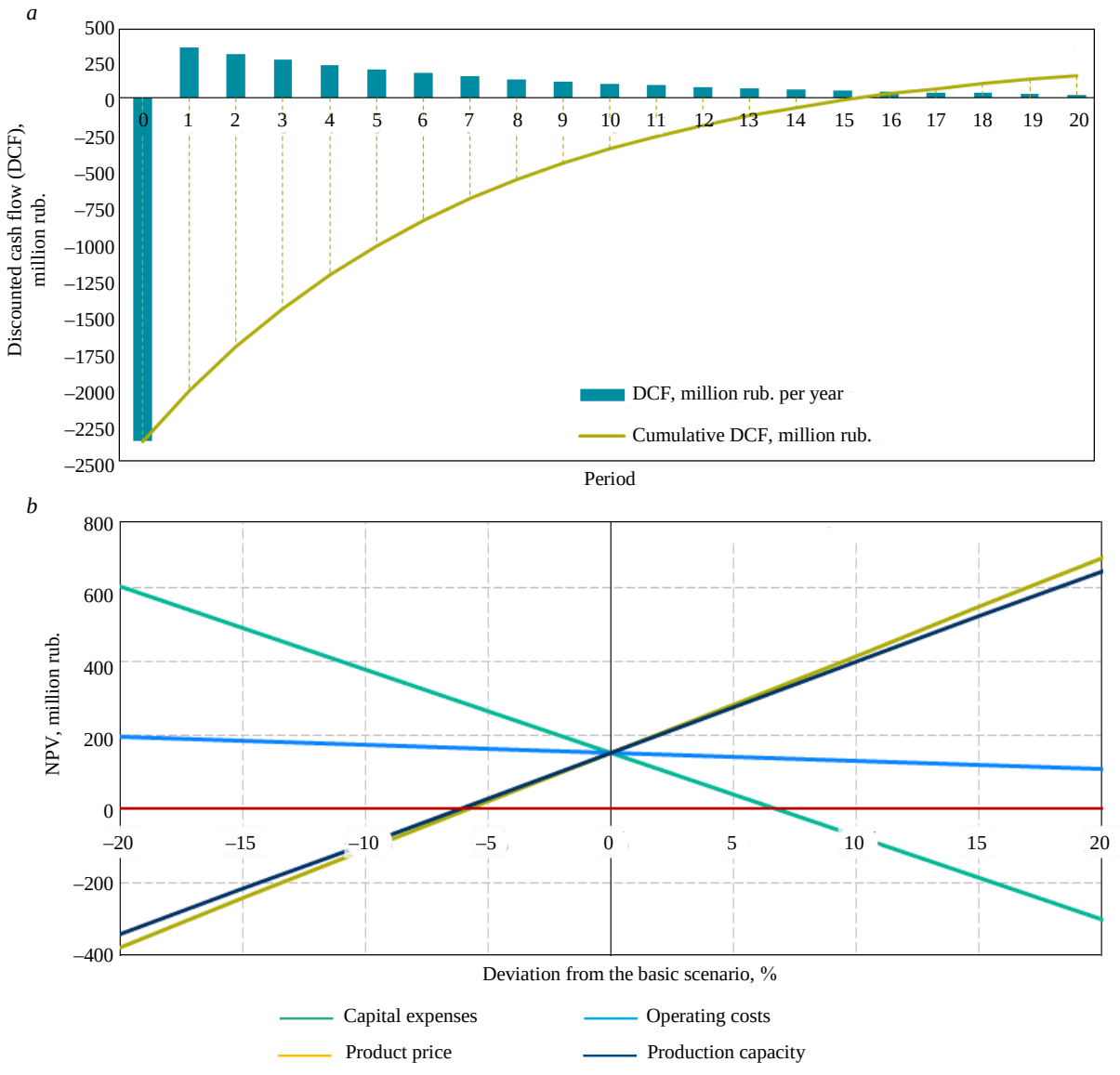


Fig.5. Results of economic efficiency assessment (*a*) and sensitivity analysis (*b*)

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degradation[4]. Thus, the carbonization process receives a CO_2 stream already purified from compounds that could affect the conversion process.

All this makes it technically possible to obtain the carbonate solution directly on site by absorbing CO_2 in an NaOH solution, which allows minimizing the costs of purchasing commercial Na_2CO_3. Thus, within the framework of the adopted model, it is assumed that the resulting cost of the reagent will be significantly lower than the market price and comparable to the revenue from the sale of commercial products. In this regard, for the purposes of this pre-project assessment, operating material costs for this expense item are not shown as a separate line item, which does not affect the final net cash flow within the accepted calculation accuracy. A detailed calculation of process profitability requires clarification of specific conditions at the engineering design stage.

An increase in the phase contact time to 180 min at a temperature of 90-100 °C certainly entails additional energy costs for maintaining the thermal regime of the reactor, which contributes to higher operating costs. Nevertheless, this set of conditions is necessary for the formation of large PC agglomerates. Achieving such a particle size ensures a high pulp filtration rate, which reduces costs at the dewatering stage compared to process conditions that produce a finely dispersed precipitate. A comprehensive environmental and economic analysis confirms that additional energy costs are not critical: with a planning horizon of 20 years, the net present value (NPV) of the project is 151.8 million rub. The discounted payback period (with capital investments of 2.35 billion rub.) is 16.3 years, which is an acceptable value for environmental projects. Thus, the chosen phase contact time is optimal in terms of the balance between energy consumption and technological efficiency.

Sensitivity analysis shows that the project is most sensitive to changes in prices for secondary raw materials (elasticity of 17.35), production capacity (16.23), and capital costs (–14.89). The project is least sensitive to changes in operating costs – elasticity is only –1.48. Thus, the most significant risk factors for the project are the volatility of prices for commercial products and the achievement of the plant's design capacity. To reduce sensitivity, it is recommended to conclude long-term contracts for the supply of secondary raw materials and to build in reserve capacity at the design stage. Moderate dependence on capital costs confirms the feasibility of using standard equipment and phased commissioning. Low sensitivity to operating costs indicates the stability of operating activities when current costs change.

The prevented environmental damage from land pollution is estimated at 40 million rub./year.

An additional environmental advantage is the possibility of using CO_2-containing flue gases for the preparation of the carbonate solution [33, 34], allowing up to 0.84 t of CO_2 to be sequestered per ton of processed waste. In a broader context, the proposed technical solution meets the principles of sustainable development, as it ensures a transition from the practice of long-term phosphogypsum storage to a circular economy model in which waste is considered as a secondary mineral resource [35]. Such a scheme not only reduces the burden on land resources and lowers long-term environmental risks, but also decreases the need for primary raw materials by returning calcium- and sulfur-containing components to economic circulation. Recent studies show that the resource-oriented processing of phosphogypsum, as compared to its disposal, is characterized by more favorable environmental and economic indicators based on life cycle assessment results, and the integration of CO_2 mineralization with the production of several marketable products enhances the climate and resource-saving benefits of the technology [36, 37]. Consequently, the implementtation of the considered solution contributes to the achievement of the Sustainable Development

[4] Manufacture of ammonia, fertilizers and inorganic acids: information and technical reference book on best available technologies. ITS 2-2019. Moscow: NDT Bureau, 2019, p. 825 (in Russian).

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Goals (SDG), primarily SDG 9 “Industry, innovation and infrastructure” and SDG 12 “Responsible consumption and production”, and, through carbon dioxide sequestration and reduced emission loads, also aligns with the climate agenda objectives of SDG 13 “Climate action” [38, 39].

Thus, the studies performed confirm the fundamental possibility of controlling the granulometric composition of PC obtained during the carbonate conversion of PG and allow recommending a certain range of technological parameters for achieving the tailored product particle size. It has been established that the formation of large particles occurs predominantly by the mechanism of agglomeration of primary crystals rather than their growth, which is important for understanding the processes of structure formation and developing practical recommendations. The results obtained provide the basis for developing a technological scheme for processing technogenic gypsum-containing raw materials, aimed at producing materials in demand in the metallurgical industry.

Conclusion

This study identified the conditions for the carbonate conversion of phosphogypsum that make it possible to obtain phosphochalk with tailored particle size characteristics while maintaining a high degree of feedstock conversion.

The fundamental possibility of reducing the technogenic load from PG disposal facilities by converting the waste into a sought-after secondary resource – phosphochalk – has been substantiated. It has been demonstrated that a necessary condition for the environmental and economic efficiency of such processing is the control of product particle size, which can be achieved by varying the process parameters. The carbonate solution concentration, temperature, L:S mass ratio, and phase contact time exert the most significant effect on product properties.

The dependence of $CaCO_3$ particle size on Na_2CO_3 concentration is extreme in nature: an increase in concentration to 275 g/dm^3 promotes the growth of *D*90; however, a further increase leads to a decrease in this parameter with an almost unchanged *D*50. A conversion degree above 98 % is ensured only at an L:S ratio of not less than 4.5. When the amount of the liquid phase is insufficient, the reaction does not proceed to completion – the precipitate is a mixture of unreacted gypsum and calcium carbonate. A further increase in L:S not only solves this problem but also contributes to the coarsening of $CaCO_3$ agglomerates, thereby improving the filtration characteristics of the pulp. An increase in the phase contact time has a similar effect on the agglomeration of $CaCO_3$.

Based on a series of experiments, it was established that the maximum PC particle size (*D*90 = 45 μm) and conversion degree (98.8 %) are achieved under the following conditions: a carbonate solution (Na_2CO_3) concentration of 250 g/dm^3, a temperature of 90-100 °C, an L:S mass ratio of 10, and a phase contact time of 180 min. The formation of stable, large CaCO3 agglomerates ensures a high pulp filtration rate (3.17 m^3/(m^2·h). The specific yield of the commercial product – phosphochalk – is 0.59 t per ton of feedstock.

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The authors declare no conflict of interests.

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