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Study on the behavior of zinc and associated metal-impurities in the process of chlorinating roasting of dross

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ABSTRACT

In this work the issue of processing of dross with preliminary separation of the metal part from the oxide one is reviewed. Based on a comparative analysis of the results of known works it is shown that this approach can be quite effective if a technology is developed for the separate processing of the oxide part to obtain a commercial product. The authors of this work proposed carrying out chlorinating roasting under oxidizing conditions with various chlorine-containing reagents as one of the effective ways of processing the oxide part of the dross. Based on the thermodynamic analysis of the reactions of interaction between the components of the oxide part of the dross and calcium and ammonium chlorides, the principal possibility of obtaining pure zinc oxide, suitable for use as mineral additives in animal feed has been shown. As a result of thermodynamic calculations of the reactions of interaction of impurity metals with calcium chloride and ammonium chloride, the values of Gibbs free energy and reaction rate constants were determined in the temperature range 973 and 1373 K. It has been established that under the conditions of roasting the oxide part of the dross with both calcium chloride and ammonium chloride, a high degree of sublimation of impurity metals from the dross in the form of their chlorides into dust is achieved. It has been shown that roasting the oxide part of the dross using ammonium chloride is more efficient than with calcium chloride.

Key words: Dross, metallic zinc, zinc oxide, roasting, thermodynamics, impurity metals, Gibbs free energy, calcium chloride, ammonium chloride.

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Introduction

The problem of metal protection against corrosion is one of the most important technical problems all over the world. Among the large number of known methods of protecting steel structures from corrosion the hot-dip galvanizing is effective, technological and fairly cheap. Zinc coating has a good appearance and allows increasing the service life of products by 2-3 times and reliably protects them from atmospheric and other types of corrosion [1].

The high prices for zinc, which make up the main part of the cost of galvanizing, require the economical use of zinc and the disposal of its waste, one of which is

dross. The output of dross is from 0.5 to 3.5% of the mass of the passed products. Dross in its composition is represented by zinc oxide. It contains 30-40% of metallic zinc [2]. The complex chemical composition of the dross makes it difficult to further usage of it. Its main processing in practice is about extraction of the metal part of zinc, which due to its low quality is not commercial product and is used in the process of galvanizing products. At the same time, the non-metallic part of the dross remaining after the separation of zinc is accumulated and stored at enterprises and plants, occupying large territories, due to the significant content of lead and other impurity metals in it and the lack of rational processing technology on the other

hand. Well organized work of the galvanizing section gives an opportunity of minimizing the amount of waste and creating own production of their processing with the production of additional marketable products.

Currently, pyrometallurgical methods of processing substandard zinc-containing polymetallic products are widely used in practice. The known methods are characterized by large material and capital costs and do not provide a high complex extraction of zinc and other valuable metals [3, 4, 5]. Hydrometallurgical methods have been developing in recent years [2, 6-8].

The most common ways of processing dross are their heating in cylindrical drums or retorts of various designs to a temperature higher than the melting point of zinc [9]. The disadvantage of these methods is the lack of sealing the internal volume of the drum. The second significant drawback is the absence of direct contact of the walls of the heated combustion chamber with the raw material loaded into the drum.

The patented pyrometallurgical methods of processing of dross with the use of various reagents (acids, alkaline methods, the addition of sodium and aluminum fluorides, etc.) have become widespread [10-15]. The main disadvantages of these works include: the laboriousness of the separation of the metal fraction, the formation of a large volume of wastewater requiring additional purification and evaporation to obtain zinc chloride; energy costs associated with the use of additional equipment, an increase in the number of workers. Organization of an additional site for processing zinc dross; the need for additional transport equipment to move the liquid metal into the hot-dip galvanizing bath; additional costs for evaporation of zinc chloride solution; the high cost of some reagents used in the processing of dross.

One of the most effective methods for processing the dross can be the preliminary separation of the metal and oxide parts of the dross with further separate processing of each of them [16]. The metal part is melted in an induction furnace under a layer of ammonium chloride and charcoal. Metallic zinc is obtained from the metal part with the composition, %: 95.9 Zn; 1.54 Pb; 0.9 Fe; 0.4 Cu.

The oxide part of the dross is subjected to roasting at 800-900 °C and zinc oxide is obtained, which is used for the preparation of whitewash. At the same time, due to the high content of lead (1.2%) and iron (0.95%) in the dross, the quality of the produced whitewash is low.

The disadvantages of this method include: the difficulty of separating the metallic and non-metallic oxide fractions; zinc oxide remains in the metal drops of zinc, which greatly impacts the quality of the produced zinc whitewash.

Despite the indicated disadvantages, the approach proposed in [16] for the preliminary separation of the metal and oxide parts of the dross with further separate processing of each of them seems to be quite effective. At the same time, if the metal part can be used as a material for secondary use in galvanizing, then the investigating the ways to process the non-metallic, oxide part of the dross containing such impurities as Pb, Fe, As, Sb requires additional research.

It has a fundamental importance to study the behavior of lead and iron, in the vision of their high concentrations in the oxide part of the dross which can reach 1.2 and 1%, respectively. The high content of metal impurities in the dross hinders their further sale and use, and leads to accumulation. The results of well-known works on the processing of dross do not give an unambiguous picture of removing impurities.

In this work, based on the study of the behavior of zinc and accompanying impurities (Pb, Fe, As, Sb) under the conditions of chlorinated roasting of the oxide part of the dross with calcium and ammonium chlorides, the fundamental possibility of obtaining pure commercial zinc oxide suitable for use as mineral additives in animal feed is substantiated.

Research methods

The study of the behavior of zinc and accompanying metals-impurities under the conditions of roasting the oxide part of the dross was carried out based on a thermodynamic analysis of the reactions occurring between the components of the dross and calcium chloride and ammonium chloride.

The probable direction of the reactions was estimated from the change in the thermodynamic values of the system. Calculations of thermodynamic analysis were carried out taking into account the dependence of the Gibbs free energy of reactions on temperature. The change in Gibbs free energy (ΔG°_T) was calculated by the formula: in (1).

$$\Delta G^{\circ}_T = \Delta H^{\circ}_T - \Delta S^{\circ}_T \times T \quad (1)$$

where, ΔH°_T , ΔS°_T – standard values of enthalpy (J) and entropy (J/T) of the system,

T – temperature, K.

During calculating the Gibbs energy (ΔG°_T) of the reactions the standard state of metals and compounds present in dross, as well as quartz, iron oxide and chlorinating reagents (CaCl_2 , NH_4Cl) is taken as their solid state. For gaseous compounds

obtained as a result of reactions, gases are taken as the standard state.

The calculations used reference data from [17] and the NIST-JANAF Thermo chemical Tables website (<http://kinetics.nist.gov/janaf>). Thermodynamic calculations were carried out using a special program developed by the authors of the work. The change in the reaction equilibrium constant (Kp) depending on temperature was determined based on the expression in equation (2).

$$\Delta G^{\circ}_T = -RT \ln K_p = -19.155 \times T \lg K_p \quad (2)$$

where, R – universal gas constant (R = 8.31 J/K·mol).

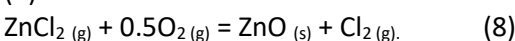
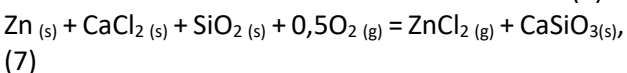
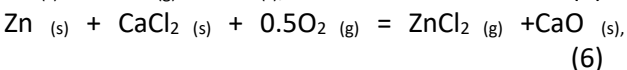
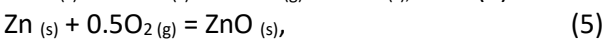
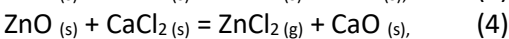
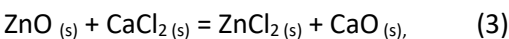
Results and discussion

The main purpose of oxidative roasting is the deep sublimation of impurities in the form of chlorides into dust to obtain pure zinc oxide in the condensed phase (cinder). This will give an opportunity already at the initial stage of joint processing of the oxide part of the dross with chlorine-containing reagents, to obtain dust concentrated with lead chloride, which is sent for the further processing to extract lead by known methods, and to isolate pure zinc oxide into a commercial product suitable for use as a mineral additive in animal feed [18].

The study of the behavior of zinc and accompanying metals, as well as their compounds under the conditions of roasting the oxide part of the dross, was carried out using calcium chloride and ammonium chloride.

Thermodynamic analysis of the reactions of interaction between the components of the oxide part of dross and calcium chloride in an oxidizing atmosphere.

In the oxidative roasting of the oxide part of the dross together with CaCl₂, the general chemistry of the process is described by a system of reactions in equation (3) - (8).



The temperature dependence of the calculated values of the Gibbs free energy and rate constants of reactions (3) - (8) is shown in Figure 1.

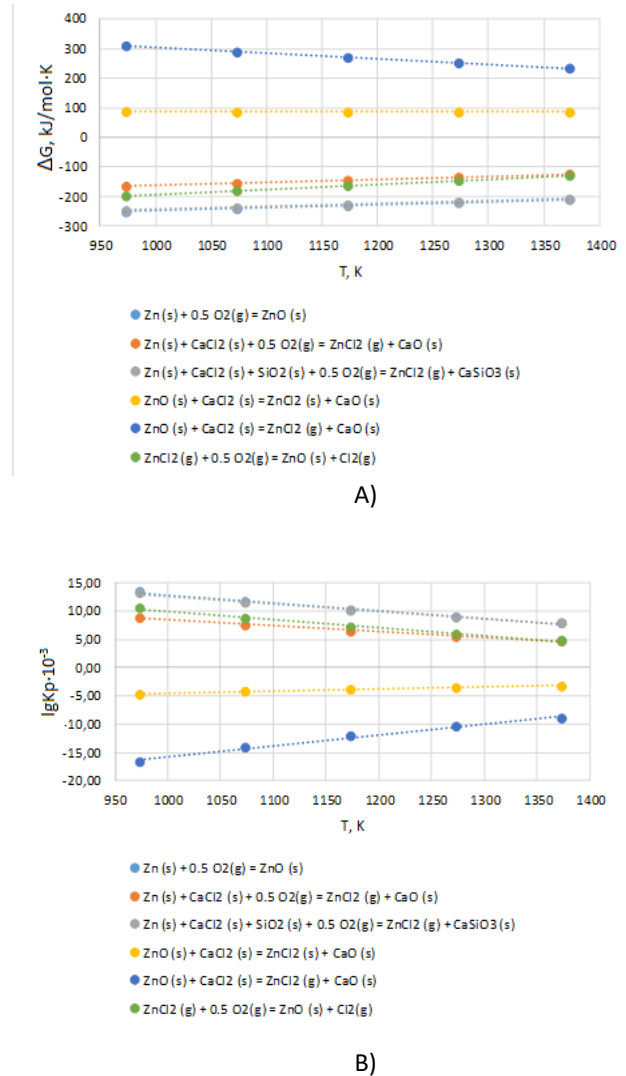


Figure 1 - Dependence of Gibbs free energy (A) and rate constants of reactions (2) - (8) on temperature (B)

It can be seen that the occurrence of reactions (3) and (4) between zinc oxide and CaCl₂ in the entire investigated temperature range is not possible, due to the positive values of the Gibbs free energy.

The presence of metallic zinc in the oxide part of the dross makes it necessary to conduct a thermodynamic analysis of its behavior during roasting. As can be seen in Figure 1, under the conditions of an oxidizing roasting atmosphere, metallic zinc can undergo active oxidation according to reaction (5) with the formation of solid zinc oxide. This is confirmed by the high value of the Gibbs free energy at a roasting temperature of 1373 K, which is ΔG_{1373 K} = -209.36 kJ/mol. It is necessary to note that

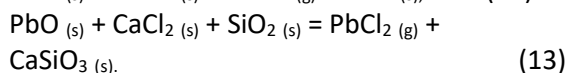
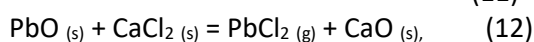
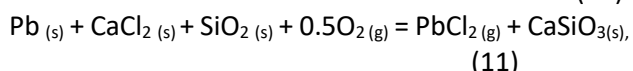
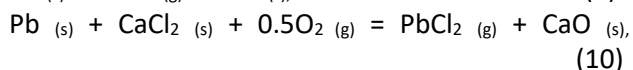
the values of the Gibbs free energy of reaction (3) in the temperature range 973 and 1373 K practically coincide with the values of the Gibbs free energy of reaction (7).

During roasting the active chlorination of metallic zinc with calcium chloride according to reaction (6) should be expected. Large negative values of Gibbs free energy, varying with increasing temperature (from 973 to 1373 K) and constituting $\Delta G_{973\text{K}} = -165.07 \text{ kJ/mol}$ and $\Delta G_{1373\text{K}} = -123.79 \text{ kJ/mol}$, indicate the possibility of the formation of gaseous chloride zinc and solid calcium oxide in the cinder. Reaction (6) is enhanced in the presence of quartz and is described by reaction (7). The Gibbs free energies of reaction (7) at 973 and 1373 K, which are $\Delta G_{973\text{K}} = -247.5 \text{ kJ/mol}$ and $\Delta G_{1373\text{K}} = -208.92 \text{ kJ/mol}$ indicate the possibility of the formation of gaseous zinc chloride and solid wollastonite obtained in as a result of the binding of free calcium oxide by silica.

Gaseous zinc chloride released as a result of reactions (6) and (7), interacting with atmospheric oxygen according to reaction (8), forms solid zinc oxide, which is concentrated in the cinder. Despite the decrease in Gibbs free energy values with increasing temperature from $\Delta G_{973\text{K}} = -197.5 \text{ kJ/mol}$ to $\Delta G_{1373\text{K}} = -128.6 \text{ kJ/mol}$, its high values show a high probability of a reaction towards the formation of solid zinc oxide with the release of gaseous chlorine.

Thus, it can be assumed that under conditions of joint roasting of the oxide part of the dross with CaCl_2 in an oxidizing atmosphere, favorable conditions are created for the conversion of metallic zinc into oxide. From a practical point of view, this means an increase of the content of zinc oxide in the final product, on the other hand, an increase of the quality of the resulting zinc oxide.

The mechanism of the behavior of lead and its oxide during the roasting of the oxide part of the dross with CaCl_2 can be represented by a system of reactions in equation (9) - (13).



The calculated values of the Gibbs free energy and the rate constant of reactions in equation (9) -

(13) in the temperature range 973 and 1373 K are presented in Figure 2.

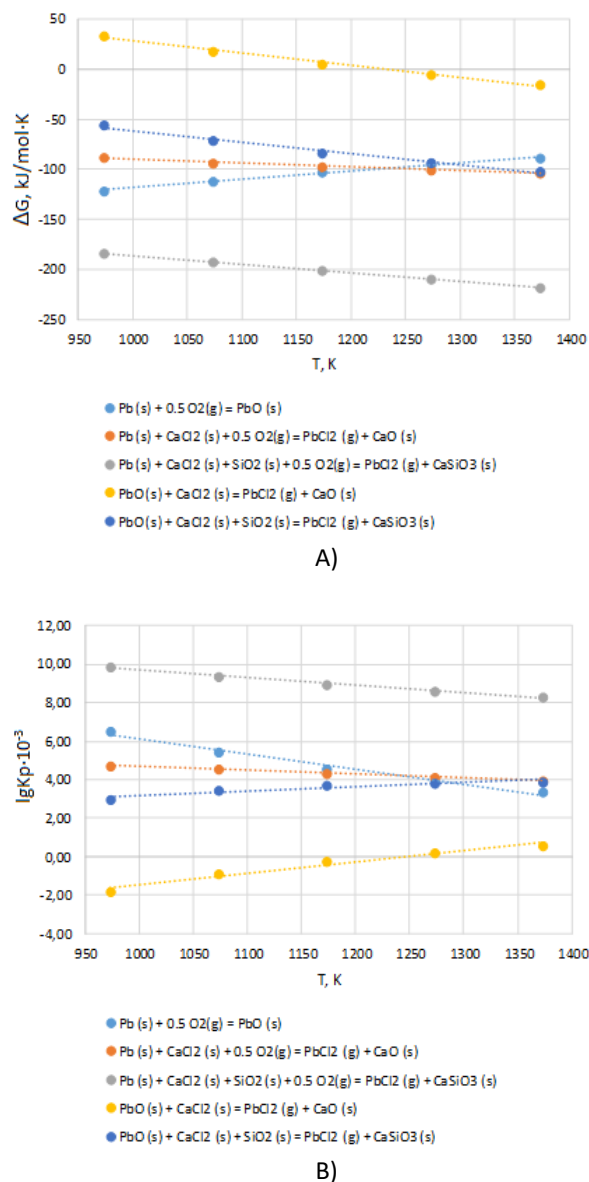


Figure 2 - Dependence of Gibbs free energy (A) and rate constants of reactions (9) - (13) on temperature (B)

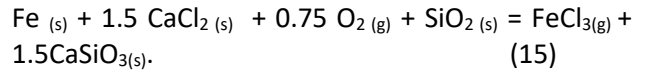
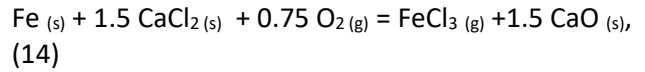
At the roasting temperature (1373 K) all reactions are characterized by negative values of the Gibbs free energy, and reactions (10) - (13) with increasing temperature show insignificant increase in the values of the Gibbs free energy. Reaction (9) is undesirable due to the formation of solid lead oxide, which being concentrated in the cinder, degrades the quality of zinc oxide. However, at a roasting temperature of 1373 K solid lead oxide formed according to reaction (9) interacting with CaCl_2 according to reaction (12) can promote the formation of gaseous PbCl_2 . In this case, the value of the Gibbs free energy at a temperature of 1373 K is

$\Delta G_{1373K} = -15.07$ kJ/mol. In the presence of quartz, this reaction is activated, as confirmed by an increase in the Gibbs free energy, the value of which at a temperature of 1373 K is $\Delta G_{1373K} = -101.95$ kJ/mol.

The course of reactions (10) - (13) favors the deep sublimation of lead in the form of its gaseous chloride which will have a significant effect on the quality of zinc oxide. The presence of free quartz enhances the effect of lead sublimation from the dross by reaction (9) with the formation of gaseous $PbCl_2$ and $CaSiO_3$. The obtained results show that in the process of roasting the oxide part of the dross jointly with $CaCl_2$ favorable conditions are achieved for the deep sublimation of lead in the form of its gaseous chloride.

From a practical point of view, this means the possibility of obtaining pure zinc oxide, which does not contain lead, under the conditions of chlorinating roasting of the oxide part of the dross with calcium chloride.

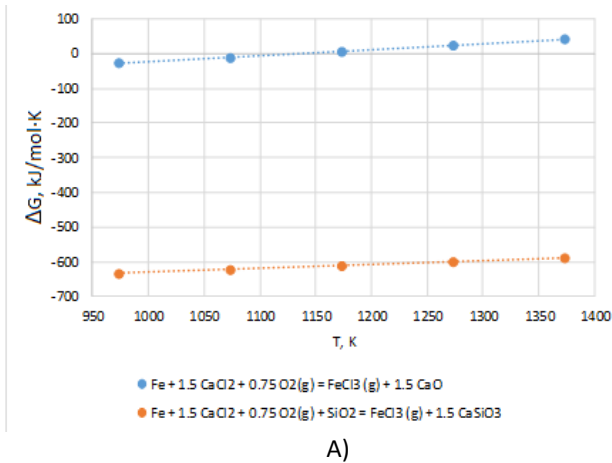
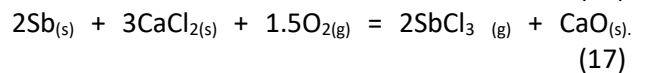
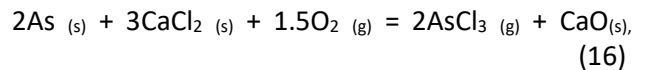
The behavior of iron under conditions of chlorinating roasting can be represented by the course of reactions in equation (14) and (15).



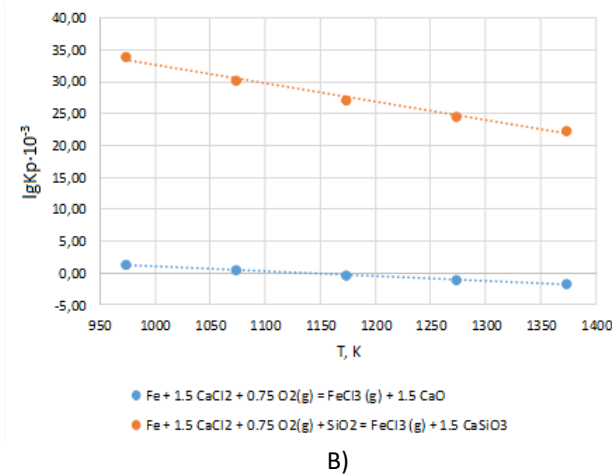
The probability of reaction (14) is unlikely, due to the positive value of the Gibbs free energy at the roasting temperature, which is $\Delta G_{1373K} = 43.73$ kJ/mol (Figure 3).

The ability to remove iron from the dross is sharply intensified in the presence of quartz, according to reaction (15). The Gibbs free energy of reaction (15) at the roasting temperature increases sharply and is $\Delta G_{1373K} = -588.22$ kJ/mol, which is seen on Figure 3. The high value of the Gibbs energy indicates the possibility of deep sublimation of iron in the form of its chloride.

Considering that insignificant amounts of arsenic and antimony may be present in the dross their behavior during roasting was analyzed. The possibility of removing arsenic and antimony from the oxide part of the dross under the conditions of chlorinating roasting can be represented by the reactions in equation (16) and (17).



A)

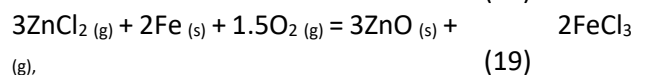
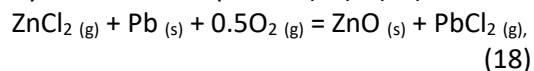


B)

Figure 3 - Dependence of Gibbs free energy (A) and rate constants of reactions (14), (15) on temperature (B)

In contrast to iron, reactions (16), (17) are characterized by high values of the Gibbs free energy in the entire investigated temperature range (Figure 4). Under the conditions of the roasting process (1373 K), the Gibbs free energy values for reactions (16) and (17) are: $\Delta G_{1373K} = -105.0$ kJ/mol and $\Delta G_{1373K} = -157.3$ kJ/mol, respectively, which indicates the deep sublimation of arsenic and antimony from dross in the form of their trivalent chlorides.

When analyzing the behavior of impurity metals under roasting conditions, it is necessary to take into account the possibility of interaction of gaseous zinc chloride formed by reaction (8) with impurity metals by reactions in equation (18) - (21).



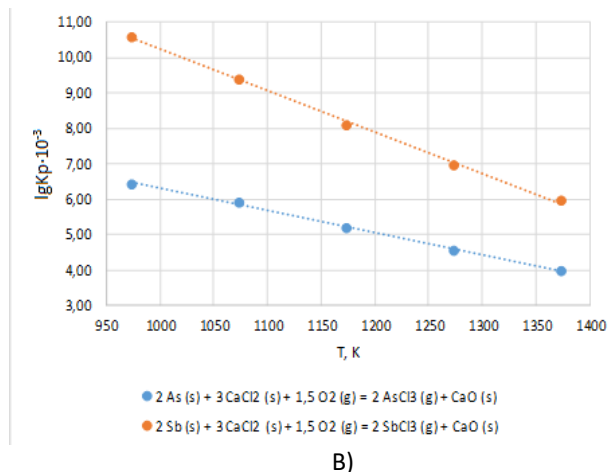
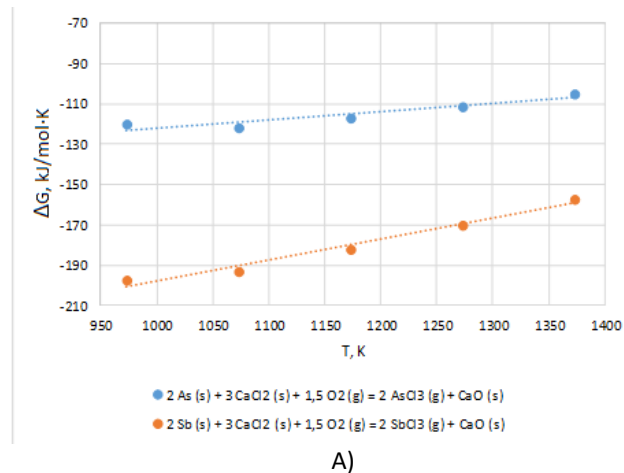
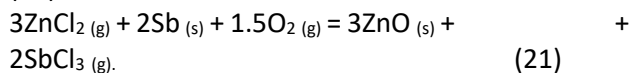
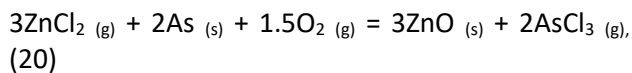


Figure 4 - Dependence of Gibbs free energy (A) and rate constants of reactions (16), (17) on temperature (B)

The established higher values of the Gibbs free energy and rate constants of reactions (18) - (21) and their growth with increasing temperature (Figure 5) indicate their priority over reaction (8) and reactions (9) - (17), which describe the direct chlorination of metal impurities with calcium chloride. The course of reactions (18) - (21) promotes deep sublimation of impurity metals and an increase in the quality of zinc oxide.

Thermodynamic analysis of the reactions of interaction between the components of the oxide part of dross and ammonium chloride in an oxidizing atmosphere.

The chemistry of the process of roasting the oxide part of dross with ammonium chloride in an oxidizing atmosphere can be represented by a system of the following reactions in equation (22) - (28).

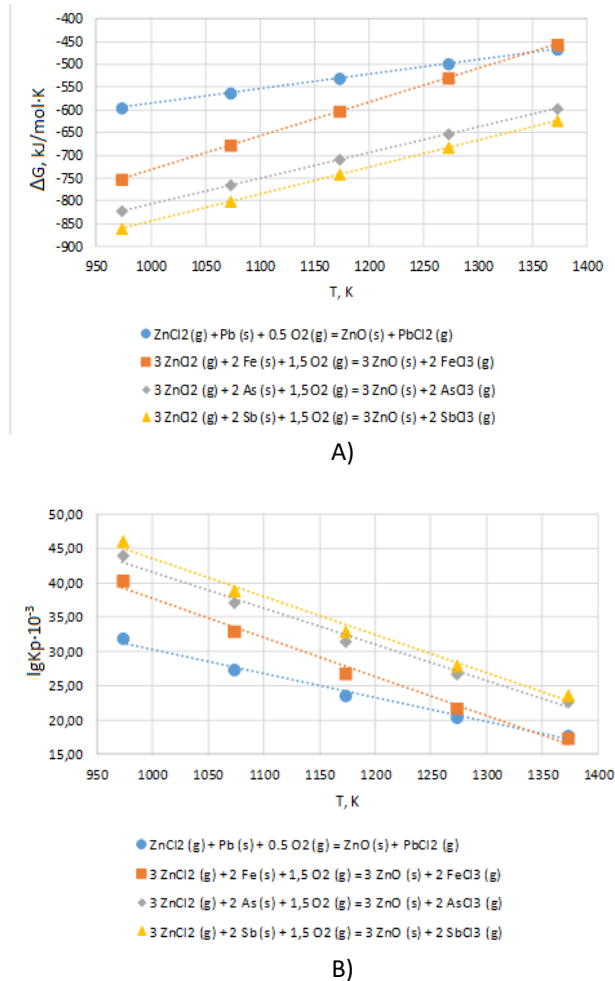
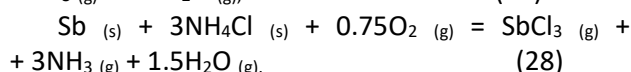
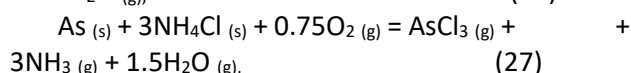
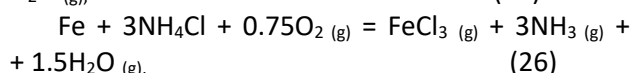
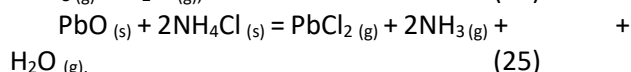
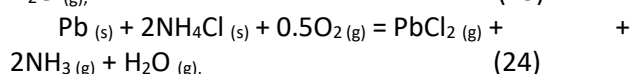
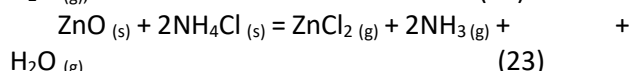
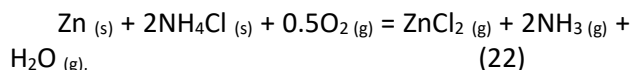


Figure 5 - Gibbs free energy dependence (A) and rate constants of reactions (18) - (21) on temperature (B)



The temperature dependence of the calculated values of the Gibbs free energy and rate constants of reactions (23) - (28), shown in Figure 6, shows a high probability of their occurrence.

The course of reactions (8) and (18) - (21) compensate for the growth of gaseous zinc chloride formed by reactions (22), (23) and contribute, together with reactions (24) - (28), to obtain high purity zinc oxide due to deep sublimation of metal impurities into dust.

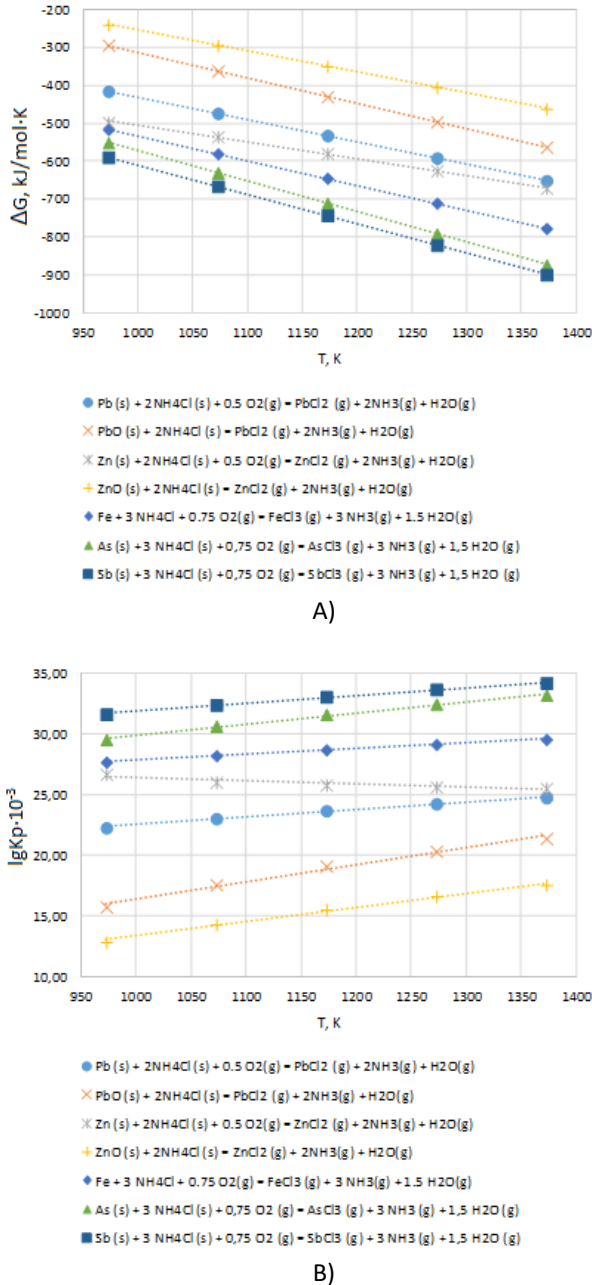


Figure 6 - Gibbs free energy dependence (A) and rate constants of reactions (22) - (28) on temperature (B)

The values of Gibbs free energy and rate constants of reactions (18) - (28) in the entire investigated temperature range are much higher than the values established for roasting the oxide

part of dross together with CaCl₂ and show an increase with increasing temperature. The obtained results indicate the preference and better efficiency of the chlorinated roasting of the oxide part of the dross with ammonium chloride than with calcium chloride.

Conclusions

1. Based on the thermodynamic analysis of the reactions of interaction between the components of the oxide part of the dross and various chlorine-containing reagents the possibility of obtaining pure zinc oxide has been shown. The results of thermodynamic calculations of Gibbs free energy and rate constants of chlorination reactions of metallic zinc and metal impurities with calcium and ammonium chlorides in the temperature range 973 and 1373 K are presented.

2. It was found that under the conditions of oxidative roasting the reactions of interaction of impurity metals (Pb, Fe, As, Sb) present in the oxide part of the dross with calcium and ammonium chlorides proceed with the formation of their chlorides and sublime. The high Gibbs free energies of chlorination of metals with ammonium chloride show better roasting efficiency than roasting with calcium chloride.

3. It is shown that the use of calcium chloride, even it is safer than the usage of ammonium chloride, the best technical and economic indicators of roasting, ensuring the production of pure zinc oxide, should be expected in the chlorination of dross with ammonium chloride.

Conflict of interests. On behalf of all authors, the corresponding author declares that there is no conflict of interest.

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Күйіндіні хлорлап күйдіру процесі кезінде мырыштың және қоспа-металдардың таралуын зерттеу

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<p>Мақала келді: 05 мамыр 2021 Сараптамадан өтті: 21 маусым 2021 Қабылданды: 06 тамыз 2021</p>	<p>ТҮЙІНДЕМЕ Жұмыста алдын ала металдық бөлігінен оксидті бөлігін бөліп күйіндіні қайта өңдеу мәселесі қарастырылған. Белгілі жұмыстардың нәтижелерін салыстырмалы талдау негізінде, егер тауарлық өнімді алу үшін оксидті бөлігін бөлек өңдеу технологиясы жасалса, бұл тәсіл өте тиімді болуы мүмкін екендігі көрсетілген. Бұл жұмыстың авторлары күйіндінің оксидті бөлігін өңдеудің тиімді әдістерінің бірі ретінде тотықтыру жағдайында әр түрлі хлорлы реагенттермен хлорлап күйдіруді ұсынады. Күйіндінің оксидті бөлігінің компоненттері мен кальций мен аммоний хлориді арасындағы өзара әрекеттесу реакцияларын термодинамикалық талдау негізінде жануарлардың жеміне минералды қоспалар ретінде пайдалануға жарамды таза мырыш оксидін алудың принципалды мүмкіндігі көрсетілген. Металл қоспаларының кальций хлориді мен аммоний хлоридімен әрекеттесу реакцияларының термодинамикалық есептеулерінің нәтижесінде Гиббстің еркін энергиясының мәні және 973.-1373 К температуралық аралықтағы реакциялар жылдамдығының константалары анықталды. Күйіндінің оксидті бөлігін кальций хлоридімен де, аммоний хлоридімен де бірге күйдіру жағдайында қоспа-металдарды күйіндіден олардың хлоридтері түрінде шаңға айдаудың жоғары дәрежесіне қол жеткізілетіні анықталды. Аммоний хлоридін қолдана отырып, күйіндінің оксидті бөлігін күйдіру кальций хлоридіне қарағанда тиімді екендігі көрсетілген. Түйін сөздер: Күйінді, металдық мырыш, мырыш оксиді, күйдіру, термодинамика, қоспа-металдар, Гиббстің еркін энергиясы, кальций хлориді, аммоний хлориді.</p>
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Исследование поведения цинка и сопутствующих металлов-примесей в процессе хлорирующего обжига изгари

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<p>Поступила: 05 мая 2021</p>	<p>АННОТАЦИЯ В настоящей работе рассматривается вопрос переработки изгари с предварительным разделением металлической части от оксидной. На основании сравнительного анализа результатов известных работ показано, что данный подход может быть вполне эффективным в том случае, если будет разработана технология отдельной переработки оксидной части с получением товарного продукта. Авторы настоящей работы в качестве одного из эффективных</p>
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путей переработки оксидной части изгари предлагают проведение хлорирующего обжига в окислительных условиях различными хлорсодержащими реагентами. На основании термодинамического анализа реакций взаимодействия между компонентами оксидной части изгари и хлоридами кальция и аммония показана принципиальная возможность получения чистого оксида цинка, пригодного для использования в качестве минеральных добавок в корм животных. В результате термодинамических расчетов реакций взаимодействия металлов-примесей с хлоридом кальция и хлоридом аммония определены значения свободной энергии Гиббса и констант скорости реакций в температурном интервале 973-1373 К. Установлено, что в условиях обжига оксидной части изгари совместно как с хлоридом кальция, так и хлоридом аммония достигается высокая степень возгонки металлов-примесей из изгари в виде их хлоридов в пыль. Показано, что обжиг оксидной части изгари с использованием хлорида аммония более эффективен, чем с хлоридом кальция.

Ключевые слова: Изгарь, металлический цинк, оксид цинка, обжиг, термодинамика, металлы-примеси, свободная энергия Гиббса, хлорид кальция, хлорид аммония.

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