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Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
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CHLORINATION OF IRON PHOSPHIDE WITH CHLORINE AT THE PRESENCE OF OXYGEN TO PRODUCE PHOSPHORUS (V) OXIDE AND IRON (II, III) CHLORIDES

Abstract. This paper examines the research results on predicting the production of phosphorus (V) oxide and iron (II, III) chlorides from Fe_2P, which is a basic component of electrothermal ferrophosphorus. The research methods were thermodynamic modeling using the HSC-6.0 software package based on the minimum Gibbs energy and second-order rotatable designs (Box-Hunter). The aim of the research was to determine the thermodynamic possibility of obtaining gaseous iron chlorides and phosphorus (V) oxide from iron phosphide. The temperature in a range of 300-1500°C and chlorine amount effect on the behavior of iron and phosphorus in a $2Fe_2P – mCl_2 – 2.5O_2$ system was determined at pressure of 1 bar. It was found that the temperature of the onset of iron chloride sublimation depends on the amount of chlorine and decreases from 804 to 693°C (for $FeCl_2$) and from 777 to 412°C (for $FeCl_3$) with an increase in the amount of chlorine from 4.0 to 6.7 kmol; the complete iron chloride sublimation occurs at 4.5-6.0 kmol of chlorine in the temperature range of 1040-1100°C; the complete transition of phosphorus from Fe_2P to P_4O_{10} is in the temperature range of 730-840°C and the amount of chlorine of 4.0-6.0 kmol; the minimum chlorine transition degree into a gas phase (up to 0.1%) with simultaneous complete transition of phosphorus into P_4O_{10} and the iron extraction degree from Fe_2P into its gaseous chlorides of 91,8% occurs at 4.0-4.3 kmol of chlorine in the temperature range of 1090-1125°C.

Key words: ferrophosphorus, iron phosphide, chlorination, chlorine, oxygen, thermodynamic modeling, iron chlorides, phosphorus oxides

Introduction. Ferrophosphorus is a by-product of the production of elemental phosphorus by the electrothermal method [1]. Each ton of the phosphorus gives from 100 to 600 kg of ferrophosphorus (depending on the amount of iron in a charge and a mode of consumption of electrodes) [2]. It contains 15-28% of phosphorus, 1-15% of Si, 1-15% of Mn, 0.3-2,2% of Ti, 0.2-0.8% of V, up to 2% of C, the rest is Fe [1,3,4]. Ferrophosphorus is used in the production of special grades of steel as an alloying additive and in the foundry industry as an additive to cast iron to improve its casting properties [1].

However, recently, despite the introduction of the grade for ferrophosphorus [3], the problems with its sale have arisen, which are associated with an increase of requirements to the ferrophosphorus quality [5]. For these reasons, at present, there is an excess of electrothermal ferrophosphorus in relation to its need for metallurgy [6]. This problem primarily applies not only to the main world producers of phosphorus: China (84%), Kazakhstan (6%), the USA (5%), the Netherlands, but also to a number of other countries producing phosphorus in a smaller volume (Vietnam, Germany, etc.) [7].In world practice, there are several directions for the processing of ferrophosphorus to obtain iron phosphate, phosphoric acid, monocalcium phosphate and iron oxide, phosphorus and iron oxide, iron phosphate and iron oxide, phosphorus oxides, mineral fertilizers, iron metal and calcium phosphate, ferrosilicon and calcium phosphide, ferrosilicon and phosphorus, phosphorus chloride, iron chlorides and elemental phosphorus [8-16]. From the point of view of enterprises producing phosphorus and its compounds, it is rational to process ferrophosphorus at the same enterprises to obtain phosphorus-containing products as well as other products in demand on the market.

The purpose of this work was to determine the possibility of using iron phosphide Fe2P (the main component of ferrophosphorus) to obtain gaseous phosphorus (V) oxide – a raw material for the production of phosphoric acid [17], the world production market of which will grow by almost 4% per year [18], as well as iron chlorides – reagents for natural and waste water treatment. At the end of the last century, the world annual production of iron chlorides was about 250 thousand tons. Currently, only in Russia, iron chlorides are produced by more than 20 enterprises, for example: LLC SiNOR, LLC LANHI, OJSC Brom, PJSC Brom, Shostka, CJSC Ekros-Engineering, OJSC Khimprom, KEMIRAOYJ, LLC NPF Nevsky Khimik, and also by foreign companies, for example, Hawkins (USA), Biochem (France), Wasser Hygiene Chemie (Germany), Dongda (China).

Methods and materials. A prerequisite for studying the Fe_2P chlorination process was our preliminary calculation of ΔG_T using the HSC-6.0 program (Reaction Equations option) [19] for obtaining phosphorus (V) oxide and iron chlorides according to the reactions:

$$2Fe_2P+4Cl_2+2,5O_2=4FeCl_2+P_2O_5, \quad (1)$$

$$2Fe_2P+6Cl_2+2,5O_2=4FeCl_3+P_2O_5. \quad (2)$$

As follows from table 1, from a thermodynamic point of view, the formation of iron chlorides and P_2O_5 from Fe_2P takes place already at 300^0C.

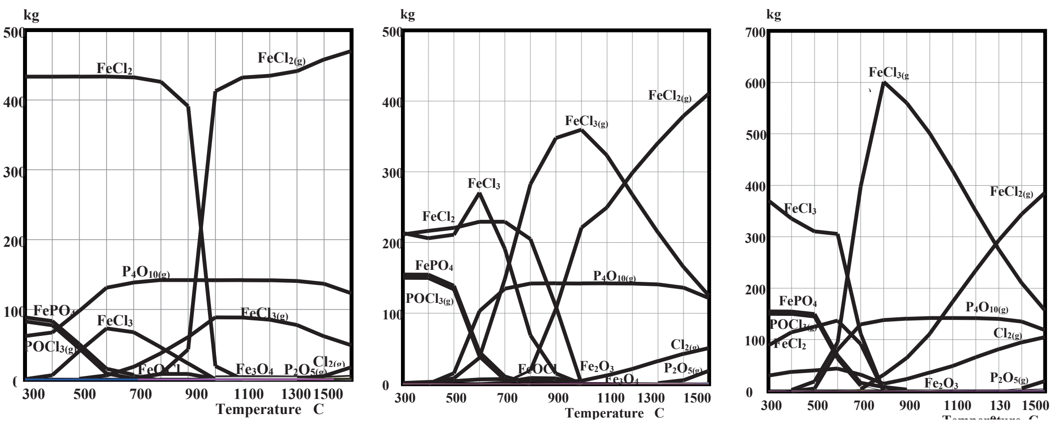
Table 1 – Temperature effect on the changing ΔG^o (kJ) for the reactions of Fe_2P chlorination with chlorine at the presence of oxygen

Reaction #	Temperature, °C						
	300	500	700	900	1100	1300	1500
1	-1382,3	-1380,1	-1377,1	-1373,3	-1368,6	-1363,1	-1356,7
2	-1660,5	-1609,0	-1554,7	-1500,5	-1445,3	-1389,1	-1331,9

A more thorough study of the process was carried out using a complete thermodynamic analysis by means the HSC-6.0 software package developed by the Finnish metallurgical company Outokumpu and based on the Gibbs energy minimization (Equilibrium Compositions option) [19]. The influence of temperature and the amount of chlorine on the behavior of iron and phosphorus in a $2Fe_2P – mCl_2 – 2.5O_2$ system was determined. The equilibrium distribution degrees of phosphorus, iron and chlorine were determined according to the algorithm developed by us, which has the status of intellectual property [20]. During the research, the possibility of the formation of the following compounds in the systems was considered: Fe_3P, Fe_2P, FeP, FeP_2, $FeCl_2$, $FeCl_3$, $FeCl_2$(g), $FeCl_3$(g), FeP, Fe_2O_3, Fe_3O_4, FeOCl, $FePO_4$, gaseous PO, PO_2, P_2O_4, P_2O_5, P_2O_3, P_4O_9, P_3O_6, P_4O_6, P_4O_7, P_4O_8, P_4O_{10}, $POCl_3$, PCl_3,PCl_2, PCl, PCl_5 and also the equilibrium distribution degrees (α, %) of phosphorus, iron and chlorine in these compounds were calculated.

Results and discussion. Fig. 1 shows the effect of temperature and the amount of chlorine (4.0, 5.0 and 6.0 kmol) on the equilibrium quantitative distribution of the substances in the $2Fe_2P – 2,5O_2 – mCl_2$ systems.

a b c



a – 4,0 kmol of Cl_2, b – 5,0 kmol of Cl_2, c – 6,0 kmol of Cl_2

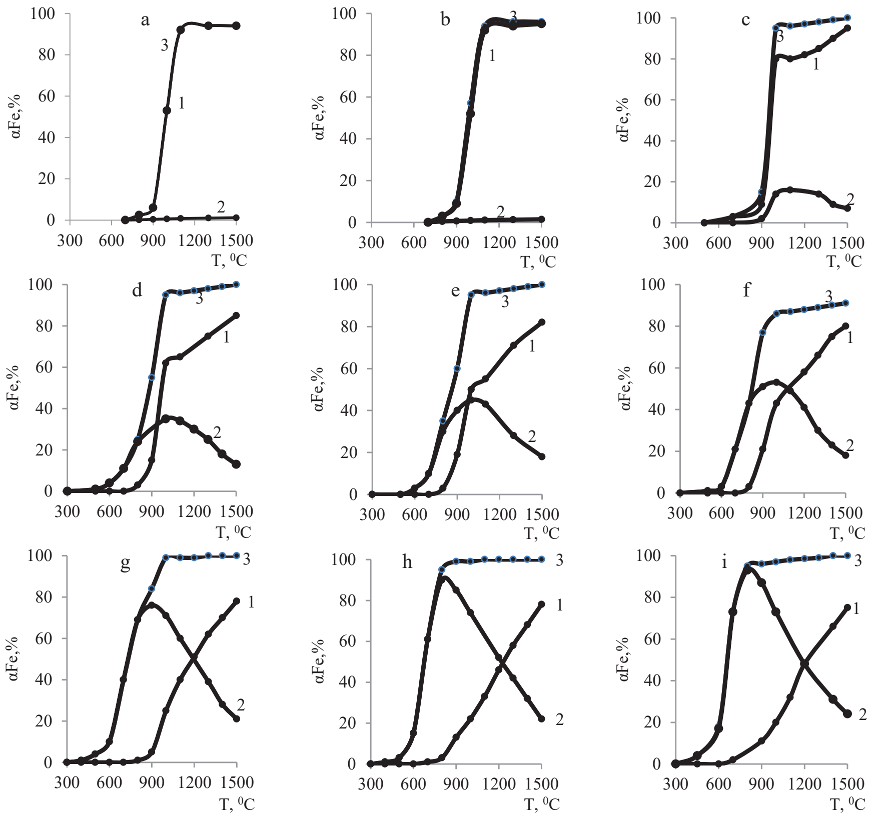
Figure 1 – Effect of temperature and amount of chlorine on the quantitative distribution of the substances in the $2Fe_2P – 2.5O_2 – mCl_2$ system

As follows from the fig.1, in the temperature range of 300-1500°C, depending on the amount of chlorine, the main substances are $FePO_4$, $FeCl_2$, $FeCl_3$, FeOCl, Fe, Fe_2O_3, $POCl_3$, P_4O_{10}, and P_2O_5. In addition, iron phosphate is formed at 300°C, and its quantity increases with an increase in the chlorine amount from 4.2 to 6.0 kmol. Gaseous iron chlorides ($FeCl_2$ and $FeCl_3$) in the systems are in the temperature range of 400-1500°C. Moreover, at first (at the low temperatures) $FeCl_3$ is formed, and then $FeCl_2$. The beginning temperature of the iron chlorides formation (i.e. the temperature of 1% of their formation) T_B depends on the amount of chlorine introduced in the system (Table 2) and decreases as the chlorine amount increases.

Table 2 – Effect of the chlorine amount on the temperature of the beginning of the $FeCl_2$ and $FeCl_3$ formation

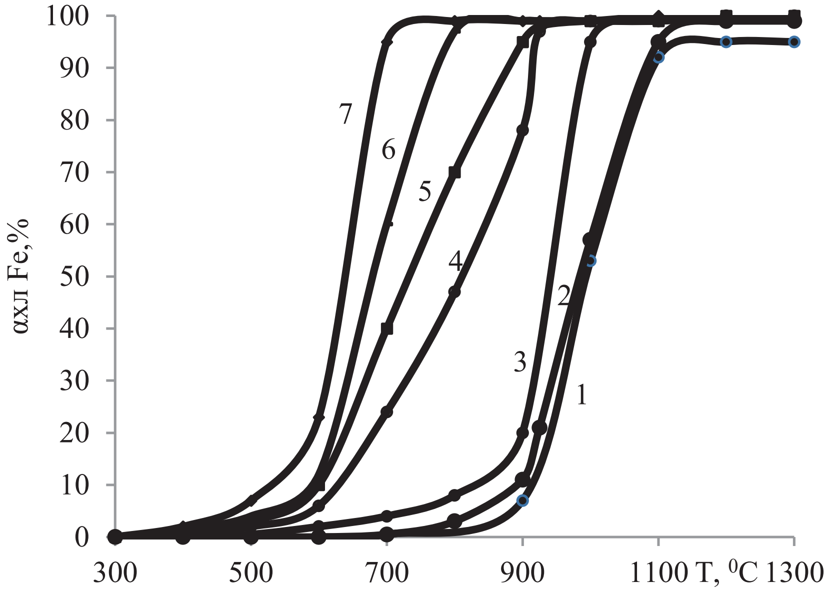
Cl_2, kmol	4	4,2	4,5	4,7	4,9	5,1	5,6	6	6,25
$T_B(FeCl_2)$, °C	804	756	744	732	724	717	712	707	705
$T_B(FeCl_3)$, °C	777	604	545	522	509	474	440	427	414

The temperature and chlorine amount effect on the equilibrium iron distribution degree in gaseous chlorides is represented in Fig.2.



1 – $FeCl_2$, 2 – $FeCl_3$, 3 – $FeCl_2$+$FeCl_3$
m values: a-3.8, b-4.0, c-4.3, d-4.7, e-4.9, f-5.1, g-5.6, h-6.0, i-6.25
Figure 2 – Effect of temperature and amount of chlorine (m, kmol) on the iron distribution degree in gaseous iron chlorides in the $2Fe_2P – 2.5O_2– mCl_2$ system.

The maximum of the curve of the iron distribution degree (α) in gaseous $FeCl_3$ (Fig. 2) is associated with the decomposition of $FeCl_3$ into $FeCl_2$ and Cl_2 [21]. The maximum iron transition into gaseous $FeCl_3$ increases to 95.5% at 900°C and changing the chlorine amount in the system from 3.8 to 6.25 kmol. On the contrary, the maximum of the iron transition into gaseous $FeCl_2$ at the growth in the amount of chlorine decreases at 1500°C from 96.9% to 73.5%. The total iron transition degree from Fe_2P into gaseous $FeCl_2$ and $FeCl_3$ (α_{chl}Fe) increases with the growth of temperature and chlorine amount (Fig. 3). Thus, at 1000 °C, the increase in the amount of chlorine from 4 to 6.7 kmol leads to the increase in the total iron transition into its gaseous chlorides from 57.2% to 99.5%.



m values: 1 – 3.8, 2 – 4.0, 3 – 4.2, 4 – 5.1, 5 – 5.6, 6 – 6.0, 7 – 6,7

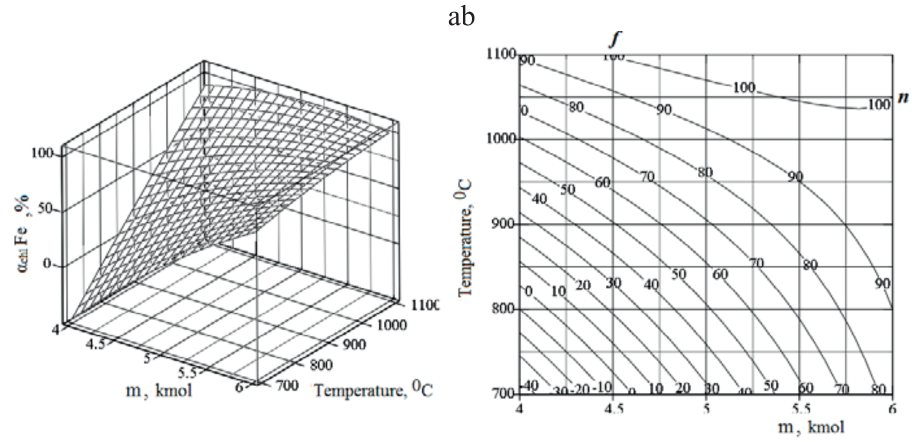
Figure 3 – Effect of temperature and amount of chlorine (m, kmol) on the total iron distribution degree into gaseous $FeCl_2$ and $FeCl_3$ for the $2Fe_2P – 2.5O_2 – mCl_2$ system.

Based on the data in Fig. 3, using the second-order rotatable planning technique [22], a two-factor research matrix was developed. The matrix independent variables were the amount of chlorine (m, kmol) and temperature (T, °C), the output parameter was the total iron distribution degree in a kind of gaseous $FeCl_2$ and $FeCl_3$. On the basis of the matrix results, the following adequate regression equation was obtained:

$$\alpha_{chl}Fe= -1238,35 + 258,89\cdot m +1,08\cdot T – 8,78\cdot m^2 – 7.02\cdot 10^{-5}\cdot T^2 – 0,15\cdot m\cdot T \qquad (3)$$

Using this equation according to the method [23], volumetric and planar dependences $\alpha_{chl}Fe$ =f(T, m) were constructed. Fig.4 shows the influence of the amount of chlorine and temperature on the shape of the response surface – α_{chl} Fe and its horizontal sections. As follows from the fig.4, the complete iron chlorides formation can be realized along the technological line *fn*, i.e. in the range from 4.5 to 6.0 kmol of chlorine in the temperature interval from 1040 to 1100°C.

ab

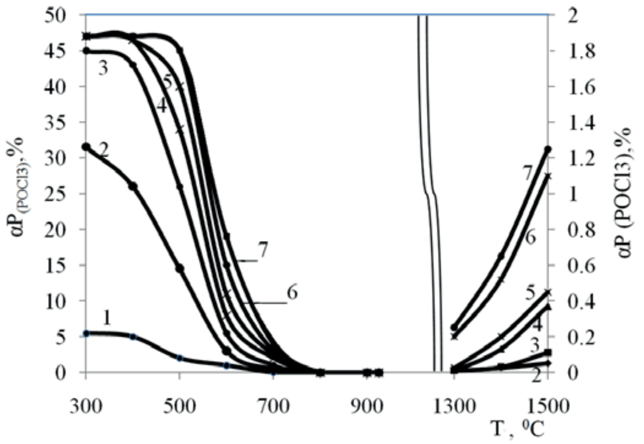


a – response surface, b – horizontal sections of the response surface

Figures on the lines – α_{chl} Fe, %.

Figure 4 – Effect of temperature and chlorine amount on the shape of the response surface $\alpha_{chl}Fe$ and its horizontal sections.

The next stage of the research was to determine the conditions of maximum phosphorus transition into its gaseous oxide (V). The curve of the phosphorus transition from Fe_2P into the unwanted substance $POCl_3$, which pollutes the gaseous phosphorus (V) oxide, depending on the temperature has the minimum in the temperature range of 850-900°C (Fig. 5).

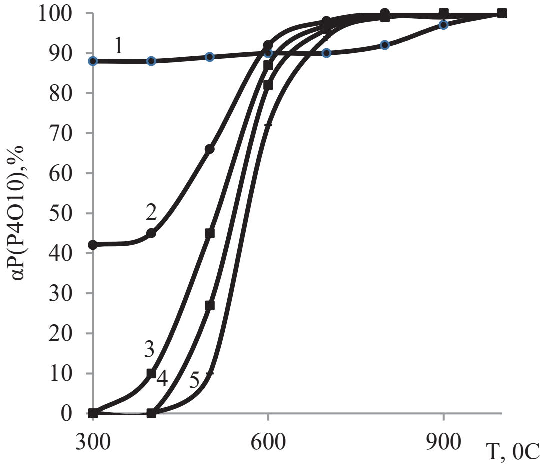


m values: 1 – 4.0, 2 – 4.2, 3 – 4.5, 4 – 4.7, 5 – 4.9, 6 – 5.6, 7 – 6.0

Figure 5 – Effect of temperature and chlorine amount (m, kmol) on the phosphorus transition degree into gaseous $POCl_3$ in the $2Fe_2P – 20.5O_2 – mCl_2$ system.

In the temperature interval of 300-900°C, as the amount of chlorine increases, the phosphorus transition degree into $POCl_3$ increases to its maximum value (48.8%) at 300°C and the amount of chlorine from 4.7 to 6 kmol. Then, at the temperature of more than 900-1000°C, the formation of secondary $POCl_3$ occurs. It is especially noticeable at m>4.9, for example, 1.25% at 1500°C and 6 kmol of chlorine.

The transition of phosphorus into the target product – gaseous P_4O_{10} – takes place at T ≥ 300-400°C (Fig. 6).



1 – m=4.0, 2 – m=4.2, 3 – m=4.5, 4 – m=4.9, 5 – m=6.0

Figure 6 – Effect of temperature and amount of chlorine (m, kmol) in the $2Fe_2P – 2.5O_2 – mCl_2$ system on the phosphorus transition degree into P_4O_{10}

As follows from Figure 6, the phosphorus transition degree into P_4O_{10} ($\alpha P(P_4O_{10})$) increases at the decrease in the amount of chlorine. In the temperature interval of 600-800 °C and at 4-6 kmol of Cl_2, the equation of $\alpha_P (P_4O_{10})$ = f(m, T) has the form:

$$\alpha_{P(P4O10)}=90.03-60.23\cdot m+0.38\cdot T-6.12\cdot 10^{-2}\cdot m^2-4.56\cdot 10^{-4}\cdot T^2+7.65\cdot 10^{-2}\cdot T\cdot m. \quad (4)$$

Based on this equation, the response surface $\alpha P(P_4O_{10})$=f(m, T) and its horizontal sections were constructed (Fig. 7). The complete phosphorus transition degree into P_4O_{10} (≥99.5%) can be achieved in the ***abcd*** technological area when *m* is 4-6 and T ≥ 680-800°C.

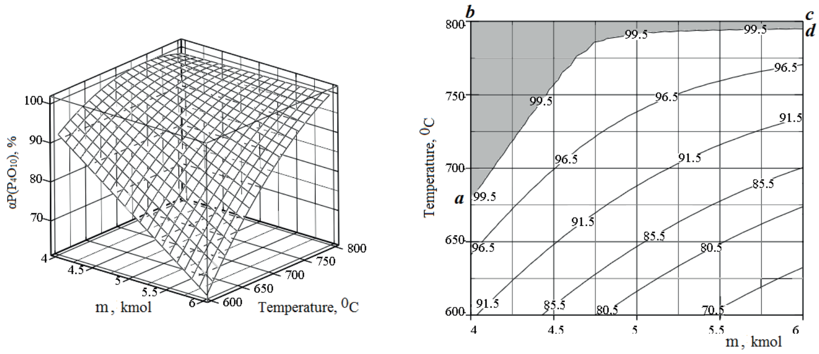
The comparison of the temperature effect on $\alpha_{chl}Fe$ and $\alpha P(P_4O_{10})$ shows that during the interaction of Fe_2P with chlorine and oxygen a restraining factor of the complete conversion of iron into gaseous iron chlorides and phosphorus into gaseous P_4O_{10} is the extraction of iron into iron chlorides, because it takes place at a higher (by 220-260⁰C) temperature.

To use gaseous P_4O_{10} for manufacturing phosphoric acid, a minimum transition of chlorine to the gas is required. An adequate regression equation of the temperature and chlorine amount effect on the degree of its residual transition into the gas phase is:

$$\alpha_{Cl_2} = 195{,}101\text{-}48{,}322\cdot m\text{-}0{,}179\cdot T\text{+}3{,}283\cdot m^2\text{-}4.77\cdot 10^{-5}\cdot T^2\text{+}1.938\cdot 10^{-2}\cdot T\cdot m; \qquad (5)$$

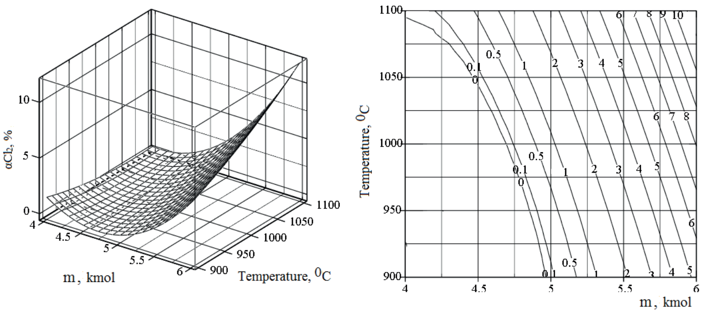
Based on equation 5, a graphical representation of the change in was constructed (Fig.8). As it follows from the figure, the loss of the initial amount of chlorine with the gas phase will be absent when it increases from 4 to 4.9 kmol with a decrease in the temperature from 1090 to 900°C. The chlorine losses to 0.1% will be at 4 kmol and 1125°C, and in the case of an increase in the chlorine amount to 5 kmol - at 900°C.

ab



a – response surface, b – horizontal sections of the response surfaces
Figures on lines – $\alpha P_{(P4O10)}$, %
Figure 7 – Effect of temperature and chlorine amount in the $2Fe_2P – 2.5O_2 – mCl_2$ system on the phosphorus transition degree into P_4O_{10}

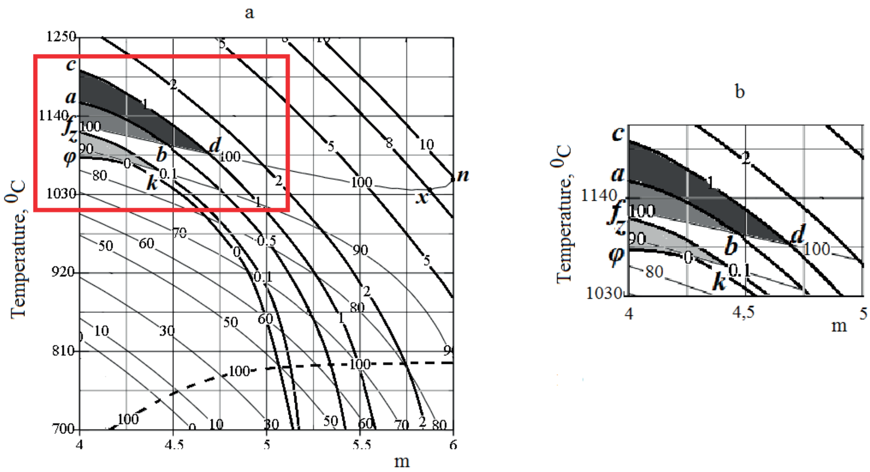
a b



a – response surface, b – horizontal sections of the response surfaces
Figures on lines– αCl_2, %
Figure 8 – Effect of temperature and chlorine amount in the $2Fe_2P – 2.5O_2 – mCl_2$ system on the chlorine transition degree in the gas phase.

To determine the optimal parameters of Fe_2P chlorination with maximum transition of phosphorus into P_4O_{10}, iron into gaseous chlorides as well as at maximum losses of chlorine with the gas we have combined the $\alpha P_{(P4O10)}$, $\alpha_{chl}Fe$ and isolines in one figure. Fig. 9 shows that in the $2Fe_2P – mCl_2 – 2.5O_2$ system it is impossible to completely transfer phosphorus into P_4O_{10} and iron into gaseous iron chlorides with the full use of chlorine, because the line= 0.0% does not intersect with the line of the complete iron – gaseous iron chlorides transition *fn*. The complete extraction of iron into the iron chlorides and the residual chlorine transition degree into the gas phase of no more than 0.5% take place in the *fab* area (T=1100-1160°C and m=4-4.5 kmol). The expansion of the temperature interval to 1280°C and the m range from 4 to 4.75 kmol is possible (*fcd* area), but in this case the upper limit of rises to 1%.

The minimum temperature, at which iron is completely converted to iron chlorides and phosphorus to P_4O_{10}, is 1040°C (point *x* on the *fbdn* line). However, in this case, ≈8% of chlorine is not used and passes into the gas phase together with P_4O_{10} and iron chlorides, complicating the subsequent separation of Cl_2 and P_4O_{10}. To obtain the minimum (0-0.1%) chlorine transition degree to the gas phase and the complete phosphorus transition degree to P_4O_{10}, the process must be carried out in the technological area *zφk* (Fig. 9(b)), in which the amount of chlorine is 4-4.3 kmol, and the temperature is 1090-1125°C. In this case, the iron chloride sublimation degree will be 90-91.8%.



( )- α_{chl}Fe, %, (- - -)$\alpha P_{(P4O10)}$, %, ( )αCl_2, %
a– at 700-1250^{0}C and 4-6 kmol of Cl_2, b – at 1030-1230 ^{0}C and 4-5 kmol of Cl_2
Figure 9 – Combined information on the effect of temperature and chlorine amount in the $2Fe_2P – 2.5O_2 – mCl_2$ system on the phosphorus, iron and chlorine distribution degrees.

The proposed method makes it possible to produce up to 650 kg of orthophosphoric acid [17], as well as up to 600 kg of $FeCl_2$ and 900 kg of $FeCl_3$ using 1 ton of ferrophosphorus containing 21% of phosphorus and 70% of iron.

The 5-6 ths t of ferrophosphorus annually generated by LLP "Kazphosphate" can additionally give 3.25-3.9 ths t of orthophosphoric acid and up to 7.5-9ths t of iron chlorides.

Conclusion. The results of modeling the interaction in the $2Fe_2P – 2.5O_2 – mCl_2$ system allow us to draw the following conclusions:

- the beginning temperature of the target gaseous iron chlorides formation decreases from 804 to 693°C (for $FeCl_2$) and from 777 to 412°C (for $FeCl_3$)at increasing the chlorine amount from 4.0 to 6.7 kmol; the complete chloride sublimation of iron occurs at 4.5 to 6.0 kmol of chlorine in the temperature range from 1040 to 1100°C, and full extraction of phosphorus from Fe_2P to P_4O_{10} takes place in the temperature interval of 730-840°C and 4.0-6.0 kmol of chlorine;
- to achieve the complete transition of phosphorus from Fe_2P to gaseous P_4O_{10} and minimal (0,0-0,1%) losses of chlorine with the gas, the Fe_2P chlorination should be implemented in the temperature range of 1090-1125°C; in this case the iron extraction degree from Fe_2P in the gaseous chlorides is 91.8%.

The processing of 1 ton of ferrophosphorus containing 21% of phosphorus and 70% of iron in accordance with the proposed method makes it possible to produce up to 650 kg of orthophosphoric acid, as well as up to 600 kg of $FeCl_2$ and 900 kg of $FeCl_3$.

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ФОСФОР (V) ОКСИДІ МЕН ТЕМІР (II, III) ХЛОРИДТЕРІНІҢ АЛЫНУЫМЕН ТЕМІР ФОСФИДІН ОТТЕГІНІҢ ҚАТЫСУЫМЕН ХЛОРМЕН ХЛОРЛАУ

Аннотация. Мақалада электротермиялық феррофосфордың негізі болып табылатын Fe_2P фосфор (V) оксиді мен темір (II, III) хлоридтерін өндіруді болжау бойынша жүргізілген зерттеулердің нәтижелері келтірілген. Зерттеу әдістері - Гиббстың минималды энергиясы мен екінші ретті рототабелді жоспарлауға (Бокс-Хантер жоспары) негізделген HSC - 6.0 бағдарламалық кешенін қолданумен термодинамикалық модельдеу. Жұмыстың мақсаты темір фосфидінен темір тәрізді хлоридтер мен фосфор (V) оксидін алудың термодинамикалық мүмкіндігін анықтау болып табылады. 1 бар қысымда $2Fe_2P - mCl_2 - 2,5O_2$ жүйесіндегі темірдің, фосфордың өзгеруіне 300-1500^{0}C аралықтағы температура мен мен хлор мөлшерінің әсері анықталды. Темір хлоридінің хлорлы айдалуының басталу температурасы хлордың мөлшеріне байланысты хлор мөлшерінің 4,0 -ден 6,7 кмольге дейін ұлғаюынан 804-тен 693°С-қа дейін ($FeCl_2$ үшін) және 777-ден 412°С-қа дейін ($FeCl_3$ үшін) төмендейді; Темірдің хлорлы айдалуының толық дәрежесі хлордың 4,5-тен 6,0 кмоль мөлшерінде 1040-тан 1100°С-қа дейінгі температура аралығында, ал фосфордың Fe_2P-ден газ тәрізді P_4O_{10}-ға өтуі- 730-840°С және 4,0-6, 0 кмоль хлор мөлшерінде болады; хлордың газ фазасына өтуінің минималды дәрежесі (0,1% дейін) фосфордың P_4O_{10}-қа толық өтуі кезінде темірдің Fe_2P-ден газды хлоридтерге 91,8% бөліну дәрежесінде, 4,0-4,3 кмоль хлор мөлшерінде, 1090 -1125°С температуралық аралықта байқалады.

Түйінді сөздер: феррофосфор, темір фосфиді, хлорлау, хлор, оттегі, термодинамикалық модельдеу, темір хлориді, фосфор оксидтері.

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ХЛОРИРОВАНИЕ ФОСФИДА ЖЕЛЕЗА ХЛОРОМ В ПРИСУТСТВИИ КИСЛОРОДА С ПОЛУЧЕНИЕМ ОКСИДА ФОСФОРА(V)И ХЛОРИДОВ ЖЕЛЕЗА (II, III)

Аннотация. В статье приводятся результаты исследований по прогнозированию получения оксида фосфора (V) и хлоридов железа (II, III) из Fe_2P, являющегося основой электротермического феррофосфора. Методы исследований-термодинамическое моделирование с использованием программного комплекса HSC - 6.0, основанного на минимуме энергии Гиббса и рототабельное планирование второго порядка (план Бокса - Хантера). Цель работы заключалась в определении термодинамической возможности получения из фосфида железа газообразных хлоридов железа и оксида фосфора (V). Определялось влияние температуры в интервале 300-1500^{0}С и количество хлора на поведение железа, фосфора в системе $2Fe_2P – mCl_2 – 2,5O_2$ при давлении 1 бар. Установлено, что температура начала хлоридовозгонки железа зависит от количества хлора, уменьшаясь от 804 до 693°С (для $FeCl_2$) и от 777 до 412°С (для $FeCl_3$) при увеличении количества хлора от 4,0 до 6,7 кмоль; полная степень хлоридовозгонки железа происходит при 4,5 до 6,0 кмоль хлора, в температурном интервале от 1040 до 1100°С, а переход фосфора из Fe_2P в газообразный P_4O_{10} - в температурном интервале 730-840°С и 4,0-6,0 кмоль хлора; минимальная степень перехода хлора в газовую фазу (до 0,1%) при полном переходе фосфора в P_4O_{10} со степенью извлечения железа из Fe_2P в газообразные хлориды на 91,8% отмечается при 4,0-4,3 кмоль хлора, в температурном интервале 1090-1125°С

Ключевые слова: феррофосфор, фосфид железа, хлорирование, хлор, кислород, термодинамическое моделирование, хлориды железа, оксиды фосфора.

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