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## Optimizing Oxidative Roasting of Low-Grade Molybdenum Intermediates on Phase-Controlled Process Parameters

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### Abstract

Processing low-grade molybdenum intermediates demands oxidative-roasting regimes that maximize Mo recovery and resource efficiency. This work proposes a phase-oriented optimization approach for a 22.23% Mo intermediate, grounded in the multifactor Protodyakonov-Malyshev model and tabular nomographs as a practical parameter-selection tool. Control experiments varied temperature to 700 °C with roasting limited to 20 min and bed height to 0.010 m. Phase evolution during roasting and Mo recovery after alkaline leaching were quantified from integrated mineralogical-chemical data. A robust process window was identified at 550 °C, achieving 97% Mo recovery. Increasing temperature above 600 °C initiates a mechanism shift that forms MoO<sub>2</sub>, reducing selectivity. Using the developed nomographs enables 20 min roasting while favoring MoO<sub>3</sub> formation (88%) and minimizing undesired phases, thereby improving overall resource efficiency for low-grade feed. The study's novelty is the adaptation of the Protodyakonov-Malyshev model to phase-composition control during roasting, steering the transformation from MoS<sub>2</sub> to MoO<sub>3</sub>, and proposing a resource-efficient alternative to conventional regimes by exploiting an exothermic reaction that supplies heat and requires no additional energy input. The results support the development of technological regulations, furnace design and operation, and industrial trials at copper-molybdenum plants for producing molybdenum products (CaMoO<sub>4</sub>).

**Keywords:** Molybdenum; Calcium Molybdate (CaMoO<sub>4</sub>); Low-Grade Molybdenum Concentrate; Oxidative Roasting; Alkaline Leaching; Phase-Controlled Processing; Multifactor Optimization; Protodyakonov-Malyshev Model; Digital Nomographs.

## 1. Introduction

Molybdenum (Mo) is one of strategically important metals. The dominant share of its consumption is connected with the metallurgy industry itself, since more than 80% of molybdenum is used as an alloying element in steels, cast irons and alloys for increased strength, corrosion resistance and heat resistance; the remaining part is utilized in chemical applications and production of molybdenum compounds. The main industrial mineral source of molybdenum is molybdenite (MoS<sub>2</sub>), although molybdenum is also found in other minerals, for example, in the form of lead and calcium molybdates [1-4].

In many cases, molybdenum ores are characterized by a low content of a useful component, and their extraction is often confined to complex facilities where molybdenum accompanies other metals (such as Cu, W, Pb, Ni and Re). In the industrial practice, molybdenum concentrates are usually obtained by selective flotation from poor ores, including

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Cu-Mo ores, where the flotation scheme provides separation of molybdenum and copper sulfides. Further processing of concentrates is traditionally based on a combination of pyrometallurgical and hydrometallurgical steps such as oxidative roasting of molybdenum to produce a cinder consisting mainly of  $\text{MOO}_3$ , and subsequent ammonia leaching to produce soluble molybdate forms (for example, ammonium molybdate) followed by purification and crystallization [5-9]. The structure of the global molybdenum supply is largely determined by related production: it is estimated that about 50% of the world's molybdenum production is formed at enterprises where it is extracted as an associated product from Cu-Mo ores. This feature enhances the relevance of technological solutions focused on the processing of intermediate and low-grade molybdenum-containing streams while simultaneously controlling the extraction and resource intensity of the process.

In Kazakhstan, molybdenum (Mo) is extracted on an industrial scale mainly as an associated component in the development of copper-porphyry Cu-Mo objects, which corresponds to the global geological and technological pattern. There are no operating specialized molybdenum mines in Kazakhstan, due to the low ore concentration and the complexity of producing conditioned commodity concentrates, or they are processed to produce mainly copper or tungsten, all molybdenum is extracted as a by-product during the development of copper-porphyry deposits [2, 10]. The main active deposits are the copper-molybdenum deposits of Aktogay and Bozshakol, where molybdenum is present in the copper-molybdenum mine. The estimated reserves contain: 115 thousand tons of molybdenum in ores in Aktogay, and 57 thousand tons in Bozshakol. The volume of molybdenum concentrate production is 3.5-4 thousand tons per year, being the 11th largest exporter of molybdenum concentrate in the world. However, further processing in the country is limited due to the lack of production facilities for processing molybdenum concentrate. The resulting molybdenum concentrates are exported abroad. The main buyers of Kazakhstani concentrate are China and Belgium, where molybdenum powder or oxide is obtained. In June 2025, Kazakhstan signed a memorandum with the Chilean Molymet company, the world leader in molybdenum processing, to develop a strategic partnership and joint deep processing of raw materials. The country plans to expand the production of finished molybdenum products, moving away from exporting only raw concentrates.

The importance of resource-saving and phase-controlled firing modes is particularly relevant for countries where molybdenum is mainly associated with large-scale molybdenum production. The increased involvement of poor ores, intermediates and byproducts of molybdenum-containing streams in the processing leads to a steady need for technological solutions that simultaneously ensure a high level of molybdenum extraction and reduce the overall environmental burden of the process. A reduction in the share of free-milling ores, an outstripping increase in mining and conversion costs, stricter regulations on  $\text{SO}_2$  emissions and energy intensity, as well as increased instability of global supply chains actualize the transition to resource-saving, phase-oriented modes of pyrohydrometallurgical processing of molybdenum raw materials, in which targeted management of the phase composition of the product is considered as a key factor of technological efficiency. Therefore, the processing of intermediate flotation products and metallurgical wastes represents a strategically important task for the conservation of refractory mineral reserves and the sustainable management of the raw material base.

## 2. Research Background

Recent studies have highlighted the need for efficient and environmentally compliant routes for processing molybdenum (Mo) concentrates and intermediate products. Industrially, molybdenite ( $\text{MoS}_2$ ) is commonly converted to molybdenum trioxide ( $\text{MoO}_3$ ) by oxidative roasting at elevated temperatures ( $\approx 700$  °C), which requires effective off-gas treatment because of sulfur-bearing emissions [11, 12]. Hydrometallurgical alternatives, including oxidative leaching in acidic or alkaline media and autoclave-based processes, can achieve high Mo recovery; however, they may involve high reagent demand, complex reagent regeneration, and operational challenges related to liquid waste streams, and often require more complex equipment and process control [10, 13-20].

These constraints have stimulated interest in hybrid flowsheets that combine reduced-temperature roasting with subsequent selective leaching [21, 22]. This approach is particularly relevant for lean concentrates and industrial intermediates, where elevated impurity contents (e.g., Cu, Pb, Fe, Zn) and adverse phase evolution during high-temperature roasting can increase sintering propensity and promote the formation of low-solubility molybdates or refractory oxides, thereby limiting downstream leachability. Conversely, purely hydrometallurgical routes remain sensitive to solution chemistry (redox control and stabilization of soluble Mo species) and may generate secondary effluents that require treatment, especially under strongly oxidizing conditions [14-20].

Hydrometallurgical intensification has been explored via oxidative decomposition of  $\text{MoS}_2$  in acidic media coupled with stabilization of soluble molybdenum complexes. In particular, Kenzhaliyev et al. [23] and Safarov et al. [24] reported conditions for oxidative decomposition and stable complexation in nitric-acid-based systems (including mixed-acid schemes), aiming to improve leaching efficiency while reducing environmental burden.

Against this backdrop, low-temperature ( $\leq 550$  °C) phase-selective roasting warrants particular attention. In this temperature window, molybdenum sulfides and impurity-bearing phases may exhibit distinct oxidation and molybdate-formation kinetics, which can be leveraged to improve selectivity and leachability relative to conventional high-temperature roasting [14, 15]. Recent work on pelletized molybdenum concentrates has demonstrated routes to high-purity molybdenum oxide and clarified molybdate-formation pathways during thermal oxidation, providing a mechanistic basis for defining selective operating windows [14, 15]. When integrated with downstream leaching, such operating windows may reduce specific energy demand and moderate sulfur-bearing emissions relative to higher-temperature practice, although the magnitude of these benefits is process- and site-dependent and requires quantitative validation.

Despite progress, important scientific and technological gaps remain. First, the coupled influence of key roasting parameters (temperature, residence time, and bed height) on phase evolution in the calcine, including the  $\text{MoS}_2$  to  $\text{MoO}_3$  conversion, the potential formation of  $\text{MoO}_2$  under local oxygen limitation, and the generation of molybdates (e.g.,  $\text{CaMoO}_4$ ), has not been sufficiently quantified for lean intermediates, particularly with respect to leachability-relevant phase assemblages. Second, many studies report local single-factor trends or pointwise dependencies without translating them into admissible operating windows that can be implemented at the furnace-control level. Third, validated multifactor models that simultaneously link phase transformations to integral extraction metrics and provide operational control maps (e.g., tabular nomograms) remain limited in the available literature. Finally, the transferability of low-temperature regimes to industrial Cu–Mo intermediates with variable mineralogy and impurity profiles has not been demonstrated systematically.

The broader literature covers both process innovations for Mo conversion and methodological tools for process modeling, but it remains fragmented across disciplinary boundaries. For example, Sun et al. [25] propose metaphosphate-quartz-assisted volatilization roasting for producing high-purity  $\text{MoO}_3$  from low-grade concentrates, highlighting the potential of additive-assisted control; however, additive supply, additive-derived impurities, off-gas handling, and scale-up robustness may constrain generalization beyond the studied feed and window. Hydrometallurgical recovery schemes for low-grade molybdenite, such as those discussed by Tripathy & Rakhasia [26], illustrate the feasibility of aqueous routes but may increase chemical consumption and sensitivity to mineralogical variability, complicating industrial deployment without systematic parametric optimization. In parallel, generalized mathematical frameworks for chemical-metallurgical processes [27] and the justification of permissible extrapolation for multiplicative multifactor modeling [28] provide a rigorous basis for compact, decision-oriented models; nonetheless, defensible extrapolation under non-stationary feed characteristics and coupled heat/mass-transfer effects requires explicit validation and clearly bounded domains of applicability. Upstream studies on mining and comminution [29, 30] further indicate that feed preparation and particle-size distributions can materially affect roasting and leaching kinetics, yet these effects are rarely incorporated into end-to-end Mo processing models. Collectively, the evidence supports the need for an integrated, experimentally validated framework that links mineralogical characteristics and phase evolution to multifactor roasting parameters and operational constraints, rather than treating roasting, leaching, and modeling as isolated tasks [25-31].

Accordingly, this study addresses these gaps by developing and validating a compact multifactor model for phase-controlled low-temperature roasting of lean molybdenum-bearing intermediates, explicitly parameterized in terms of  $T$ ,  $\tau$ , and  $h$ , and operationalized as digital tabular nomograms for process control. The framework is evaluated against Mo recovery measured during standardized subsequent alkaline leaching, thereby linking roasting-induced phase evolution to an integral technologically relevant extraction outcome.

### 3. Research Methodology

The aim of this study is to maximize molybdenum recovery during the oxidative roasting of low-grade industrial molybdenum intermediates. This study was designed as a phase-controlled process optimization of oxidative roasting for a low-grade molybdenum-bearing industrial intermediate, with subsequent validation through fixed-condition alkaline leaching. The methodological logic couples-controlled variation of roasting parameters that govern gas-solid reaction and oxygen availability, phase-resolved characterization of roasted products to identify phase boundaries limiting leachability, and multifactor mathematical modeling to translate experimental results into operational control maps suitable for furnace adjustment. This structure provides a consistent progression from the broader context and the identified research gap to a reproducible methodology that can be independently verified and subsequently adapted for industrial implementation.

Figure 1 presents a flowchart of the research methodology.



Figure 1. Structured research workflow outlining the methodological steps

### 3.1. Research Design and Sample Preparation

For the low-temperature roasting study, an industrial molybdenum-bearing intermediate derived from copper-molybdenum porphyry ores mined in the Republic of Kazakhstan was used. Low-grade (lean) intermediate material was selected to reflect industrially relevant feed characteristics for resource-efficient roasting.

Sample mineralogy was examined on polished sections under reflected light using an OLYMPUS BX53 optical microscope equipped with a SIMAGIS XS-3CU digital camera and SIAMS Mineral C7 image-analysis software. Bulk chemical composition (major elements) was quantified by inductively coupled plasma optical emission spectrometry (ICP-OES) using an Avio 500 instrument (PerkinElmer), whereas trace elements and impurity levels were determined by inductively coupled plasma mass spectrometry (ICP-MS).

In the industrial processing route, copper-molybdenum ores are typically upgraded by collective flotation to produce a collective Cu-Mo concentrate, followed by selective flotation to separate copper and molybdenum products. The flowsheet comprises primary collective flotation (rougher and control stages with subsequent cleaning) and selective flotation (rougher stage with multiple cleaning stages and, where applicable, additional flotation of cleaner tailings) [32, 33]. Selective separation of the Cu-Mo concentrate was carried out using sodium sulfide as the depressant under pre-oxidative conditioning, and kerosene (a nonpolar hydrocarbon collector) was applied to enhance molybdenite flotation [10, 33].

### 3.2. Oxidative Roasting

Oxidative roasting of the molybdenum-bearing concentrate was carried out in a muffle furnace (SNOL 15/1200). The experimental design involved varying the roasting temperature ( $T$ ), roasting time ( $\tau$ ), and bed height ( $h$ ) in a crucible to quantify both main and interaction effects. The investigated ranges were  $T = 350\text{-}700\text{ }^{\circ}\text{C}$ ,  $\tau = 15\text{-}120\text{ min}$ , and  $h = 0.004\text{-}0.010\text{ m}$ .

The central (reference) experiment was conducted at a sample mass of 100 g (with 80 wt% of particles passing a 71  $\mu\text{m}$  sieve),  $T = 550\text{ }^{\circ}\text{C}$ ,  $\tau = 90\text{ min}$ , and  $h = 0.004\text{ m}$ . The minimum bed height ( $h = 0.004\text{ m}$ ) was included to map the practical limits of process controllability: at greater bed thicknesses, gas-solid diffusion and heat transfer limitations intensify, which can slow oxidation kinetics and reduce the uniformity of phase transformation across the bed.

The furnace setpoint temperature was maintained throughout each run and monitored using a thermocouple (DEPK 145-0919.1000.1). After roasting, the samples were cooled to ambient temperature prior to downstream processing and analysis.

### 3.3. Alkaline Leaching and Product Recovery

After cooling, the calcine was crushed and homogenized. Alkaline leaching was performed in a thermostatically controlled reactor equipped with mechanical stirring to extract soluble molybdenum into solution. The leaching conditions were  $T = 90\text{ }^{\circ}\text{C}$ , liquid-to-solid ratio  $L/S = 10:1$ , and  $\tau = 120\text{ min}$ , using a 10 wt% sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) solution (analytical grade). The pregnant leach solution was subsequently processed to obtain a marketable molybdenum product, following the procedure described in previous literature [13].

### 3.4. Model Construction and Nomogram Development

The experiments were planned and processed using the previously described approach [27] to construct a Protodyakonov-Malyshev multiplicative multifactor model (PMMM). In this framework, partial response functions are first derived for each factor and then normalized and combined into a single multiplicative relationship. In the present study, normalization was anchored to the response obtained in the central experiment ( $y_{(c)}$ ), which was explicitly incorporated into each partial dependency. This choice improves comparability among factor effects and stabilizes the model within the experimentally investigated ranges. Based on the PMMM, multifactor tabular nomograms were constructed to delineate admissible and non-admissible combinations of operating parameters for process control [27].

### 3.5. Statistical Analysis

Model adequacy was assessed using the nonlinear multiple correlation coefficient ( $R$ ) and the corresponding significance statistic ( $tR$ ) as recommended in Malyshev [27] and Karimova et al. [28]. Additional details on the model outputs and their interpretation are provided in the Results section.

## 4. Results

This section reports feedstock characterization, including bulk chemical composition (ICP-OES) and mineralogical observations (reflected-light microscopy); phase evolution in the calcine, with emphasis on the  $\text{MoO}_3/\text{MoO}_2$  ratio and the formation of  $\text{CaMoO}_4$  and impurity-associated molybdates (Cu/Fe/Pb/Zn); performance of the Protodyakonov-Malyshev multiplicative multifactor model (PMMM), including model adequacy and statistical significance assessed using the nonlinear multiple correlation coefficient ( $R$ ) and its associated test statistic ( $tR$ ); and the resulting tabular digital nomogram defining admissible operating combinations for process control and decision support.

### 4.1. Characterization of the Raw Materials

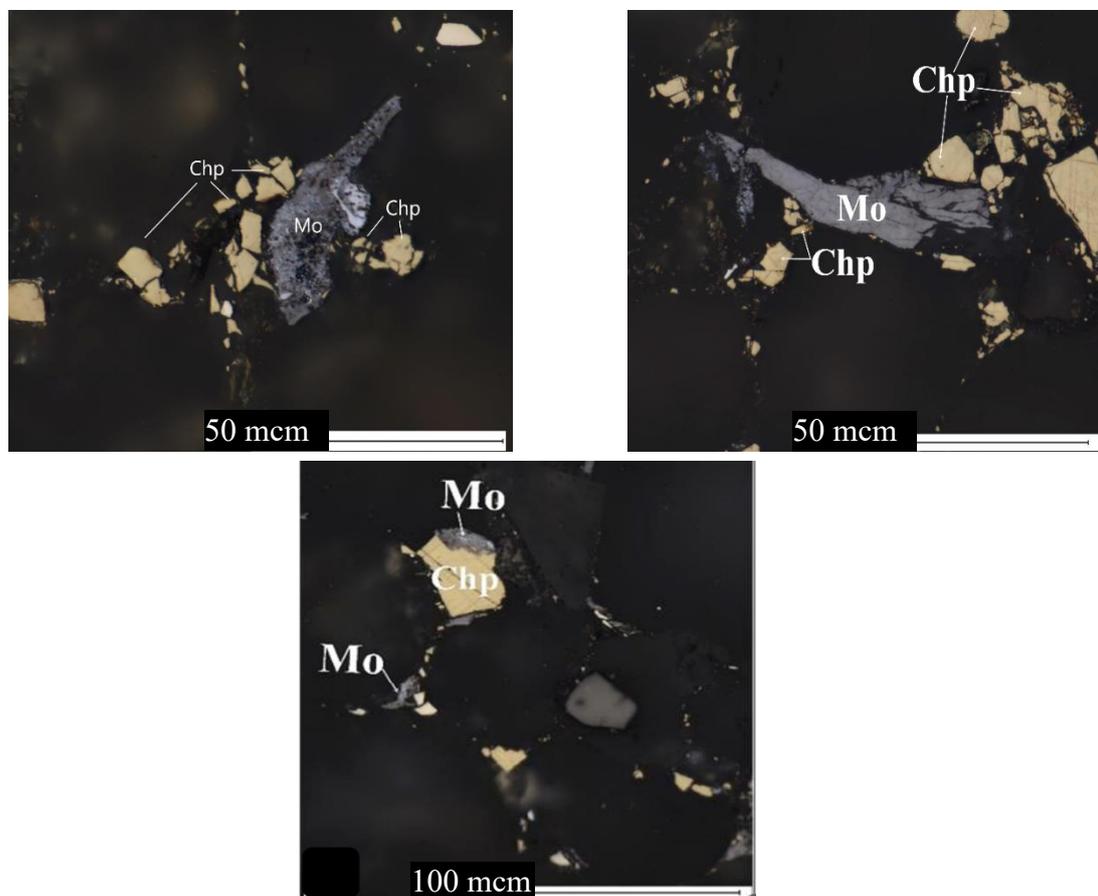
Following the selective flotation of the collective Cu-Mo concentrate under the proposed process flowsheet and reagent regime, an industrial molybdenum-bearing intermediate was obtained with a mass yield of 0.07%, a Mo grade of 22.23 wt%, and a Mo recovery of 74.91% (relative to the Mo in the flotation feed) [10]. The bulk chemical composition of the sample used in the subsequent roasting and leaching experiments is summarized in Table 1.

**Table 1. Bulk chemical composition of the industrial molybdenum-bearing intermediate used in this study**

| Components                        | Content, %  | Components         | Content, %        |
|-----------------------------------|-------------|--------------------|-------------------|
| Mo                                | 22.23       | As                 | 0.0180            |
| Cu                                | 7.516       | Co                 | 0.0278            |
| Au, g/t                           | 10.6        | P                  | 0.12              |
| Ag, g/t                           | 167.0       | Cd                 | < detection limit |
| Fe                                | 16.54       | Re, g/t            | 62.3              |
| Zn                                | 0.049       | Bi                 | < detection limit |
| Pb                                | 0.053       | Ni                 | 0.0143            |
| Al/Al <sub>2</sub> O <sub>3</sub> | 1.126/2.128 | Ca/CaO             | 1.155/1.617       |
| Si/SiO <sub>2</sub>               | 5.33/11.422 | C                  | 9.57              |
| Mg/MgO                            | 0.741/1.230 | S <sub>total</sub> | 27.85             |

The main valuable components were as follows: molybdenum (22.23%), copper (7.516%), gold (10.6 g/t), and silver (167.0 g/t). With the mineralogical study of the molybdenum industrial product, the approximate ratio of ore and NMMs was visually established as 80:20%. The following minerals represent ore mineralization: molybdenite (40%), chalcopyrite (22%), magnetite, hematite, iron hydroxides (10% in total), pyrite (10%), rare covellite, and isolated chalcosine and bornite. All ore minerals are in an open state, free of accretions, with molybdenite being the most frequently accreted with chalcopyrite. The predominant grain sizes of Mo are 0.003-0.5 mm, and chalcopyrite are 0.003-0.06 mm.

The molybdenum deposits are finely scaled in the form of lamellar, tabular, leafy, sometimes deformed, and rarely barrel-shaped shapes. Nearly all molybdenum (80% of the total content) is in a splice-free state, with an elongated grain size of 0.003-0.5 mm. Approximately 20% of the Mo grains are found in accretions with other ore minerals, most often with chalcopyrite, and very rarely with hematite, pyrite, and non-metallic minerals. These grains form simple and complex accretions, predominantly in the binary form, occasionally postmineral aggregates with the above minerals (Figure 2). Chalcopyrite is in a splice-free state. The predominant grain size of chalcopyrite is in the range of 0.003-0.06 mm. Approximately 20% of the total chalcopyrite content in the concentrate forms close associations with molybdenite and hematite, less often with pyrite and magnetite, and occasionally with non-metallic minerals, forming simple and complex intergrowths. Chalcopyrite is rarely susceptible to varying covellin substitution.

**Figure 2. Characteristics of molybdenum precipitates (Mo – molybdenum, Chp – chalcopyrite); scale = 500**

#### 4.2. Effect of Oxidative Roasting Parameters on Mo Recovery

Oxidative roasting of molybdenum sulfide concentrates converts sulfide phases into oxide and molybdate species that are readily leachable under alkaline conditions, thereby enabling high Mo recovery into solution. Table 2 summarizes the experimental roasting conditions and the corresponding Mo recovery values obtained after standardized alkaline leaching.

**Table 2. Roasting conditions and Mo recovery into solution (%) after alkaline leaching**

| Studied factor, experimental conditions                                 | Cake output (%) | Mo content in the cake (%) | Experimental data on the extraction into the solution ( $\epsilon$ ), % | Extraction using the generalized equation (5), % |
|---|-----------------|----------------------------|---|--|
| $t, ^\circ\text{C}$<br>( $\tau = 90 \text{ min}, h = 0.004 \text{ m}$ ) | 350             | 86                         | 15.58   | 39.73  |
|   | 400             | 71                         | 9.94  | 68.27  |
|   | 500             | 64                         | 1.64  | 95.28  |
|   | 550             | 67                         | 1.07  | 96.78  |
|   | 600             | 64                         | 1.12  | 96.78  |
|   | 700             | 63                         | 1.46  | 95.86  |
| $\tau, \text{min}$<br>( $t = 550 ^\circ\text{C}, h = 0.004 \text{ m}$ ) | 15              | 93                         | 13.30   | 44.34  |
|   | 30              | 86                         | 11.52   | 55.42  |
|   | 45              | 73                         | 6.60  | 78.33  |
|   | 60              | 66                         | 2.70  | 91.98  |
|   | 90              | 67                         | 1.07  | 96.78  |
|   | 120             | 65                         | 1.0   | 97.08  |
| $h, \text{m}$<br>( $t = 550 ^\circ\text{C}, \tau = 90 \text{ min}$ )    | 0,004           | 67                         | 1.07  | 96.78  |
|   | 0,006           | 72                         | 2.74  | 91.13  |
|   | 0,01            | 81                         | 3.65  | 86.70  |

#### 4.3. Phase Composition of the Calcine

A targeted phase analysis was performed to assess the abundance of oxidized molybdenum species in the calcine as a function of roasting temperature and to evaluate the degree of oxidation achieved during roasting. The results are summarized in Table 3.

**Table 3. Molybdenum-bearing phases in the calcine as a function of roasting temperature**

| Temperature, $^\circ\text{C}$ | Mo content (%)           |                              |        |       |        |          |         |                        |       |          |      |
|-------------------------------|--------------------------|------------------------------|--------|-------|--------|----------|---------|------------------------|-------|----------|------|
|                               | Mo content in the cinder | Oxidized minerals, including |        |       |        | Sulphide |         | Me(MoO <sub>n</sub> )* |       | Total, % |      |
|                               |                          | abs, %                       | abs.   | rel.  | abs.   | rel.     | abs.    | rel.                   | abs.  | rel.     | abs. |
| 350                           | 22.11                    | 6.725                        | 30.416 | 0.261 | 1.1805 | 15.124   | 68.403  | 0                      | 0     | 22.11    | 100  |
| 400                           | 22.00                    | 13.247                       | 60.241 | 0.555 | 2.5239 | 8.189    | 37.2397 | 0                      | 0     | 22.00    | 100  |
| 500                           | 21.96                    | 19.299                       | 87.875 | 1.243 | 5.6598 | 1.200    | 5.46398 | 0.22                   | 1.002 | 21.96    | 100  |
| 550                           | 22.00                    | 19.422                       | 88.282 | 1.478 | 6.718  | 0.760    | 3.4545  | 0.34                   | 1.545 | 22.00    | 100  |
| 600                           | 21.96                    | 19.290                       | 87.842 | 1.411 | 6.425  | 0.890    | 4.0528  | 0.37                   | 1.685 | 21.96    | 100  |
| 700                           | 22.00                    | 18.516                       | 84.164 | 2.860 | 13.000 | 0.095    | 0.4318  | 0.53                   | 2.409 | 22.00    | 100  |

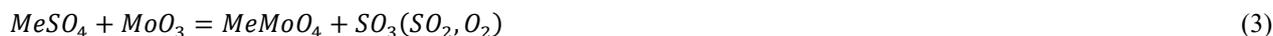
\* Me (metal) can be Cu, Fe, Zn, and Pb

Analysis of the data demonstrated that the content of oxidized molybdenum minerals increased with an increase in the roasting temperature from 350  $^\circ\text{C}$  to 550  $^\circ\text{C}$ : for MoO<sub>3</sub> from 30.416% to 88.282%, and for CaMoO<sub>4</sub> from 1.18% to 6.718%. With a further increase in the roasting temperature to 700  $^\circ\text{C}$  and a lack of oxygen, molybdenum dioxide is formed in the cinder. Because molybdenum dioxide is practically insoluble in an alkaline medium, roasting at a temperature of no more than 600  $^\circ\text{C}$  is necessary to avoid sintering of the material and the formation of insoluble MoO<sub>2</sub>.

Previous studies have reported that, during oxidative roasting of molybdenum concentrates at temperatures above ~500  $^\circ\text{C}$ , molybdenum disulfide (MoS<sub>2</sub>) is readily oxidized by oxygen in air to molybdenum trioxide (MoO<sub>3</sub>) via an overall exothermic reaction [31]:



During the oxidation process, the molybdenum particles are coated with resulting molybdenum trioxide. In addition, copper, iron, zinc, and lead sulphide minerals turn into oxides, partly into sulphates, during the oxidative roasting of molybdenum industrial products, which actively react with molybdenum trioxide in the temperature range of 500-700 °C, forming molybdates:



Reaction (1) indicates that oxidative roasting of molybdenum concentrate is exothermic and releases heat. In practice, this heat release can partially offset the external heat input required to sustain the process once the reaction is initiated, thereby potentially reducing specific energy demand under appropriate operating conditions.

The experimental results indicate that the optimum roasting temperature was 550 °C, which is consistent with previously reported values [13, 14, 31]. Figure 3 presents scatter plots with fitted curves illustrating the partial (single-factor) response relationships derived from the dataset summarized in Table 3.

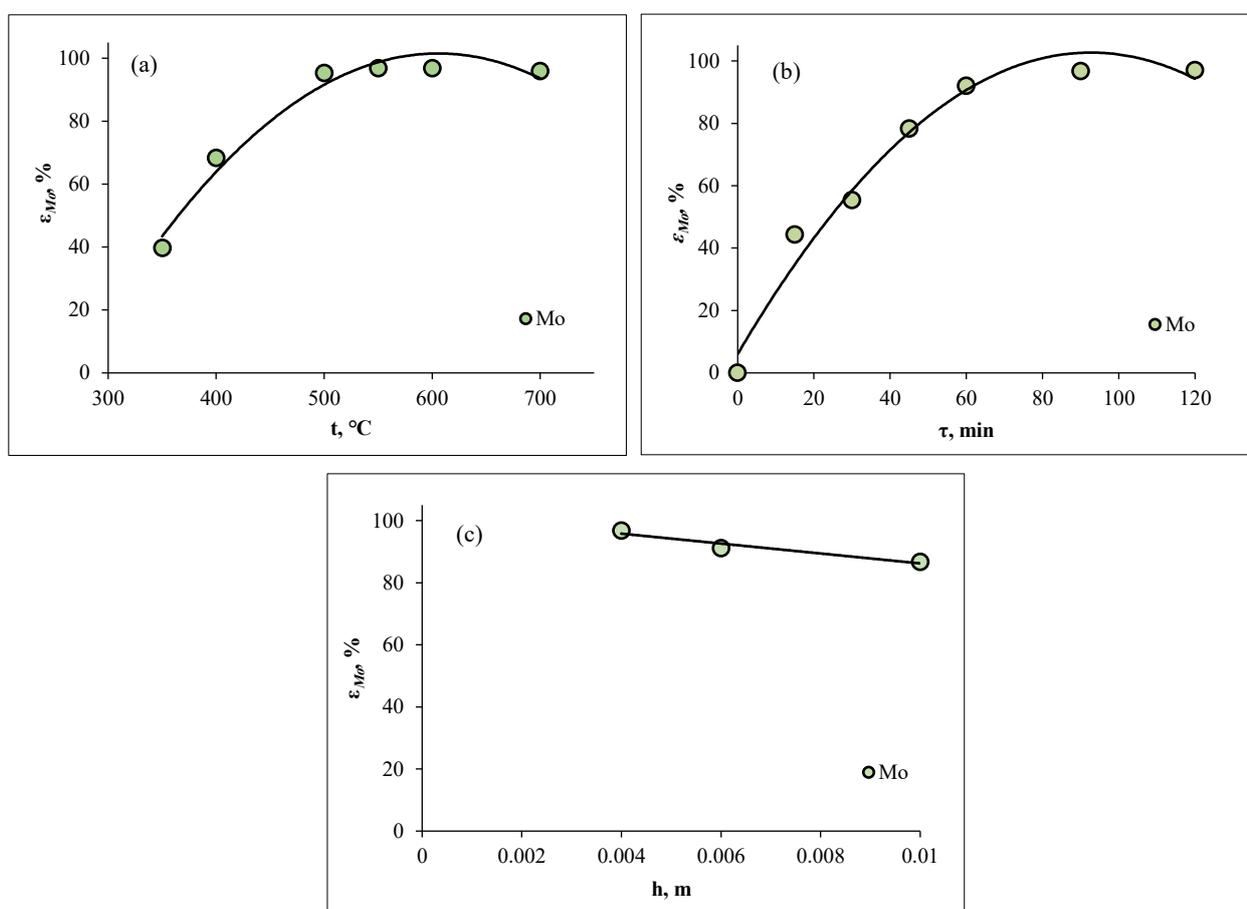


Figure 3. Effect of key process parameters on Mo recovery into solution: a) roasting temperature (°C); b) roasting duration (min); c) bed height (m). The central experiment was conducted at T = 550 °C and τ = 90 min

#### 4.4. Structure

The results shown in Figure 3 indicate that, within the investigated range, Mo recovery into solution increases with roasting temperature and reaches a maximum at 550 °C, which is consistent with previously reported values. As shown in Table 3, roasting duration has a pronounced effect on Mo recovery after subsequent alkaline leaching. Increasing the roasting time from 15 min to 90 min raised Mo recovery from 27.65% to 96.69%, representing the optimum observed in this experimental series. Partial response functions describing Mo recovery were derived from the experimental dataset (Table 4). These partial dependencies, including only statistically significant functional terms, were then combined into a Protodyakonov-Malyshev multiplicative multifactor model by multiplying the normalized partial functions; normalization was performed using the arithmetic mean experimental value for each partial function to ensure comparability across factors.

**Table 4. Partial functions of molybdenum extraction into solution with determination of the coefficient of nonlinear multiple correlation R and its significance  $t_R$** 

| Functions   | R      | $t_R > 2$ | $R^2$  |
|---|--------|-----------|--------|
| $\varepsilon = -0.0009 t^2 + 1.077t + 224.58$     | 0.966  | 28.883    | 0.9332 |
| $\varepsilon = -0.0113\tau^2 + 2.085\tau + 6.039$ | 0.9691 | 31.816    | 0.9392 |
| $\varepsilon = -1597.3 h + 102.18$                | 0.8719 | 6.8114    | 0.7602 |

The multifactor equation for the extraction of Mo into solution includes significant partial dependence on temperature, duration, and the ratio of alkali to concentrate, normalized according to the central calculated value [27]. Since the partial functions (Table 4) in the multifactor equation are combined as a product, the geometric average should be used to calculate the total average value. The generalized equation for the extraction of Mo into solution is expressed as follows:

$$\varepsilon_{Mo} = 1.068 \cdot 10^{-4} (-9 \cdot 10^{-4} \cdot t^2 + 1.077 \cdot t - 224.58) (2.085 \cdot \tau - 0.0113 \cdot \tau^2 + 6.039) (102.18 - 1597.3 \cdot h) \quad (5)$$

The nonlinear multiple correlation coefficient ( $R = 0.9742$ ) indicates a strong agreement between the model predictions and the experimental data. Model significance was supported by the corresponding test statistic ( $t_R = 68.9445$ ), which exceeded the critical value at the selected significance level [27, 28].

An additional confirmation run performed for the low-grade molybdenum concentrate at  $T = 550$  °C and  $\tau = 90$  min further demonstrated the practical feasibility of the proposed strategy and its compatibility with the subsequent hydrometallurgical stages (Table 5).

The resulting equations can be used to calculate Mo recovery across the three-factor domain within technologically feasible ranges of the significant parameters. Table 5 presents the tabulated three-factor response values and can be used as an operational decision-support map for selecting roasting conditions and anticipating Mo recovery.

**Table 5. Mo recovery into solution (%) as a function of roasting temperature, time, and bed height**

| $t, ^\circ\text{C}$ | $\tau, \text{min}$ |  | 15    | 30    | 45    | 60    | 90    | 120   |
|---------------------|--------------------|--|-------|-------|-------|-------|-------|-------|
|                     | $h, \text{m}$      |  |       |       |       |       |       |       |
| 350                 | 0.004              |  | 14.95 | 25.11 | 33.09 | 38.89 | 42.77 | 40.21 |
|                     | 0.006              |  | 13.97 | 23.46 | 30.92 | 36.33 | 39.96 | 37.57 |
|                     | 0.01               |  | 12.97 | 21.79 | 28.71 | 33.74 | 37.11 | 34.89 |
| 400                 | 0.004              |  | 22.09 | 37.11 | 48.91 | 57.47 | 63.21 | 59.43 |
|                     | 0.006              |  | 20.64 | 34.67 | 45.69 | 53.69 | 59.05 | 55.52 |
|                     | 0.01               |  | 19.16 | 32.20 | 42.43 | 49.86 | 54.84 | 51.56 |
| 500                 | 0.004              |  | 31.57 | 53.05 | 69.90 | 82.14 | 90.34 | 84.94 |
|                     | 0.006              |  | 29.50 | 49.56 | 65.31 | 76.75 | 84.41 | 79.36 |
|                     | 0.01               |  | 27.39 | 46.02 | 60.65 | 71.27 | 78.38 | 73.69 |
| 550                 | 0.004              |  | 33.91 | 56.98 | 75.09 | 88.23 | 97.04 | 91.23 |
|                     | 0.006              |  | 31.68 | 53.23 | 70.15 | 82.43 | 90.66 | 85.24 |
|                     | 0.01               |  | 29.42 | 49.43 | 65.14 | 76.55 | 84.19 | 79.15 |
| 600                 | 0.004              |  | 34.65 | 58.22 | 76.72 | 90.16 | 99.16 | 93.22 |
|                     | 0.006              |  | 32.38 | 54.40 | 71.68 | 84.23 | 92.64 | 87.10 |
|                     | 0.01               |  | 30.06 | 50.51 | 66.56 | 78.22 | 86.03 | 80.88 |
| 700                 | 0.004              |  | 31.33 | 52.64 | 69.37 | 81.51 | 89.65 | 84.29 |
|                     | 0.006              |  | 29.27 | 49.18 | 64.81 | 76.16 | 83.76 | 78.75 |
|                     | 0.01               |  | 27.18 | 45.67 | 60.18 | 70.72 | 77.78 | 73.13 |

Table 5 shows a clear and internally consistent response pattern: Mo recovery increases with roasting temperature up to approximately 600 °C, rises with residence time up to 90 min, and decreases monotonically as bed height increases from 0.004 to 0.010 m. The maximum recovery within the tabulated three-factor domain is 99.16% at  $T = 600$  °C,  $\tau = 90$  min, and  $h = 0.004$  m, while a comparably high value (97.04%) is obtained at  $T = 550$  °C,  $\tau = 90$  min, and  $h = 0.004$  m. In contrast, extending roasting to 120 min generally lowers recovery across temperatures and bed heights, indicating that prolonged residence time is unfavorable under the investigated conditions. Consequently, Table 5 functions as an operational control map that delineates a practical high-recovery window around  $T = 550\text{-}600$  °C,  $\tau \approx 90$  min, and  $h \leq 0.006$  m, supporting informed parameter selection within technologically feasible limits.

From a mechanistic standpoint, roasting near 550 °C is thermodynamically favorable for molybdenite oxidation, with negative Gibbs free energy changes reported for the governing reactions; under these conditions, MoS<sub>2</sub> typically undergoes sequential oxidation via MoO<sub>2</sub> to MoO<sub>3</sub> [34]. Relative to higher-temperature operation (e.g., ~650 °C), sulfur oxidation at ~550 °C proceeds more gradually, which can mitigate peak SO<sub>2</sub> release rates and thereby reduce transient loading of gas-cleaning systems. At the same time, depending on feed composition and process additives, part of sulfur may be retained temporarily in solid intermediates (e.g., sulfate-bearing phases) or transferred to downstream aqueous streams during leaching, while maintaining high Mo recovery as oxidized species [22]. Operation at elevated temperatures can accelerate sulfur volatilization as SO<sub>2</sub> and increase instantaneous concentrations in off-gases, potentially tightening requirements for gas-treatment performance.

Under the selected optimum conditions, a pregnant leach solution suitable for subsequent calcium molybdate production was obtained [26, 31]. Solution purification was performed by neutralization with HCl to pH 7.0-7.5 followed by boiling for 30 min, which promotes CO<sub>2</sub> stripping from carbonate species, destabilizes copper carbonate complexes, hydrolyzes sodium silicate with formation of silicic acid (silica gel), and coagulates colloidal iron-containing impurities, thereby improving solution quality for downstream precipitation. Overall, integrating oxidative roasting with alkaline leaching provides a pathway toward more complete utilization of low-grade molybdenum concentrates; further work should focus on refining the operating window under feed variability, quantifying energy and emission trade-offs, and validating the process at scale to support robust industrial implementation.

## 5. Discussion

Our study was conducted to obtain a commercial product, calcium molybdate, from a low-grade molybdenum concentrate processed by hydrometallurgical methods from Cu-Mo ore. This section interprets its essential results.

Compared to similar studies, Mo is extracted from intermediates obtained during flotation enrichment, using low-temperature roasting followed by leaching to obtain calcium molybdate. We obtained higher Mo recovery rates (97%) from the depleted concentrate, while confirming the general patterns of Mo product roasting at 550 °C.

The optimum temperature to obtain pure MoO<sub>3</sub> and stabilize ash was 550-650 °C [14, 15, 21, 22], which provided the maximum Mo recovery and high MoO<sub>3</sub> content. During oxidative roasting, a series of successive reactions of sulphide minerals and interactions between concentrate components occur. Initially, molybdenum sulphide is oxidized, and molybdenum oxide and molybdenum trioxide are formed at specific temperatures. A repeated reaction between molybdenum oxide and molybdenite occurs during the oxidation process. The resulting molybdenum trioxide reacts with impurities in the concentrate to form molybdates [5]. Previously, some scientists have established [7, 31] that at temperatures below 400 °C, MoO<sub>2</sub> is formed during the oxidation of molybdenum sulphide; however, at 400-600 °C, MoO<sub>3</sub> is formed during the roasting process, bypassing the stage of MoO<sub>2</sub> formation and interacting with oxygen. However, a thin layer of molybdenum dioxide is present on the surface of the molybdenite because the secondary interaction of MoS<sub>2</sub> with MoO<sub>3</sub> occurs at 600 °C [32]. The oxidation rate of MoS<sub>2</sub> and the phase composition of the cinder are temperature-dependent. At various temperatures, molybdenum oxides can form particles on the surface of molybdenum disulphide, and these particles can significantly reduce the reaction rate. During the roasting process, the sulphide minerals copper, lead, iron, and zinc present in the concentrate as impurities form oxides and sulphates [32, 33, 35]. The transition of calcium carbonate to sulphate is possible under certain roasting conditions. In the temperature range of 500-600 °C, molybdenum trioxide reacts with oxides, carbonates, or sulphates of various elements, which leads to the formation of molybdates [35].

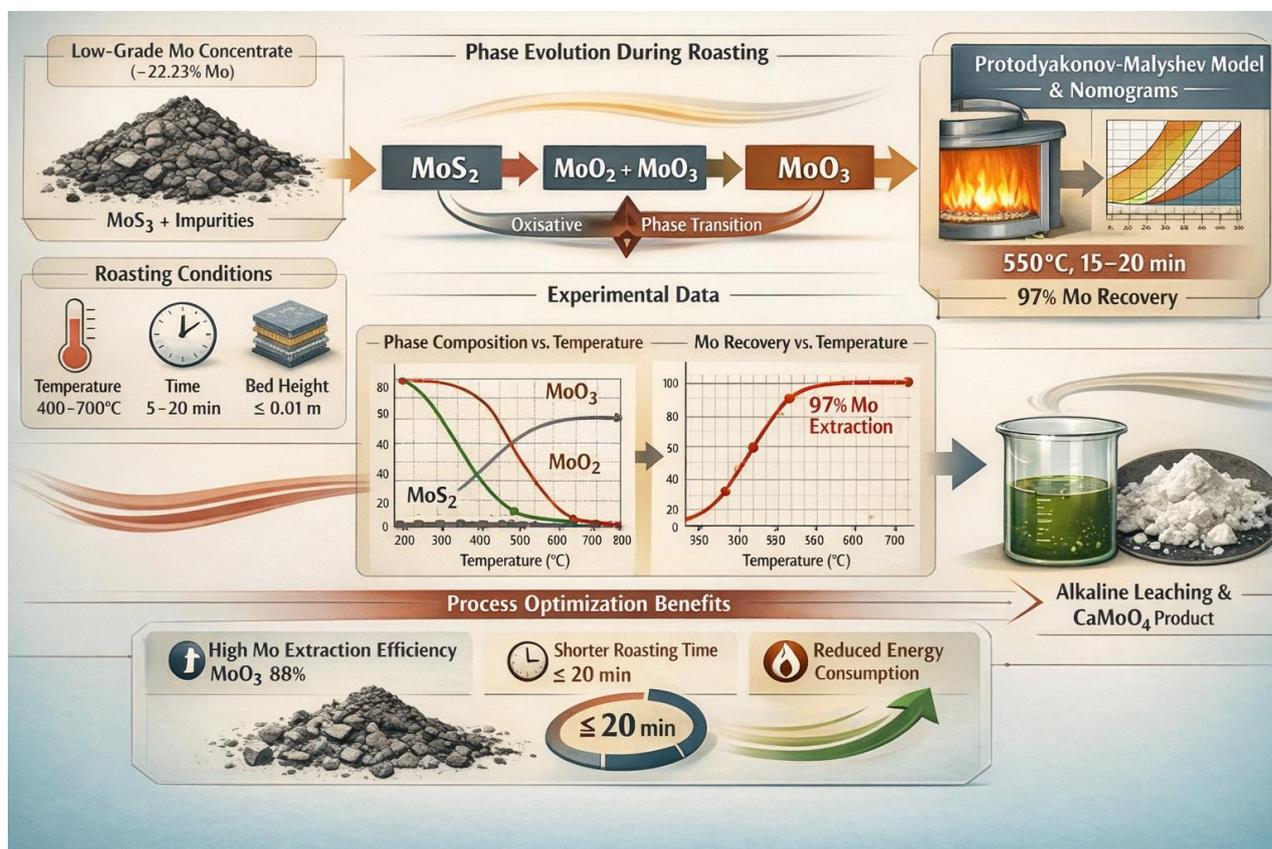
In industrial practice, pure molybdenum trioxide is often obtained by hydrometallurgical methods. The cinder was treated with an ammonia solution, from which ammonium paramolybdate tetrahydrate (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O was isolated. However, the degree of Mo transition into the solution directly depends on the cinder composition; compounds such as calcium molybdates, molybdenum dioxide, and molybdenite are poorly soluble in ammonia water, and Mo is lost [36]. The maximum degree of Mo extraction from the cinder reached 85% [13, 15, 36].

When processing the obtained experimental data, we used mathematical planning of the experiment and prepared experimental plans. There are different types of composite plans: three-level Box plans (3k), Box-Wilson plans, Box-Hunter plans, and Kono plans. These plans help find the regression equation in the following family of polynomials, for example, for coded values of input factors [26].

Box plans have the advantage of high accuracy in determining the effects of the factors. Disadvantages of the Box plan include the large number of experiments. Considering the advantages and disadvantages of the Box-Wilson method of "the fastest ascent to the region of a multifactor optimum," which uses exclusively a linear version of the regression equation, a modification of the Protodyakonov equation is proposed in the form of partial point dependencies. They are obtained with passive planning of an experiment using point dependencies, which are random and directly follow from partial algebraic dependencies without physical limitation. Based on this, V.P. Malyshev proposed a data processing method that considers the physical meaning of the partial dependencies. In this regard, for a consistent study of the active

factors, studies on the roasting of Mo intermediates were performed using the Protodyakonov-Malyshev method to obtain a multiplicative multifactor model [35]. We established optimal intervals for the low-temperature roasting parameters of Mo intermediates using multifactor models that ensure high rates of Mo extraction during subsequent leaching (Figure 4).

**Figure 4. Phase-oriented optimization scheme for oxidative roasting of low-grade molybdenum intermediates using the Protodyakonov-Malyshev model**



The Protodyakonov-Malyshev model can be applied to the analysis of a broad class of molybdenum-bearing intermediate products, provided that the feed materials are mineralogically and chemically comparable and that the model is properly parameterized using experimental data. A more rigorous procedure for constructing the multifactor relationship improves the quality of approximation and the stability of parameter estimates through explicit consideration of factor interactions and statistical verification of model adequacy thereby expanding the justified domain of applicability of the equation and enhancing the reliability of predictions in the vicinity of the investigated operating conditions. Extrapolation to substantially unexplored regions of the factor space requires separate justification, including validation against independent data, residual analysis, and the assessment of confidence intervals.

## 6. Conclusion

The study met its objective by developing a phase-controlled approach to optimizing the oxidative roasting of low-grade molybdenum intermediates and translating experimental findings into an operational decision-support format (tabular nomograms) based on the Protodyakonov-Malyshev multiplicative multifactor model.

The main findings can be summarized as follows. First, mineralogical and chemical characterization established the initial phase assemblage and element associations, providing the baseline required to interpret roasting-induced phase transformations and to define the scope of applicability to compositionally similar intermediates. Second, multifactor roasting experiments systematically varied temperature, residence time, and bed height, enabling the evaluation of both individual factor effects and their interactions on phase evolution and downstream leachability. Third, molybdenum recovery was quantified under fixed alkaline leaching conditions using a standardized response metric at the process outlet. This allowed phase composition after roasting to be directly linked to the technologically relevant outcome, namely the transfer of Mo into solution. Fourth, the experimental dataset was used to derive empirical response relationships, and model adequacy was supported by statistical tests and diagnostics reported in the Results section. On this basis, tabular nomograms were constructed to define the operating envelope (admissible combinations of temperature, time, and bed height) and to reduce uncertainty in selecting roasting conditions for routine operation.

Process feasibility was further demonstrated by converting the leach solution into a marketable product form, supporting compatibility of the proposed roasting strategy with subsequent hydrometallurgical steps. Operating within a phase-favorable region is expected to reduce process risk and stabilize calcine quality for leaching, thereby supporting resource-efficient operation.

Overall, the study reframes roasting-mode selection from point optimization toward operating-window control grounded in phase analysis and multifactor formalization. Further validation across broader feed variability and scale-up conditions would strengthen the industrial generalizability of the proposed control maps [37-43].

### 6.1. Theoretical Novelty

This study advances a phase-oriented, multifactor framework for optimizing the oxidative roasting of low-grade molybdenum intermediates through an adaptation of the Protodyakonov-Malyshev methodology. A tabular (digital) nomogram was developed to represent model outputs across the experimentally investigated ranges of roasting temperature, residence time, and bed height. This nomogram provides a transparent, implementation-ready decision-support tool for estimating Mo recovery and delineating admissible operating windows within technologically feasible factor limits.

The proposed approach explicitly captures the joint influence of significant parameters and their interactions, enabling systematic selection of roasting conditions rather than reliance on one-factor trends. Under the studied conditions, operation within the identified high-recovery window achieved up to 97% Mo recovery during subsequent alkaline leaching, supporting the practical relevance of an optimized regime centered around 550 °C for the investigated feed material.

### 6.2. Practical Significance

The proposed processing route is practically relevant for copper-molybdenum porphyry operations in Kazakhstan, where molybdenum is commonly recovered as a by-product and intermediate materials may exhibit variable grade and mineralogical composition. By combining selective flotation with phase-controlled oxidative roasting and subsequent alkaline leaching, the approach provides a structured pathway for converting low-grade molybdenum-bearing intermediates into leachable oxidized forms and for stabilizing downstream hydrometallurgical performance. A key practical outcome supports consistently high Mo recovery while remaining compatible with industrial furnace constraints. In particular, the tabular (digital) nomograms derived from the multifactor model enable rapid selection of roasting conditions across temperature, residence time, and bed height, thereby reducing reliance on trial-and-error adjustments and facilitating routine process control. For the investigated material, the high-recovery domain is centered around 600 °C,  $\tau \approx 90$  min, and  $h \leq 0.006$  m, which is directly applicable to industrial-scale setting of roasting regimes and monitoring of bed geometry. The resulting pregnant leach solutions can be integrated with established purification and precipitation steps to obtain marketable molybdenum products, including calcium molybdate ( $\text{CaMoO}_4$ ), supporting the development of practical process regulations and the transfer of the proposed strategy to pilot and plant trials.

### 6.3. Limitations and Further Research Directions

Several limitations define the scope of applicability of the findings. First, the experiments were performed on a specific Cu-Mo-derived intermediate within a limited compositional and mineralogical range; therefore, direct transfer of the results to feedstocks with substantially different impurity suites, textures, or phase assemblages should be treated with caution. Second, the experimental basis is laboratory-scale roasting with controlled variation of temperature, residence time, and bed height. Industrial continuous units may exhibit additional thermal and gas-flow non-uniformities (e.g., temperature gradients, gas-dynamic dispersion, and residence-time distributions) that are not explicitly captured by the present formulation. Third, the liquid-solid stage was intentionally standardized to ensure comparability across roasting series; consequently, the derived relationships should be interpreted as “roasting – leachability” dependencies under fixed leaching conditions rather than as an optimization of the leaching chemistry. Fourth, potential energy and environmental implications of the low-temperature strategy were discussed conceptually but were not quantified through direct industrial measurements; robust assessment requires dedicated instrumentation and plant-scale data acquisition.

Methodological limitations should also be noted. The model employs a multiplicative composition of normalized partial response functions, which is convenient for implementation and interpretation; however, alternative specifications may describe edge regions of the factor space differently. In addition, phase analysis focused on the dominant Mo-bearing forms governing leachability; minor or transient phases may not have been fully resolved and could influence local behavior. These constraints do not invalidate the conclusions but delineate the conditions under which the results can be applied: the proposed operating windows and control maps should be validated and, if necessary, recalibrated for a given facility, accounting for feed variability and equipment-specific features.

Future work should aim to strengthen generalizability and industrial transferability. Priority directions include: testing robustness on an expanded set of raw materials covering broader impurity profiles, particle morphologies, and mineralogical variants; extending geometric and transport considerations by incorporating additional bed configurations and explicitly assessing heat- and mass-transfer limitations; and characterizing the gas environment and spatial distributions in continuous installations, followed by systematic comparison of laboratory results with pilot-scale trials to clarify parameter transferability. From a modeling perspective, comparative evaluation of the multiplicative scheme against alternative regression or mechanistic formulations, supported by expanded experimental designs and deeper statistical verification, would be valuable. Finally, integrating phase analysis with in situ observations and thermodynamic calculations would strengthen causal interpretation, while plant-scale quantification of energy use, emissions, and economic performance is needed to substantiate sustainability claims for the selected operating regimes.

## 7. Declarations

### 7.1. Author Contributions

Conceptualization, T.O.; methodology, L.K.; software, G.M.; validation, A.K.; formal analysis, I.T.; investigation, L.K.; resources, T.O.; data curation, T.T.; writing—original draft preparation, L.K.; writing—review and editing, I.T.; visualization, T.T. and G.M.; supervision, T.O.; project administration, A.K. All authors have read and agreed to the published version of the manuscript.

### 7.2. Data Availability Statement

The data presented in this study are available in the article.

### 7.3. Funding

The authors received no financial support for the research, authorship, and/or publication of this article.

### 7.4. Institutional Review Board Statement

Not applicable.

### 7.5. Informed Consent Statement

Not applicable.

### 7.6. Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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