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BEHAVIOR OF THE COMPONENTS OF CARBON-CONTAINING ASH FROM THE COMBUSTION OF POWER COALS UNDER THE CONDITIONS OF CHLORINATION ROASTING

On the basis of thermodynamic calculations of interaction reactions between mullite, oxides of titanium, iron, and Cl_2 (gas) in the presence of carbon, the values of the Gibbs free energy are determined in the current work. It has been established that the probability of reactions occurring in the temperature range 1073–1473 K is very high. The most negative value $\Delta G^\circ_T = -1029.9$ kJ/mol is typical for the titanium oxide reduction reaction at the roasting temperature (1100 °C). The value of the Gibbs free energy of the destruction reaction of mullite (Al_2SiO_5), which is important for practice, is $\Delta G^\circ_T = -269.2$ kJ/mol. This makes it possible to predict the complete decomposition of mullite from ash to easily soluble aluminum chloride under the conditions of chlorination roasting.

The study of the behavior of carbon under the conditions of chlorination roasting of ash with $CaCl_2$ in an oxidizing atmosphere is also important. The results of the differential thermal analysis of the non-magnetic fraction of ash, previously separated from the TPP ash by magnetic separation, showed clearly defined manifestations on the DTA, DTG and TG curves, caused by the presence of various types of reactions in the system. Low-temperature (20–115 °C) sample dehydration and ash dehydration were determined in the temperature range of 115–375 °C. In the range of further heating of the sample (375–685 °C), an exothermic projection was found on the DTA curve, which is characteristic of the oxidation of thermally active carbon present in the powder. The amount of carbon removed from the ash established as 6.75% by weight of the sample.

Keywords: *ash, carbon, roasting, thermodynamics, Gibbs free energy, chlorine, calcium chloride, differential thermal analysis*
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Introduction

An analysis of modern literature shows that the issue of processing accumulated and current ash and slag waste from TPPs obtained from coal combustion, for the most part, goes in the direction of their use in the construction industry [1]. In most cases, ash contains, % (wt.): SiO_2 50–58; $A1_2O_3$18–25; Fe_2O_3 11–17; K_2O 2.3–4.1; Na_2O 0.5–1.35; TiO_2 0.9–1.1; CaO 1.5–3.7; MgO 1.7–3.1; P_2O_5 0.091.70; S 0.6–0.5; Cl 0.01–0.11. Aluminum is predominantly present in the ash in the form of mullite (Al_2SiO_5) [2–4], which is a very stable alumina-silicate compound.

The use of ash and slag waste from thermal power plants brings the technology of heat and energy generation by burning coal closer to its wastelessness by 80%.

Currently, new technologies for ash processing are being developed [5–9], which, due to the sharp demand for REE, have begun to be applied in the direction of extracting small amounts of these metals. From the point of view of the complexity of the use of raw materials, this approach to ash processing seems to be inefficient. According to the material composition, coal ash can be considered not only as a separate complex raw material for non-metallic metals, but also as a raw material for extracting a complete complex of valuable metals from it.

The problem of accumulation and storage of ash has a particular relevance for Kazakhstan, where the development of electricity generation and processing of TPP waste is one of the main state priorities. The annual output of ash and slag mixtures in the republic from coal combustion is about 19 *million tons*, and more than 300 *million tons* of waste have been accumulated in ash dumps to date [10].

The paper [11] presents the theoretical features of the technology, in terms of roasting ash with $CaCl_2$, and the behavior of its components during roasting and leaching of cinder. Expanding the boundaries of theoretical knowledge of the technology requires an additional comprehensive study of the behavior of carbon contained in the ash and its effect on the technological parameters of the process. The study of the above issue has a fundamental importance for practice, since a significant amount of carbon remains in fly ash and slag obtained after coal combustion. Thus, according to [12], the carbon content in ash and slag obtained after burning coal from the Karazhyra deposit is ~29 and 19%, respectively. This indicates a large "underburning" and loss of the active mass of thermal coal. It is quite obvious that when developing a new technology for processing ash, such a high carbon content cannot be neglected, and it is necessary to evaluate its effect on the overall mechanism of the process.

The purpose of this work is to study and investigate the behavior of carbon and its influence on the technological parameters of the roasting process under conditions of ash chlorination with gaseous chlorine and calcium chloride in an oxidizing atmosphere.

Research methods

The assessment of the probable direction of reactions occurring between the ash components and gaseous chlorine and $CaCl_2$ in the presence of carbon was carried out by changing the thermodynamic values of the system. Thermodynamic analysis was related to determining the Gibbs free energy and establishing its temperature dependence.

When calculating the Gibbs free energy (ΔG°_T) of the expected reactions, the standard state of the mineral-forming compounds present in the ash, as well as quartz and iron oxides, is taken as "pure solid" (s). For gaseous compounds, gas (g) is taken as the standard state. When carrying out thermodynamic calculations in relation to the conditions of chlorinating roasting (1000 °C), "pure liquid calcium chloride" is taken as the standard stateconsidering that the melting point of calcium chloride is 772 °C. The initial data for calculations were obtained from [13].

Thermodynamic calculations of the reactions taken for analysis were carried out using a special program developed by the authors. The calculations used the results of the forms of aluminum, silica and iron in the original ash.

As an object of study, an average sample of ash from TPP-2 (Almaty) was used.

Table 1. Mean values of elemental composition of ash

Elements	Al	Si	Fe	Ti	Mg	K	Ca	Mn	O	Na	Oth.
Content, % wt.	14.12	28.15	4.33	0.67	0.52	0.86	1.84	0.15	47.10	0.56	1.62

Table 3. Estimated elemental composition of ash

Phasename	Content, % wt.							Total:
	Al	**Si**	**Fe**	**Ca**	**Na**	**K**	**O**	
Mullit $Al_{4.984}Si_{1.016}O_{9.508}$	9.12	2.00					10.67	**21.79**
Quartz SiO_2		17.09					19.47	**36.56**
Hematite Fe_2O_3			1.08				0.47	**1.55**
Anorthite PSCa$(Al_2Si_2O_8)$	2.87	3.09		2.20			7.04	**15.20**
Microcline KPS $(K_{0.95}Na_{0.05})AlSi_3O_8$	1.71	5.52			0.75	2.43	8.38	**18.80**
Magnetite Fe_3O_4			4.42				1.68	**6.10**
Total:	**13.70**	**27.70**	**5.50**	**2.20**	**0.75**	**2.43**	**47.71**	**100**

Table 2. Results of semi-quantitative X-ray phase analysis of ash

Phasename	Formula	Concentration, %
Mullit	$Al_{4.984}Si_{1.016}O_{9.508}$	21.79
Quartz	SiO_2	36.56
Hematite	Fe_2O_3	1.55
Anorthite PS	$Ca(Al_2Si_2O_8)$	15.20
Microcline	$(K_{.95}Na_{.05})AlSi_3O_8$	18.80
Magnetite	Fe_3O_4	6.10

The elemental composition of the initial ash samples was determined by X-ray spectral microanalysis using a Super Probe 733 electron probe microanalyzer (Jeol, Japan). Analysis of the elemental composition of the samples and photography in various types of radiation were performed using an Inca Energy energy dispersive spectrometer (Oxford Instruments, England).

X-ray diffraction analysis was carried out on a DRON-3 diffractometer with Cu Ka-radiation, β-filter. Conditions for taking diffraction patterns: U=35 kV; I=20 mA; shooting θ–2θ; detector 2 deg/min. X-ray phase analysis on a semi-quantitative basis was performed on the basis of powder samples diffraction patterns using the method of equal weights and artificial mixtures. Quantitative ratios of crystalline phases were determined. The interpretation of the diffraction patterns was carried out using the data from the ICDD file cabinet: the PDF2 (Powder Diffraction File) database of powder diffraction data and the diffraction patterns of minerals free of impurities.

Thermal analysis (DTA and TGA) of the non-magnetic ash fraction, previously separated from the original ash by magnetic separation, was performed on a MOM derivatograph (Budapest, Hungary). The method used is based on the registration by the device of changes in the thermochemical and physical parameters of a substance that can be caused when it is heated.

Results and discussion

The mean values of the elemental composition of ash obtained from measurements of 3 ash samples using a Super Probe 733 electron probe microanalyzer (Jeol, Japan) is shown in **Table 1**.

The results of the EDS analysis of the original ash (**Fig. 1**) show the presence of a high carbon content (10.84% wt.), which confirms the presence of “underburning” of the coal.

The bar diagram of X-ray diffraction analysis of ash on a DRON-3 diffractometer (**Fig. 2**), and the results of semi-quantitative analysis (**Table 2**) showed that the dominant phases in the ash are: 22% mullite ($Al_{4.984}Si_{1.016}O_{9.508}$), ~37 % quartz and 1.6% hematite (Fe_2O_3).

Taking into account the obtained results of the elemental and phase composition of the ash, its elemental composition was calculated, which is shown in **Table 3**.

Comparative analysis of the results of the measured elemental composition of ash (see Table 1), with the data of the calculated composition (see Table 3) shows good agreement with each other, which indicates a high reliability of the results obtained by independent research methods.

Thermodynamic analysis of reactions of interaction of ash components with Cl_2 in the presence of carbon

The general mechanism of the process of roasting ash with gaseous chlorine in the presence of carbon can be described by a system of reactions:

$$Al_2SiO_5\ (s) + 3Cl_2\ (g) + 1.5C\ (s) = 2AlCl_3\ (g) + SiO_2\ (s) + 1.5CO_2\ (g),$$
$$\Delta G^{o}_{T} = -191.895\text{-}0.053\times T. \qquad (1)$$

$$TiO_2\ (s) + 2Cl_2\ (g) + C\ (s) = TiCl_4\ (g) + CO_2\ (g),$$
$$\Delta G^{\circ}_{T} = -1008.327\text{-}0.016\times T. \qquad (2)$$

$$Fe_3O_4\ (s) + 4.5Cl_2\ (g) + 2C\ (s) = 3FeCl_3\ (g) + 2CO_2\ (g),$$
$$\Delta G^{\circ}_{T} = -829.749+0.254\times T. \qquad (3)$$

$$FeO\ (s) + 1.5Cl_2\ (g) + 0.5C\ (g) = FeCl_3\ (g) + 0.5CO_2\ (g),$$
$$\Delta G^{\circ}_{T} = -311.164+0.12\times T. \qquad (4)$$

$$Fe_2O_3\ (s) + 3Cl_2\ (g) + 1.5C\ (s) = 2FeCl_3\ (g) + 1.5\ CO_2\ (g),$$
$$\Delta G^{\circ}_{T} = -542+0.12\times T. \qquad (5)$$

In reactions with chlorine (1)–(5) oxygen is not present, since experiments with chlorine were carried out in a stream of pure gaseous chlorine [11].

Figure 3 shows the dependence of the change in the Gibbs free energy for reactions (1)–(5) in the temperature range 1073÷1473 K, typical for the conditions of roasting ash with chlorine in the presence of carbon.

It can be seen that the probability of mullite decomposition by chlorine in the presence of carbon according to reaction (1) is quite high, and is accompanied by the formation of $AlCl_3$ and the release of CO_2. The Gibbs free energy of reaction (1) increases with increasing temperature, and is characterized by a high negative value throughout the studied temperature range: $\Delta G_{1073\ K} = -248.04$ kJ/mol; $\Delta G_{1373\ K} = -269.2$ kJ/mol.

The probability of reactions (2)–(5) occurring in the studied temperature range is also high, and the most negative value of the Gibbs free energy at a roasting temperature of 1373 K is observed for reaction (2), $\Delta G_{1373\ K} = -1029.9$ kJ/mol.

Chlorination of iron oxides in the presence of carbon according to reactions (3), (4), (5) shows a high activity of transferring iron into a readily soluble chloride form ($FeCl_3$) with the release of CO_2. The values of the Gibbs free energy of reactions (3), (4) and (5) at a roasting temperature of 1373 K are $\Delta G_{1373\ K} = -480.96$ kJ/mol; $\Delta G_{1373\ K} = -145.64$ kJ/mol; $\Delta G_{1373\ K} = -377.48$ kJ/mol, respectively.

Obtained results show that the highest probability of reactions (1)–(5) can be expected immediately after gaseous chlorine is injected into the reactor, which makes it possible to selectively isolate metal chlorides in the condensed phase (cinder). The presence of carbon in the ash, as shown by the results of thermodynamic calculations, enhances the reduction activity of sparingly soluble mullite, iron and titanium oxides according to reactions (1)–(5). Then carbon is removed from the reaction zone into the gas phase in the form of CO_2.

Under the conditions of chlorination roasting, the formation of dioxins is possible, but at the roasting temperature (1000 °C) they thermally decompose ("dioxin is a crystalline substance with a high melting point (305 °C) and very low volatility, poorly soluble in water (2x10–8% at 25 °C) and better – in organic solvents. It is characterized by high thermal stability: its decomposition is noted only when heated above 750 °C, and is effectively carried out at 1000 °C").

Behavior of carbon under conditions of oxidative roasting of ash with $CaCl_2$

The thermodynamics of the "ash – $CaCl_2$ – SiO_2 – O" system is considered in detail in [11]. Required amount of both chlorine and calcium chloride is determined by the composition of the ash, depending on the amount of SiO_2, CaO, Al_2O_3 in the ash. The amount of added reagents determines the ratio of anorthite and gelenite in the obtained cinder.In contrast to chlorinated ash roasting with chlorine, under the conditions of ash roasting with $CaCl_2$ in an oxidizing atmosphere, it seems that it is not worth expecting a significant effect of carbon on the general mechanism of the chlorinating roasting process. It can be assumed that it is oxidized due to atmospheric oxygen with release into the gas phase already at the initial stage of ash heating to a given roasting temperature. To confirm this assumption, we studied the process of thermal heating of ash using the method of differential thermal analysis.

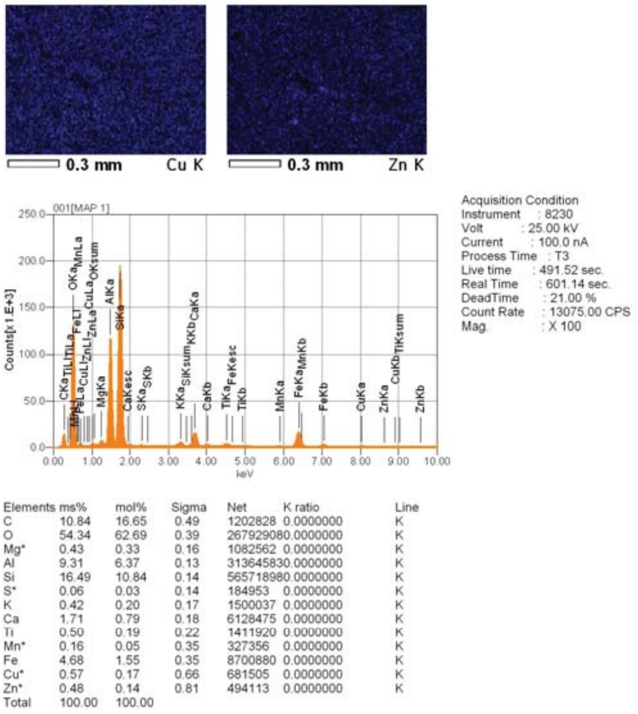
As a result of dynamic heating of the ash, the DTA, DTG and TG curves manifestations revealed caused by various types of reactions occurring in the system. Low-temperature (20–115 °C) sample dehydration was determined, as well as ash dehydration in the range of 115–375 °C. In addition, the thermal curves revealed residual carbon in the ash and the presence in it (in small quantities) of calcium carbonate and dolomite.

The presence of mechanically bound water in the powder was established from the endothermic peaks on the DTA and DTG curves detected at 105 and 75 °C, respectively. In turn, the thermogravimetric (TG) line in the marked temperature range described a clearly defined step of mass loss, equal to the release into the atmosphere of 0.45% of adsorbed water from the weight of the mass of the original sample (**Fig. 4**).

A further decrease in the mass of the investigated feed in the range of 115–375 °C refers to the removal of differently bound water (H_2O and OH^-) introduced into the system after coal roasting. The amount of such water relative to the mass of the sample corresponds to 0.95%.

Subsequent heating of the sample leads to the oxidation of the thermally active component present in the powder. According to the morphology of the exothermic change on the DTA curve in the range of 375–685 °C, the indicated active substance refers to the part of carbon, the amount of which in the sample according to thermogravimetric data (see Fig. 4) is 6.75% of the sample mass. Along with the identified inclusions, technogenic calcite and dolomite were found in the composition of the sample, which left two poorly developed endothermic peaks at 720 and 750 °C at the top of the considered exothermic effect. According to the degree of loss (Δm_4) as a result of the destruction of these minerals, the amount of these carbonates in the sample in total does not exceed 1.5% of the mass of the sample.

The results of the study of the elemental composition of the cinder [9] and the EDS analysis of the cinder sample (**Fig. 5**) obtained after roasting the ash with $CaCl_2$ in an oxidizing atmosphere (T = 1100 °C, τ = 60 min), fully confirm the established patterns of carbon behavior during heating the ash. No carbon was found in the cinder, which indicates complete combustion and its volatilization in the form of CO_2 with the exhaust gases.



Acquisition Condition
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Process Time : T3
Live time : 491.52 sec.
Real Time : 601.14 sec.
DeadTime : 21.00 %
Count Rate : 13075.00 CPS
Mag. : X 100

Elements	ms%	mol%	Sigma	Net	K ratio	Line
C	10.84	16.65	0.49	1202828	0.0000000	K
O	54.34	62.69	0.39	26792908	0.0000000	K
Mg*	0.43	0.33	0.16	1082562	0.0000000	K
Al	9.31	6.37	0.13	31364583	0.0000000	K
Si	16.49	10.84	0.14	56571898	0.0000000	K
S*	0.06	0.03	0.14	184953	0.0000000	K
K	0.42	0.20	0.17	1500037	0.0000000	K
Ca	1.71	0.79	0.18	6128475	0.0000000	K
Ti	0.50	0.19	0.22	1411920	0.0000000	K
Mn*	0.16	0.05	0.35	327356	0.0000000	K
Fe	4.68	1.55	0.35	8700880	0.0000000	K
Cu*	0.57	0.17	0.66	681505	0.0000000	K
Zn*	0.48	0.14	0.81	494113	0.0000000	K
Total	100.00	100.00				

Fig. 1. Results of the EDS analysis of the initial ash

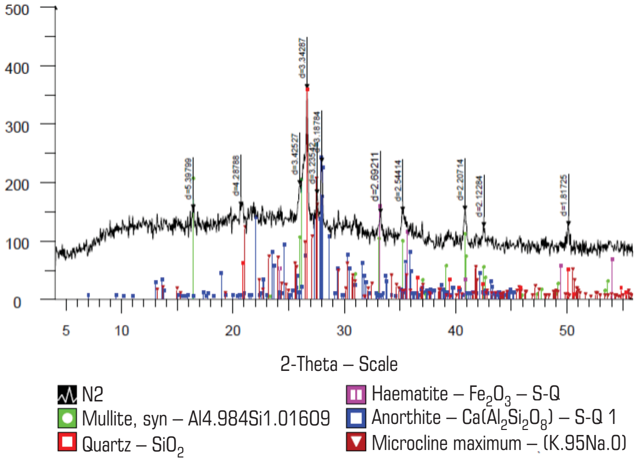


Fig. 2. The bar diagram of X-ray diffraction analysis of ash

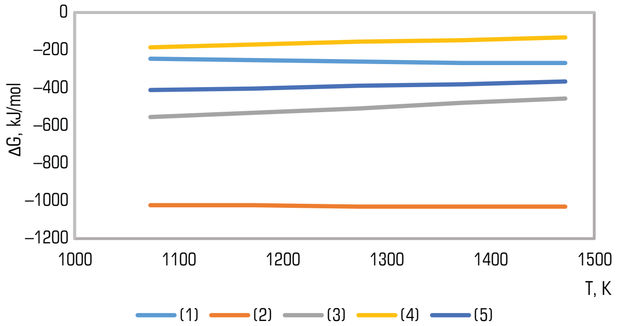


Fig. 3. The dependence of the change in the Gibbs energy of reactions (1)–(5) on temperature

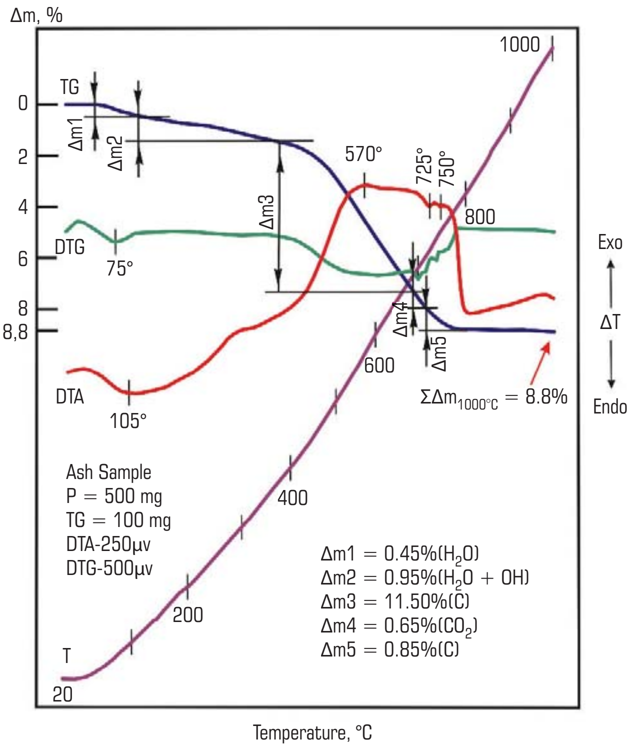


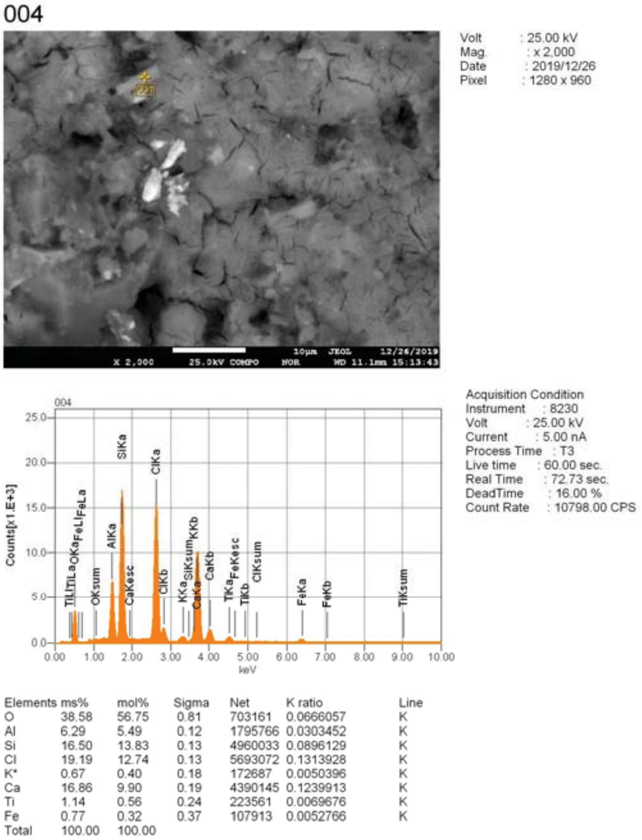
Fig. 4. Ash derivatogram

When organizing the technology of chlorinating ash roasting with $CaCl_2$ to obtain cinder and further leaching it in hydrochloric acid, it is fundamental to preliminarily separate iron by magnetic separation into a commercial product [14]. In addition, conditions for converting mullite, which is not easily soluble in hydrochloric acid, present in the ash, into easily soluble aluminum compounds – anorthite and geleniteshould be provided. The results of exploratory experiments on the magnetic separation of ash showed the possibility of obtaining a commercial iron-containing product with a high iron content (up to 50%). At the same time, a significant part of carbon up to 80% of its total content in the ash passes into the magnetic fraction. The removal of iron at the initial stage of the technology into a commercial product and the provision of conditions for the conversion of aluminum into easily soluble compounds during the further process of leaching the cinder ensures the production of high-quality silica in the form of a commercial product and the maximum conversion of aluminum into solution [11]. Implementation of the proposed technology does not require large capital and material costs and can be used for processing accumulated ash and slag wastes near existing thermal power plants.

Conclusions

1. The behavior of the carbon contained in the ash in the process of chlorination roasting of ash with gaseous chlorine and calcium chloride in an oxidizing atmosphere was studied. It is shown that under the conditions of roasting ash with chlorine in the presence of carbon, the probability of interaction between mullite, titanium and iron oxides is very high. In the process of roasting ash with $CaCl_2$ in an oxidizing atmosphere, the carbon present in the ash does not affect the general mechanism of the chlorination process.

2. It has been established that the Gibbs free energy of the reaction of decomposition of mullite with chlorine in the presence of carbon is characterized by a high negative value and at a roasting temperature of 1373 K is $\Delta G_{1373\ K} = –269.2$ kJ/mol. Large negative values of the Gibbs free energy have been established, indicating a high activity in the reduction reaction of titanium and iron oxides to their chlorides: for titanium oxide, $\Delta G_{1373\ K} = –1029.9$ kJ/mol; for FeO, $\Delta G_{1373\ K} = –145.64$ kJ/mol; for Fe_2O_3, $\Delta G_{1373\ K} = –377.48$ kJ/mol; for Fe_3O_4, $\Delta G_{1373\ K} = –480.96$ kJ/mol.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Mag. : x 2,000
Date : 2019/12/26
Pixel : 1280 x 960

Acquisition Condition
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Process Time : T3
Live time : 60.00 sec.
Real Time : 72.73 sec.
DeadTime : 16.00 %
Count Rate : 10798.00 CPS

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Elements ms%     mol%    Sigma   Net      K ratio         Line
O        38.58   56.75   0.81    703161   0.0666057       K
Al       6.29    5.49    0.12    1795766  0.0303452       K
Si       16.50   13.83   0.13    4960033  0.0896129       K
Cl       19.19   12.74   0.13    5693072  0.1313928       K
K*       0.67    0.40    0.18    172687   0.0050396       K
Ca       16.86   9.90    0.19    4390145  0.1239913       K
Ti       1.14    0.56    0.24    223561   0.0069676       K
Fe       0.77    0.32    0.37    107913   0.0052766       K
Total    100.00  100.00
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Fig. 5. Results of the EDS-analysis of the cinder obtained after roasting the ash with $CaCl_2$ at T = 1100 °C, τ = 60 min in an oxidizing atmosphere

3. The mechanism of carbon behavior during ash heating, which is typical for the conditions of ash roasting with $CaCl_2$ in an oxidizing atmosphere, was studied by the differential thermal method of analysis. It has been established that in the temperature range of 375–685 °C, 6.75% of carbon by weight of the sample is removed from the ash with the gas phase.

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TECHNOLOGY FOR PROCESSING QUARTZ–SERICITE ORE BY SELECTIVE GRINDING AND FLOTATION

Sericite, an aluminosilicate group mineral, has a considerable economic value as it is used in many branches of industry, including the cosmetic industry, paint-and-varnish industry and in production of polymers. Vietnam possesses explored deposits holding commercial reserves of quartz–sericite ore. Producibility of marketable sericite concentrate was tested using quartz–sericite ore samples from the Ha Tinh Province.

The data on the material constitution of the test ore from the X-ray structural analysis, optical studies and electron microscopy allowed offering a supposition on the feasible effective processing of the test ore by selective grinding and classification in hydraulic cyclones, which was experimentally proved. An essential loss of sericite in tailings required studying additional recovery of sericite by other methods. The studies into properties of cationic collector ArmacT by the Fourier Transform Infrared Spectroscopy (FTIR) to determine the nature of chemical bonds in molecules, as well as the adsorption tests of this collector at sericite and quartz in different operating conditions confirmed applicability of this agent to extract sericite from middlings (tailings of gravity concentration) and enabled flotation optimization.

Based on the implemented studies and experimental processing of the test ore using selective grinding, classification in cyclones and flotation, the flow chart was developed for processing initial ore with the total mass fraction of SiO_2 74.48%, Al_2O_3 16.20%, K_2O 3.52%, Na_2O 0.59% with production of sericite concentrate with the average particle size of 10 μm and mass fractions of SiO_2 49.33%; Al_2O_3 34.56%; (K_2O+Na_2O) 9.12%. This product meets the quality standards imposed on the raw stock for various industries.

Keywords: *quartz–sericite ore, material constitution, sericite, quartz, selective grinding, classification, flotation, cationic collectors, adsorption, concentrates*

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Introduction

Sericite is an aluminosilicate group mineral of fine dispersion, a partly hydrated mineral variety of white mica (muscovite). The chemical formula is $KAl_2[AlSi_3O_{10}](OH)_2$ or $KAl_2[AlSi_3O_{10}](OH,F)_2$,). The mineral has a low content of K_2O and an increased content of SiO_2, MgO and H_2O. It represents an accumulation of cryptoflakes with a silky gloss [1–4].

The economic value of sericite develops from the use of the mineral in the paint-and-varnish industry, in manufacture of polymers and, specifically, in the cosmetic industry. The key suppliers of sericite products to the global market is China and Finland [4, 5]. The overall output of sericite products in 2018 totaled 340 000 t at the prices of USD 500/kg.

In view of the increase in the mineral resources and thanks to the provision of the industry with the own reserves, Vietnam started prospecting sericite in the 1990s. Much of the explored deposits however were small bodies of