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EXPERIMENTAL STUDY ON THE REMOVAL OF BISMUTH FROM LEAD WITH CALCIUM MAGNESIUM

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Abstract. The separation of lead from the bismuth mixture remains a serious problem, and achieving effective separation is an important obstacle to the production of high-purity lead using the vacuum gasification method. This research focuses on lead as a core research discipline, conducting theoretical and experimental studies on the secondary conversion of lead through vacuum gasification. Gibbs free energy calculations show that in the temperature range from 600 to 610 K, the bismuth compound completely reacts with calcium and magnesium, resulting in the formation of the CaMg2Bi2 compound. Under optimal experimental conditions, the bismuth compound is converted to CaMg2Bi2 BiCa2. It should be noted that BiCa2 does not fly and remains in the crucible as a residue. Auxiliary calcium is completely converted into CaSe and CaTe, which leads to a decrease in the calcium content in volatile substances from 0.5% to 16 ppm. Similarly, the magnesium content in volatile substances is reduced from 0.66% to 187 ppm. Eventually, the bismuth content in the final product decreases from 6 ppm to 1.4 ppm, reaching a removal rate of 76.6%, and the direct yield of metallic lead reaches 71%. This process effectively facilitates the separation of metallic lead from bismuth impurities.
Keywords: vacuum separation of bismuth from lead, adjuvant transformation, vacuum gasification, high-temperature dissociation, separation and purification.

КАЛЬЦИЙ МАГНИЙІМЕН ҚОРҒАСЫННАН ВИСМУТТЫ ЖОЮ БОЙЫНША ЭКСПЕРИМЕНТТІК ЗЕРТТЕУ

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Аңдатпа. Қорғасынды висмут қоспасынан бөлу маңызды мәселе болып қала береді, тиімді бөлуге қол жеткізу вакуумды газдандыру әдісі арқылы жоғары таза қорғасынды өндіруде маңызды кедергі болып табылады. Бұл зерттеу зерттеудің негізгі пәні ретінде қорғасынға назар аударады, қорғасынды вакуумды газдандыру арқылы қосалқы түрлендіру бойынша теориялық және эксперименттік зерттеулер жүргізеді. Гиббс бос энергиясының есептеулері көрсеткендей, 600-ден 610 К дейінгі температура диапазонында қоспа висмут кальций және магниймен толық әрекеттеседі, нәтижесінде CaMg2Bi2 қосылысы түзіледі. Оңтайлы тәжірибелік жағдайларда висмут қосылысы CaMg2Bi2 BiCa2-ге айналады. Айта кету керек, BiCa2 ұшпайтын және қалдық ретінде тигельде қалады. Көмекші кальций толығымен CaSe және CaTe-ге айналады, бұл ұшқыш заттардың құрамындағы кальций мөлшерінің 0,5%-дан 16 ppm-ге дейін төмендеуіне әкеледі. Сол сияқты ұшпа заттардағы магний мөлшері 0,66%-дан 187 промиллеге дейін төмендейді. Сайып келгенде, соңғы өнімдегі висмут мөлшері 6 ppm-ден 1,4 ppm-ге дейін төмендейді, 76,6% жою жылдамдығына жетеді, ал металл қорғасынының тікелей шығуы 71% жетеді. Бұл процесс металл қорғасынды висмут қоспаларынан бөлуді тиімді түрде жеңілдетеді.
Түйінді сөздер: висмутты қорғасыннан вакуумда бөлу, адъювантты түрлендіру, вакуумды газдандыру, жоғары температурада диссоциациялау, бөлу және тазарту.

ЭКСПЕРИМЕНТАЛЬНОЕ ИССЛЕДОВАНИЕ ПО УДАЛЕНИЮ ВИСМУТА ИЗ СВИНЦА С КАЛЬЦИЕМ МАГНИЕМ

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Аннотация. Отделение свинца от смеси висмута остается серьезной проблемой, достижение эффективного разделения является важным препятствием для производства свинца высокой чистоты с помощью метода вакуумной газификации. Это исследование фокусируется на свинце как на основной дисциплине исследования, проводя теоретические и экспериментальные исследования вторичного преобразования свинца с помощью вакуумной газификации. Расчеты свободной энергии Гиббса показывают, что в диапазоне температур от 600 до 610 К соединение висмут полностью реагирует с кальцием и магнием, в результате чего образуется соединение CaMg2Bi2. В оптимальных экспериментальных условиях соединение висмута превращается в CaMg2Bi2 BiCa2. Следует отметить, что BiCa2 нелетает и остается в тигле в качестве остатка. Вспомогательный кальций полностью превращается в CaSe и CaTe, что приводит к снижению содержания кальция в летучих веществах с 0,5% до 16 ppm. Точно так же содержание магния в летучих веществах снижается с 0,66% до 187 частей на миллион. В конечном итоге содержание висмута в конечном продукте снижается с 6 ppm до 1,4 ppm, достигая скорости удаления 76,6%, а прямой выход металлического свинца достигает 71%. Этот процесс эффективно облегчает отделение металлического свинца от примесей висмута.
Ключевые слова: вакуумное отделение висмута от свинца, адъювантное преобразование, вакуумная газификация, высокотемпературная диссоциация, разделение и очистка.

MAIN PROVISION

A calcium–magnesium-assisted refining process for the removal of bismuth from molten lead is proposed and experimentally validated. The study identifies the optimum concentrations of calcium (0.10–0.13 wt.%) and magnesium (0.35–0.45 wt.%) required for efficient debismuthization and demonstrates that the formation of the thermodynamically stable intermetallic compound $CaMg_2Bi_2$ is the dominant reaction mechanism. Thermodynamic analysis based on Gibbs free energy calculations is combined with experimental verification to clarify the influence of reagent concentration and temperature on the refining process. The proposed method significantly reduces the bismuth content while maintaining satisfactory lead recovery, demonstrating its potential as an efficient and environmentally friendly technology for the production of high-purity lead

INTRODUCTION

Lead is a dense and malleable metal with the chemical symbol Pb. It possesses notable properties such as excellent ductility, corrosion resistance, and effective shielding against X-ray radiation, which contribute to its widespread application in various industries, including battery manufacturing, cable sheathing, the automotive industry, and the military sector [1-5]. High-purity lead serves as a key raw material for advanced high-performance pure lead batteries and valve-regulated lead-acid batteries. Its importance is increasingly recognized in strategic fields such as fast nuclear reactors and aerospace engineering, making it a significant area of development in China.

Vacuum volatilization refining technology refers to a metallurgical process for the purification, refining, and treatment of metals in a vacuum-sealed system operating at pressures below 0.1 MPa. This technology has been widely applied in alloy separation, deep metal purification, and the sustainable recovery of secondary resources [6-10]. Since the beginning of the 21st century, the concept of green development has received increasing attention, posing new challenges to conventional lead refining processes. The main disadvantages of traditional fire refining include outdated technologies, high energy consumption, and significant environmental pollution, resulting in substantial annual costs associated with waste treatment. With the growing demand for high-quality materials, vacuum volatilization technology offers considerable advantages in producing high-purity lead by effectively minimizing material oxidation, thereby outperforming conventional fire refining and hydrometallurgical electrolytic purification methods.

Research on the removal of bismuth impurities from lead using calcium and magnesium has a long and significant history. This technology emerged in the early twentieth century in the field of metal refining, with the primary objective of efficiently removing bismuth impurities from lead through the addition of calcium and magnesium. The process aims to improve both the purity and overall quality of lead. Lead is a widely used industrial metal employed in battery production, paint manufacturing, radiation shielding, and many other applications. However,

during sulfide ore smelting and lead refining processes, lead is often contaminated with impurities such as bismuth. The presence of bismuth adversely affects the physical and chemical properties of lead, including its density, hardness, and electrical conductivity. Furthermore, it negatively influences the subsequent processing and application of lead by reducing battery performance and deteriorating the gloss and adhesion of coatings [11-15]. Therefore, the effective removal of bismuth impurities remains an important research topic in the field of lead refining.

The removal of bismuth using calcium and magnesium has been extensively investigated as an effective lead refining technology since the early twentieth century. This method is based on the ability of calcium and magnesium to react with bismuth dissolved in molten lead, forming refractory compounds that are insoluble in liquid lead and can therefore be removed as dross. The development and implementation of this principle have significantly advanced lead refining technology. Nevertheless, despite the considerable progress achieved, fundamental research on the calcium-magnesium debismuthization process remains insufficient. In particular, further studies are required to clarify the effects of calcium and magnesium concentrations, temperature, and other key process parameters on the efficiency of bismuth removal, as well as to better understand the underlying mechanisms governing the process [16-19].

Experimental investigations of bismuth removal using calcium and magnesium not only provide valuable insights into the microscopic mechanisms of the process but also offer theoretical foundations and practical guidance for optimizing technological parameters to enhance bismuth removal efficiency. In addition, the influence of temperature on bismuth removal has been investigated. The results indicate that within the temperature range of 1123-1223 K, at a system pressure of 2-5 Pa and a holding time of 3 hours, the removal rate of bismuth impurities from lead increases with increasing temperature. In particular, when the temperature rises from 1223 K to 1323 K, a significant increase in the bismuth removal rate is observed, accompanied by a considerable reduction in its concentration in the condensed products. These findings are of great importance for optimizing process parameters and improving the efficiency of bismuth removal.

Gibbs free energy, often referred to as Gibbs free enthalpy, is a fundamental concept in thermodynamics. It quantitatively describes the maximum non-expansion work that a system can perform under isothermal and isobaric conditions. Essentially, it is used to evaluate the spontaneity and direction of chemical reactions and physical processes. The calculation of Gibbs free energy plays a crucial role not only in explaining the spontaneous nature of reactions but also in guiding chemical process design, materials development, and biological systems.

In practical applications, accurate calculation of Gibbs free energy requires prior determination of both the enthalpy change and entropy change associated with a reaction. These thermodynamic quantities can generally be obtained through experimental measurements or from published literature. For complex chemical systems or multistep reactions, more sophisticated approaches involving thermodynamic cycles may be employed to evaluate the Gibbs free energy changes for each individual reaction step. Although Gibbs free energy is a valuable criterion for predicting the spontaneity of reactions, it should be emphasized that it does not provide direct information regarding reaction rates or kinetics. Moreover, under certain conditions, additional factors must be considered to achieve a more comprehensive assessment of reaction behavior [20-22]. Overall, the calculation of Gibbs free energy provides an essential theoretical basis for solving a wide range of practical scientific and engineering problems.

Research gap. Although calcium- and magnesium-assisted lead refining has been widely investigated and successfully applied for bismuth removal, previous studies have primarily focused on empirical optimization of reagent additions and operating conditions. The thermodynamic mechanisms governing the formation of calcium–magnesium–bismuth intermetallic compounds remain insufficiently understood, and the combined effects of reagent concentration and process temperature on the efficiency of debismuthization have not been comprehensively evaluated. Furthermore, limited attention has been given to integrating thermodynamic predictions with experimental validation to explain the dominant reaction pathways responsible for bismuth removal.

To address this gap, the present study combines Gibbs free energy calculations with laboratory-scale experiments to investigate the calcium–magnesium-assisted removal of bismuth from molten lead. The work identifies the optimum concentrations of calcium and magnesium, clarifies the thermodynamic feasibility of $CaMg_2Bi_2$ formation as the dominant reaction product, and establishes the relationship between processing parameters and bismuth removal efficiency. This integrated approach provides both theoretical insight and practical guidance for the development of more efficient technologies for producing high-purity lead.

Novelty statement. This study combines thermodynamic analysis and experimental validation to clarify the calcium–magnesium-assisted mechanism of

bismuth removal from molten lead and identifies the optimum refining conditions for producing high-purity lead.

METODOLOGY

The raw materials used in the experiments are presented in Table 1. The main equipment, including the manufacturers and model specifications employed in this study, is listed in Table 2.

The vertical shaft furnace used in this investigation consists of a furnace body, an argon gas protection system, a gas injection system, a vacuum pumping system, and a central control unit. Specifically, the furnace body and base are fabricated from stainless steel, while the heating system comprises copper electrodes and a heating element.

The argon gas protection system consists of two components: an argon gas cylinder and an argon gas circulation unit. The gas cylinder continuously supplies high-purity argon to the quartz tube, whereas the circulation unit maintains a continuous inert gas atmosphere inside the tube. In combination with the argon protection system, the vacuum system thoroughly evacuates the quartz tube before introducing argon, thereby ensuring a complete protective atmosphere throughout the experiment.

The gas injection (forced stirring) system provides effective mixing of the molten metal in the crucible by regulating the flow rate of argon gas introduced into the melt. The control system is responsible for current regulation, voltage monitoring, temperature control, and pressure measurement throughout the experimental process.

Table 1. Source and purity of experimental materials

Metal	Purity
Lead (Pb)	99.992 %
Bismuth (Bi)	99.999 %
Calcium (Ca)	99.995 %
Magnesium (Mg)	99.996 %

Raw lead was initially prepared in the form of metal cubes. The oxide layer on the surface was removed using a stainless-steel knife, after which the surface was cleaned in an ultrasonic cleaner. Following ultrasonic cleaning, the lead was wiped with ethanol and dried using a hot-air dryer. The cleaned lead was then cut into small pieces and ground in a vibration mill for 3 min per cycle. The grinding procedure was repeated four times until a particle size of approximately 110–165 μm was achieved.

Bismuth granules were subsequently ground in the same vibration mill for 2 min per cycle, with the procedure likewise repeated four times. The resulting fine powders of the raw materials were accurately weighed according to the required proportions, thoroughly mixed, and loaded into a pressing die with a diameter of 30 mm. The obtained compact, measuring 30 mm in diameter and 35 mm in thickness, was placed into a graphite crucible, which was then positioned inside a quartz tube. The complete assembly was installed in the vertical shaft furnace, as illustrated in Figure 1.

The argon gas protection system was activated, and the furnace chamber was purged with argon. After the gas flow had stabilized, a vacuum pump was switched on to evacuate the air from the quartz tube. The vacuum pump was then turned off, and argon was reintroduced into the system. Once the pressure inside the quartz tube reached atmospheric pressure, the vacuum pump was operated again to remove the remaining air. This evacuation–argon purging cycle was repeated three to five times to establish a high-purity inert atmosphere.

During the experiment, argon was continuously supplied at a flow rate of 0.2 $L \cdot min^{-1}$. After the gas flow stabilized, the resistance heating system was activated, and the crucible was heated for 3 h to ensure complete melting, mixing, and homogenization of the alloy components. Subsequently, a quenched sample was collected, and the alloy was remelted in the crucible for one or two additional melting cycles to further improve its homogeneity. Finally, a homogeneous 200 g Pb–Bi alloy was obtained, as shown in Figure 2.

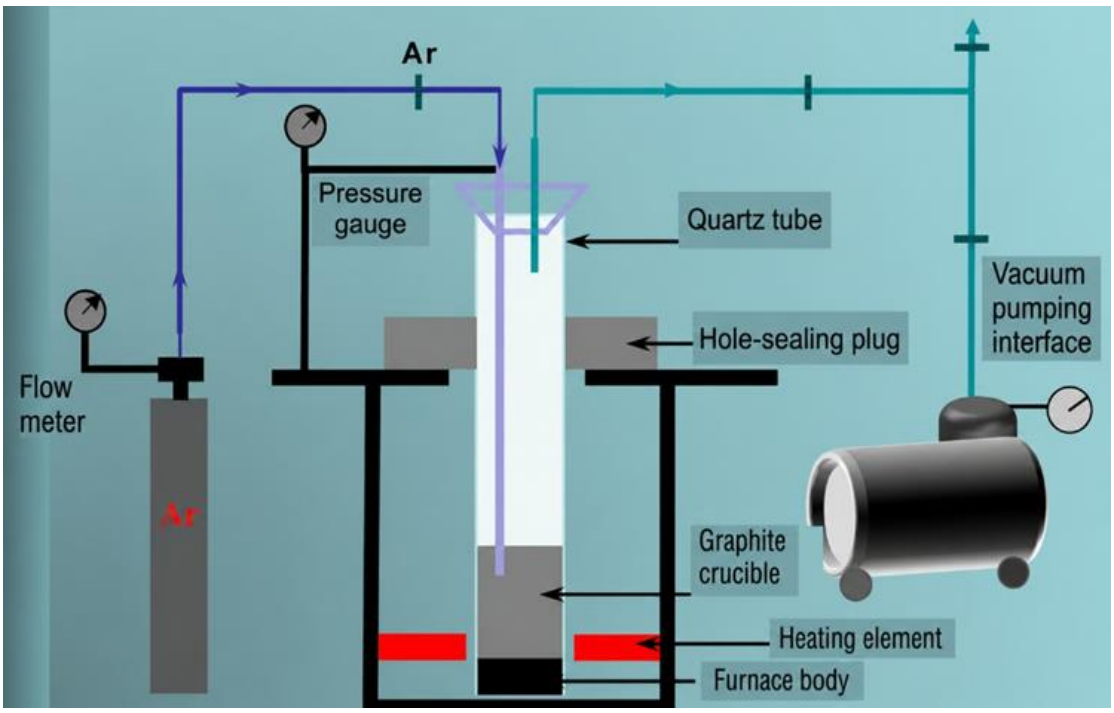

Fig. 1. Schematic diagram of the vertical shaft furnace.

Fig. 2. Pb-Bi alloy after quenching.

RESEARCH RESULTS

The optimum addition range of metallic calcium was found to be 0.10-0.13 wt.% of the total mass of metallic lead, whereas the optimum addition range of metallic magnesium was 0.35-0.45 wt.% of the total lead mass. When the calcium content was below 0.10 wt.%, the bismuth removal efficiency was less than 30%. Conversely, when the calcium concentration exceeded 0.13 wt.%, the bismuth removal efficiency remained below 30%, indicating that further addition of calcium did not improve the removal performance.

Similarly, when the magnesium concentration was below 0.35 wt.%, the bismuth removal efficiency was less than 50%. As the magnesium content increased, the removal efficiency also increased. However, when the magnesium concentration exceeded 0.45 wt.%, no significant improvement in the removal of bismuth impurities was observed.

The experimental results indicate that calcium and magnesium react with bismuth impurities within the temperature range of 600-650 K, forming low-density intermetallic compounds that float to the surface of the molten lead and can be removed as dross. According to the predicted solubility products, the principal reaction product formed between calcium, magnesium, and bismuth is likely $CaMg_2Bi_2$.

When the temperature reaches 660 K, the added calcium and magnesium begin to react extensively with the lead matrix, resulting in increased lead consumption and the formation of a considerable amount of slag. Under these conditions, metallic bismuth remains largely unreacted. However, the

reaction products formed among calcium, magnesium, and bismuth have not yet been conclusively identified due to the lack of comprehensive thermodynamic calculations and detailed phase characterization.

Based on the available thermodynamic data [23], the Gibbs free energy of the relevant chemical reactions was calculated to evaluate the possibility of forming Bi2Mg3, Bi2Ca3, and CaMg2Bi2 during the calcium-magnesium-assisted debismuthization process. A lower Gibbs free energy indicates a greater thermodynamic tendency for compound formation. The standard Gibbs free energies of formation of Ca(s), Bi(s), Bi(l), and the related substances at 600 K are presented in Table 2. The formation reactions of the products generated during the auxiliary refining process are expressed as follows:

$2Bi(l) + 3Mg(s) = Bi2Mg3(s) = 2Bi(l) + 3Mg(s) = Bi2Mg3(s)$ (1)

$2Bi(l)+3Ca(s) = Bi2Ca3(s)= 2Bi(l) + 3Ca(s) = Bi2Ca3(s)$ (2)

$2Bi(l) + Ca(s) + 2Mg(s) = CaMg2Bi2(s) = 2Bi(l) + Ca(s) + 2Mg(s) = CaMg2Bi2(s)$ (3)

$Bi(s) = Bi(l)$ (4)

Table 2. Standard Gibbs free energies of formation of Ca(s), Bi(s), Bi(l) and related species at 600 K.

Species	Standard Gibbs Free Energy of Formation, kJ·mol^{-1} (600 K)
$Ca_{(s)}$	–27.94
$Mg_{(s)}$	–22.66
$Bi_{(s)}$	–38.34
$Mg_{(s)}$	–22.66
$Bi_{(s)}$	–38.34
$Mg_{(s)}$	–22.66
$Bi_{(s)}$	–38.34

According to the Handbook of Inorganic Thermodynamics [24], bismuth undergoes a phase transition from the solid state to the liquid state at 600 K. The standard Gibbs free energy of formation of liquid bismuth reported in the handbook is −39.49 kJ·mol-1. The Gibbs free energies of the reactions represented by Equations (1)-(3) were calculated at 600 K. The Gibbs free energy of the reaction described by Equation (1) was found to be −22.56 kJ·mol^{-1}, whereas that of Equation (2) was −154.48 kJ·mol-1. Based on the Gibbs free energy values of these reactions, it can be concluded that, at 600 K, the auxiliary elements calcium and magnesium can react with bismuth impurities to form compounds such as Bi2Ca3 and CaMg2Bi2. It should be noted that the thermodynamic probability of CaMg2Bi2 formation is considerably higher than that of Bi2Mg3 formation.

DISCUSSION

The present study demonstrates that the combined addition of calcium and magnesium provides an effective approach for the removal of bismuth from molten lead under controlled refining conditions. The experimentally determined optimum concentrations (0.10–0.13 wt.% Ca and 0.35–0.45 wt.% Mg) indicate that efficient debismuthization depends on maintaining a balanced ratio between the auxiliary metals and the dissolved bismuth. Below these concentration ranges, an insufficient amount of reactive species is available for complete interaction with bismuth, whereas excessive additions do not improve the removal efficiency because the surplus calcium and magnesium preferentially react with the lead matrix or form secondary phases that do not contribute to bismuth removal.

The observed dependence of bismuth removal efficiency on reagent concentration is consistent with the thermodynamic calculations performed in this study. Gibbs free energy analysis confirmed that the formation of $CaMg_2Bi_2$ is thermodynamically more favorable than the formation of individual calcium- or magnesium-bismuth compounds. This finding suggests that the simultaneous addition of calcium and magnesium produces a synergistic effect, promoting the formation of a stable intermetallic phase with a greater driving force than reactions involving either element alone. Consequently, the experimental results support the theoretical prediction that $CaMg_2Bi_2$ is the predominant reaction product during the debismuthization process.

Temperature was also found to play a decisive role in refining performance. At 600–650 K, calcium and magnesium selectively react with bismuth, producing low-density intermetallic compounds that can be separated as dross. However, when the temperature approaches 660 K, the selectivity of the process decreases because calcium and magnesium increasingly react with molten lead. This results in higher lead consumption and greater slag formation without a corresponding increase in bismuth removal efficiency. Therefore, precise temperature control is

essential not only for maximizing impurity removal but also for minimizing valuable lead losses during refining.

The obtained results are in agreement with previous studies reporting that calcium-based refining is an effective method for removing bismuth from lead. Earlier investigations primarily focused on empirical optimization of reagent additions, whereas the present work combines experimental observations with thermodynamic analysis to explain the mechanism responsible for impurity removal. The agreement between theoretical Gibbs free energy calculations and experimental observations provides additional confidence in the proposed reaction pathway and confirms that thermodynamic modeling can serve as a reliable tool for optimizing industrial refining processes.

From an industrial perspective, the proposed calcium-magnesium refining approach offers several advantages. The process operates with relatively low reagent additions while achieving a high bismuth removal efficiency, thereby reducing reagent consumption and minimizing secondary waste generation. Moreover, the formation of stable intermetallic compounds facilitates the physical separation of impurities from molten lead, making the process compatible with conventional refining operations. Such improvements may contribute to the production of high-purity lead required for advanced applications, including lead-acid batteries, nuclear technologies, and specialized electronic materials.

Despite these promising results, several limitations should be acknowledged. The present investigation was conducted under laboratory-scale conditions using high-purity raw materials, whereas industrial lead typically contains a wider range of impurity elements that may influence reaction pathways and phase formation. Furthermore, the reaction products were identified primarily on the basis of thermodynamic calculations and process behavior. Additional microstructural characterization using techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS) would provide direct confirmation of the composition and morphology of the intermetallic compounds formed during refining. Future studies should also investigate the influence of holding time, stirring intensity, vacuum conditions, and complex impurity systems to further improve the efficiency and industrial applicability of the proposed debismuthization process.

CONCLUSION

This study proposes a novel approach to refining metallic lead and separating bismuth impurities from lead under vacuum conditions based on the principles of vacuum metallurgy. The proposed method involves auxiliary conversion followed by evaporation and volatilization. The conversion reaction between the auxiliary agents, namely calcium and magnesium, and bismuth impurities is intensified by argon purging and mechanical stirring. This process promotes the transformation of bismuth impurities in molten lead into complex compounds with physicochemical properties that differ significantly from those of lead. Subsequently, bismuth is effectively separated through vacuum evaporation, resulting in the production of high-purity lead. The proposed method is of considerable importance for advancing the lead industry, the energy sector, and the development of advanced materials requiring high-purity lead.

The principal findings of this study demonstrate that the reaction product formed between the calcium–magnesium additives and bismuth impurities is $CaMg_2Bi_2$, as predicted by Gibbs free energy calculations. Under specific operating conditions, particularly at a system pressure of 2–5 Pa, this compound undergoes volatilization during vacuum treatment, while $BiCa_2$ remains as a non-volatile residue accumulated inside the crucible. A small fraction of bismuth continues to evaporate, reducing its concentration in the condensate from 4.85 wt.% to 1.81 wt.%. The added calcium is completely converted into CaSe and CaTe, resulting in a decrease in the calcium content of the condensate from 0.5 wt.% to 16 ppm. Likewise, the magnesium concentration in the condensate decreases from 0.66 wt.% to 187 ppm. During vacuum distillation, the primary metal, lead, evaporates extensively and subsequently condenses as solid metal in the condenser vessel. As a result, the bismuth content in the final lead product is reduced from 6 ppm to 1.4 ppm, while the direct metal recovery reaches 71%, demonstrating the high efficiency of the proposed process for the removal of bismuth impurities from lead.

Author Contribution Statement (CRediT Taxonomy): N.N. Zobnin: Conceptualization, Methodology, Investigation, Formal analysis, Validation, Data curation, Visualization, Writing – Original Draft. **Немчинова Н.В:** Supervision, Methodology, Formal analysis, Writing – Review & Editing. Both authors have read and approved the final version of the manuscript and agree to be accountable for all aspects of the work.

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