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Beneficiation and Processing of Ilmenite Ores from Vietnam

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Abstract

Beneficiation of ilmenite ores from the Ha Tinh deposit (Vietnam) is studied by means of electromagnetic, electrostatic separation, and flotation. The dependences of the mass of the obtained magnetic fraction on the current strength and on beneficiation degree are determined for different numbers of the stages of magnetic separation. The conditions for the change in the concentration of the titanium product depending on the voltage on the electrodes in the process of electrostatic separation are studied. It is shown that separation brings the heavy magnetic fraction apart from the waste rock, while subsequent flotation provides a high efficiency of rutile separation from impurity components. For flotation concentration of the product after separation processes, it is established that process efficiency depends on titanium concentration in the product to be concentrated. As a result of beneficiation, ilmenite concentrate containing more than 50 % TiO_2 is obtained, which meets the most stringent requirements of the market. The design of the laboratory facility and the fluorination reactor is described, as well as the materials used to manufacture them. The research methodology is described in detail. During the fluorination of the obtained concentrate with elemental fluorine, the dependences of conversion degree on the time and temperature of fluorination are investigated (S-shaped curves). It is determined that the process is complete (conversion degree is more than 98 %) within 5 min at 700 °C. The features of the fluorination of the obtained concentrate are analyzed on the basis of kinetic equations. The activation energy and preexponential factor in the Yander kinetic equation are determined. It is shown that fluorination kinetics are limited by diffusion processes (supply and removal of reagents) rather than by the rate of the chemical reaction itself.

Keywords: ilmenite ore and concentrate, magnetic and electrostatic separation, flotation, fluorination, conversion degree, kinetic equation, titanium tetrafluoride, correlation coefficient, activation energy

INTRODUCTION

Vietnam is one of the major manufacturers of ilmenite ores in the world [1]. The amount of titanium ores mined in Vietnam accounts for ~8 % of the total amount of titanium ores produced in the world. The Ha Tinh deposit, situated at the coastal territory, is one of the best developed and most promising titanium deposits in Vietnam. For subsequent chemical processing, it is necessary to concentrate the ores of this deposit. At present, titanium ores are concentrated by means of electromagnetic separation [2–6], electrostatic separation [7–11] and flotation [12–16].

The main difficulty in concentrating titanium ores is the joint presence of ilmenite and rutile in these ores. These minerals are readily separated from the dead rocks, but it is necessary to apply special concentrating methods and conditions to separate the heavy fraction of titanium-containing minerals.

The technology of chemical treatment of the obtained ilmenite concentrates (IC), which are difficult to process, is not less important. These concentrates are usually processed using the sulphuric (sulphate) and chloride methods [17–19], as well as the methods proposed recently [20–22]. However, these methods are connected with the formation of a large amount of toxic wastes during treatment, which causes the necessity of their subsequent neutralization. For this reason, the amounts of titanium concentrates to be processed in the countries are strictly limited by the governments of these countries because of the catastrophic impact on ecology [23].

This work continues studies aimed at the development of the original methods of ilmenite ore concentrating, which would allow obtaining IC meeting the most rigid requirements of the world market [24–26]. It is proposed to carry out subsequent chemical treatment of the obtained IC using the fluoride method through fluorination of the concentrates using molecular fluorine [27]. The proposed fluoride technology allows avoiding environmental pollution with the fluorinating reagent and also with the products – fluorides of impurities, due to their multiple recycling during the process and the use during subsequent deep processing [28]. One of the key stages of the proposed fluoride technology, namely fluorination with molecular fluorine, is considered in detail in the present work.

EXPERIMENTAL

Materials

Ilmenite ore from the Ha Tinh deposit (Vietnam).

Sodium oleate (foam-forming agent, collector, NaOl) – sodium salt of oleic acid, $NaC_{18}H_{33}O_2$.

Sulphuric acid, sodium hydroxide (regulators of the acidity of medium).

Molecular fluorine.

Argon.

Object of investigation

The concentrations of major component oxides in ilmenite ore under investigation are, wt. %: titanium (TiO_2) – 27.66; iron (Fe_2O_3) – 14.62; zirconium (ZrO_2) – 9.23; silicon (SiO_2) – 4.52; cerium (CeO_2) – 0.18; hafnium (HfO_2) – 0.17; niobium (Nb_2O_5) – 0.11; vanadium (V_2O_5) – 0.044.

Equipment

The ore was separated with the electromagnetic rolling mill EVS-10/5 (Russia), which possesses the maximal magnetic induction in the working zone not less than 1.7 T, with the diameter and length of the working part of the roll 100 and 50 mm, respectively. The productivity of the electrostatic separator of cylinder type ELKOR-1 (Russia), intended for ore concentrating, is 50 kg/h; the material under treatment is to be composed of the particles 0.04–5.0 mm in size; the diameter and length of the precipitating electrode (cylinder) are 240 and 250 mm, respectively; the maximal voltage at the high-voltage electrodes is 40 kV. The volume of the chamber of flotation device FMF-3(l) (Russia) is 3 L, impeller diameter is 55 mm.

Analysis of the obtained samples was carried out with the ARL EQUINOX 100 XRD & ARL QUANT'X XRF spectrometer (Thermo Fisher Scientific, Switzerland), intended for measuring the concentrations of substances by means of X-ray diffraction (EDXRF).

The schematics of the set-up and IC fluorination reactor are shown in Fig. 1 and 2, respectively. The main units of the set-up (see Fig. 1) are the sources of argon *1* and fluorine *6*, devices for measuring their flow rates *4*, mixing chamber *5* and reaction *и* chamber *9*, connected with each other through pipelines. The components of the gas mixture are mixed with a stationary mixer *5*, which is a vessel of elongated shape, the initial components entering it at one end and the formed mixture leaving it at the other end. To improve mixing, a mechanical mixer *8* is used, which is composed of a cylindrical case with a porous nickel filler inside. Fluorination temperature (up to 700 °C) is measured with chromel-alumel thermocouples mounted in the pockets made of monel metal. Separation of the captured IC powder from the outgoing gas flow is performed in a special chamber *11*.

In the fluorination reactor (see Fig. 2), preliminarily prepared mixture of fluorine with argon is fed through jet orifices *1* in the lower part of the apparatus. A sample of the initial IC is supplied to the reaction chamber with the help of tool *2*. Tubes *3* serve to regulate the input of a mixture of F_2 with Ar. Inductors *5* are arranged on ceramic insert *4* and substrate *6*. The changes in substrate mass are recorded with the recording unit 7.

Solid and gaseous fluorides formed during fluorination are corrosion-active substances. In the

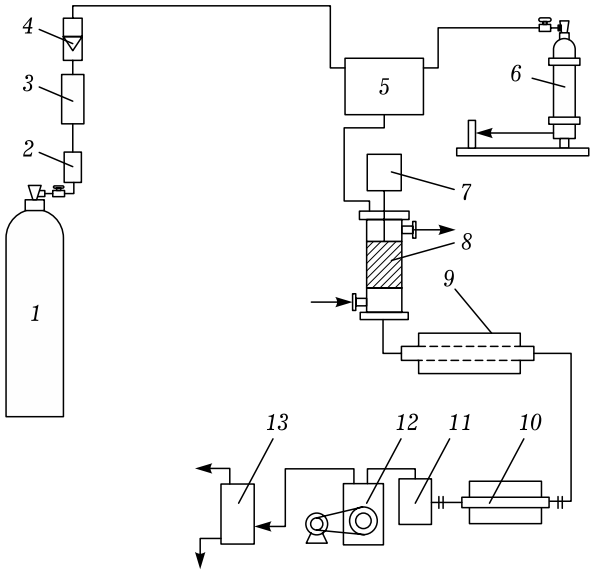

Fig. 1. Schematic of laboratory installation for fluorination of ilmenite concentrate: *1* – cylinder with Ar; *2*, *3* – filters; *4* – flow meter; *5*, *8* – stationary and mechanical mixers of F_2 with Ar; *6* – cylinder F_2; *7* – holder of metalloceramic filling; *9* – fluorination reactor; *10* – chamber for gas mixture cooling; *11* – vessel for separating the captured solid phase; *12* – vacuum pump; *13* – scrubber for removing excess fluorine.

absence of water vapour, nickel, copper, chromium, alloys monel, inconel, nichrome are corrosion-resistant in the environment of gaseous fluorides and fluorine up to 300 °C, because a dense film of non-volatile fluorides is formed, and this film protects the material from corrosion. Stainless steel 12Kh18N9T is stable up to 150 °C. At higher temperatures, it is necessary to use nickel or its alloys [29].

Investigation procedures

To study the samples of ilmenite ore and the products of its concentrating by means of EDX-RF, their reflection was measured for 15 min under the action of CuK_α radiation with a PVC-filter to decrease the fluorescence of Fe, as well as CoK_α radiation. This procedure was used to measure the concentrations of four components: titanium, iron, aluminium, and silicon.

Before analysis, calibration of the spectrometer was carried out. For this purpose, standard samples of titanium, iron, aluminium and silicon were prepared from the corresponding oxides. The mass of each sample was 5 g. These samples were thoroughly mixed, and then 2 g of each sample were placed in special cells with the bottoms made of reflecting film.

RESULTS AND DISCUSSION

Studies of electromagnetic separation of the ores

Four stages of concentrating the ore under investigation were carried out by means of electromagnetic separation. It is shown that with an in-

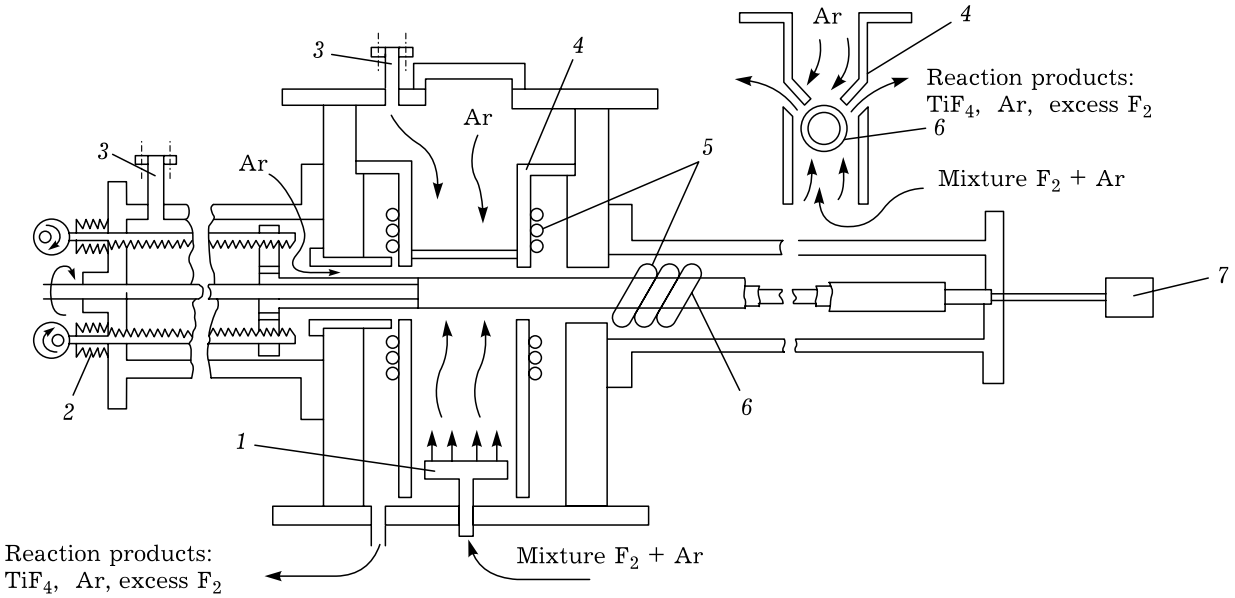

Fig. 2. Schematic of the reactor for ilmenite concentrate fluorination: *1* – jet orifice; *2* – displacement mechanism; *3* – tubes for supplying fluorine and nitrogen; *4* – ceramic insert; *5* – inductors (for substrate heating); *6* – substrate; *7* – recorder of substrate mass changes.

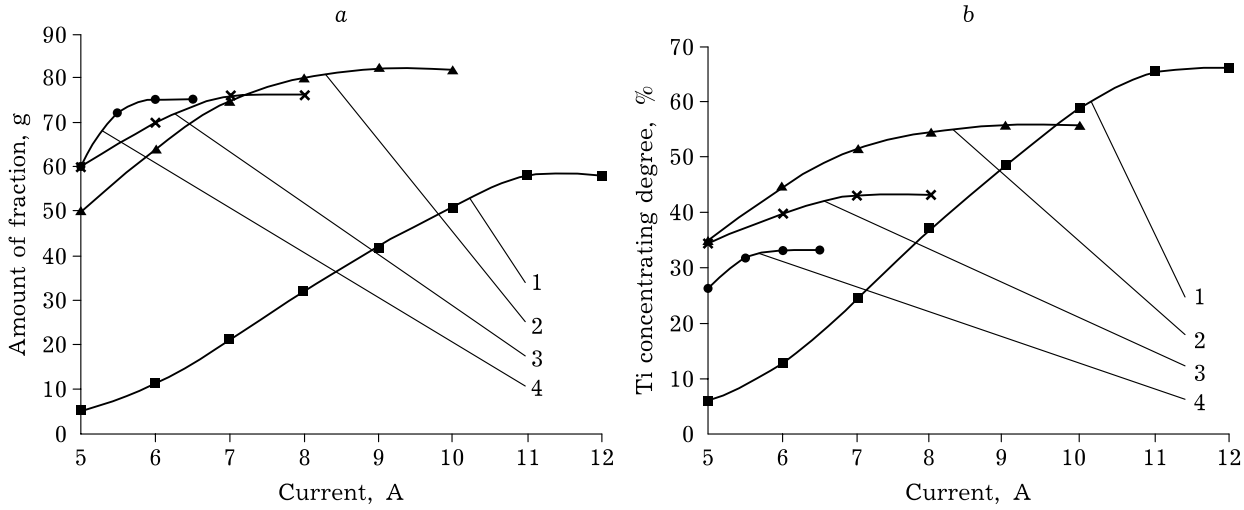

Fig. 3. Changes in the amount of the obtained magnetic fraction versus the strength of current (*a*) and concentrating degree (*b*) per the stages of magnetic separation: after the first (*1*), second (*2*), third (*3*) and fourth stage (*4*).

crease in the number of stages, ilmenite concentration in the magnetic fraction increases with an increase in the strength of current (Fig. 3, *a*), but the amount of the magnetic fraction decreases (see Fig. 3, *b*). The best results are achieved when the process is carried out in three stages.

Studies of electrostatic separation process

The features of the Vietnam ilmenite ore concentrating by means of electrostatic separation have been studied (Fig. 4). It is established that the distribution of target components (titanium minerals) changes in ten cells of the separating unit with an increase in the voltage between the electrodes of the separator from 25 to 35 kV.

The results show that titanium and iron are mainly distributed in cells 1 to 5 depending on the voltage between the electrodes of the separator. The distribution of components did not change when the voltage achieved 30 kV and higher. So, for separation to proceed efficiently, the voltage supplied to the device should not be less than 30 kV.

Features of flotation concentrating

The effect of TiO_2 concentration (C_{TiO_2}) in ilmenite ore on the efficiency of flotation was investigated. It is shown in Fig. 5 that the efficiency depends on titanium concentration in the initial ore. The higher is the latter, the higher is the

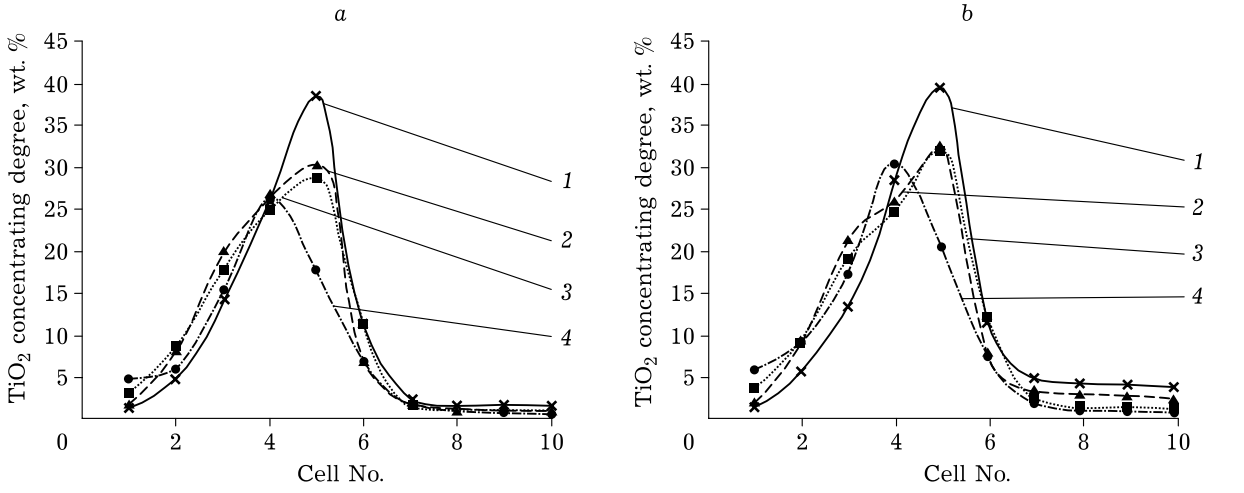

Fig. 4. Dependence of the concentrations of TiO_2 (*a*) and Fe_2O_3 (*b*) in the product in separator cells on the voltage between electrodes, kV: 25 (*1*), 27.5 (*2*), 30 (*3*), 35 (*4*).

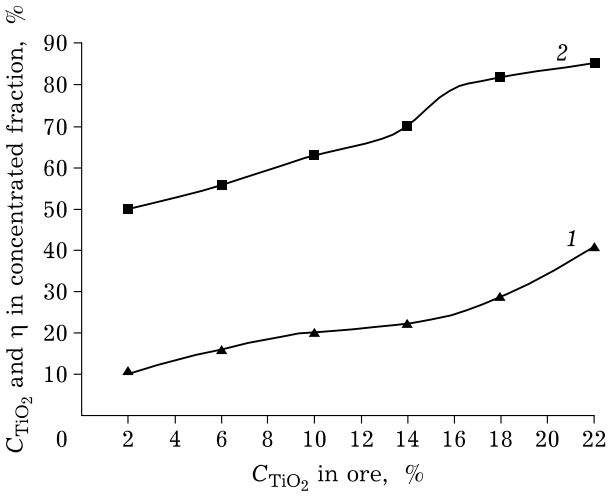

Fig. 5. Dependence of flotation efficiency on TiO_2 concentration (C_{TiO_2}, concentration range 2–22 %) in ilmenite ore: *1* – C_{TiO_2} in the concentrated fraction; *2* – concentrating degree (η). Flotation conditions: C_{NaOl} = 0.36 g/L, pH 6.5–7.5, reaction time 10 min.

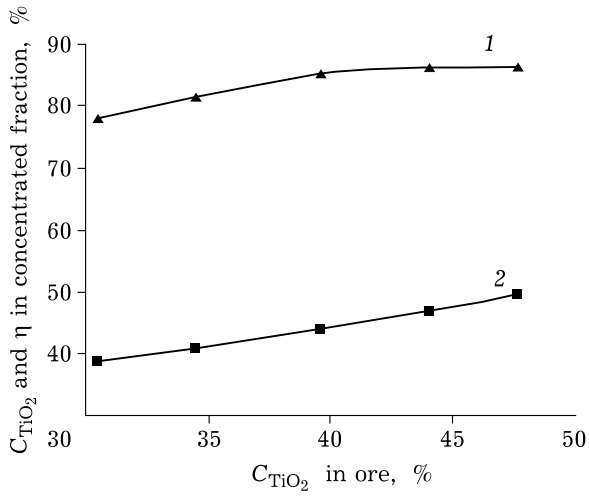

Fig. 6. Dependence of flotation efficiency on TiO_2 concentration (C_{TiO_2}, concentration range 30–50 %) in ilmenite ore: *1* – C_{TiO_2} in the concentrated fraction; *2* – concentrating degree (η). Flotation conditions: C_{NaOl} = 0.33 g/L, pH 3.4–7, reaction time 10 min.

concentrating degree (η) and the higher is the titanium oxide concentration in the final product. This may be explained as follows: the larger the amount of impurities is present in the initial ore, the stronger they will affect flotation due to their transition in the foamy phase. Flotation efficiency increases for C_{TiO_2} in the ore more than 20 wt. % and η > 80 %. In this situation, C_{TiO_2} in the concentrated product can reach ~39 %.

It is demonstrated that for C_{TiO_2} range in the ore 30–50 wt. % and higher, η changes insignificantly: from 79 to 86 % (Fig. 6). For C_{TiO_2} in the ore less than 22 wt. % (see Fig. 4), C_{TiO_2} in the concentrated product increases much faster than in the region of high concentrations. For example, with an increase in C_{TiO_2} in the initial ore from 30 to 40 % (see Fig. 5), C_{TiO_2} in the foamy fraction increases only by 5 % (from 38.91 to 43.60 %). For C_{TiO_2} in the ore more than 40 %, C_{TiO_2} in the resulting product was increasing even slower, though η was high and reached ~86 %. The reason is in the high concentration of Fe_2O_3 in the ore, which explains the insufficiently good flotation characteristics of this ore.

As far as the experimental conditions are concerned (pH, NaOl concentration (g/L), process time), flotation at the high TiO_2 content in the initial material (>30 %) leads to obtaining purified titanium products or to the separation of titanium concentrates (ilmenite and rutile). In the acid medium (at pH 3.5–4), ilmenite is separated from rutile, while at pH 6–8 light components are separated from heavy ones.

The equation for calculating the concentrating degree η depends on TiO_2 concentration in the initial ore:

$$\eta = \frac{C^{c}_{TiO_2} \cdot m_c}{C^{o}_{TiO_2} \cdot m_o}$$

where $C^{c}_{TiO_2}$ and $C^{o}_{TiO_2}$ are TiO_2 concentrations in the obtained concentrate and in the ore to be

TABLE 1
TiO_2 concentration in the product after concentrating by means of electrostatic, electromagnetic separation and flotation

Concentrating stage	TiO_2 concentration, wt. %
Initial ilmenite ore	27.66
Electrostatic separation (at the voltage of 30 kV)	30.24
Electromagnetic separation (at the strength of current 11 A):	
two-stage process	47.58
three-stage process	48.68
four-stage process	48.71
Flotation (sodium oleate concentration 0.35 g/L)	51.74

concentrated, respectively; m_c, m_o are the masses of the concentrate and the ore, respectively.

For example, if $C^c_{TiO_2}$ = 30.45 % and $C^o_{TiO_2}$ = 38.91 %, while the masses of the concentrate and the ore are m_c = 12.3 g (from experiment) and m_o = 20 g, then

$$\eta = \frac{30.45 \cdot 12.3}{38.91 \cdot 20} = 78.12\ \%$$

Thus, the concentration of the target component (TiO_2) gradually increases during Vietnam ilmenite ore concentrating by means of electromagnetic, electrostatic separation and flotation as shown in Table 1. In the concentrated IC, the concentration of TiO_2 becomes higher than 50 wt. %. These concentrates are quite suitable for subsequent processing by means of fluorination with molecular fluorine.

Fluorination of ilmenite concentrate with molecular fluorine

As a result of concentrating, the obtained IC contains the following amounts of oxides, %: TiO_2 51.74; FeO 23.31; Fe_2O_3 16.73; MnO 3.35; SiO_2 2.48; CaO 0.06; MgO 0.23; Al_2O_3 1.02; V_2O_5 0.14; P_2O_5 0.12; Cr_2O_3 0.05; U 0.0002; Th 0.005.

The kinetic features of IC fluorination with molecular fluorine were studied using powder samples with the granulometric composition varied from $9 \cdot 10^{-7}$ to $8 \cdot 10^{-4}$ m, with specific surface within the range 3–5 m^2/g. Sample mass was recorded continuously during the process at controlled temperature. Fluorine was diluted with the inert gas (argon) in order to remove the heat released during fluorination and to create isothermal conditions.

It is established that fluorination proceeds only slowly at 550 °C and below, maybe with the formation of intermediate titanium oxyfluorides covering the surface of initial particles. More detailed analysis of non-fluorinated residue revealed grey-brown powder, and its composition changes from the periphery to the centre of a particle: the deep part had a dark-coloured shell, in which fluorine was not detected by means of analysis. The process rate becomes noticeable only at 550 °C, and at 700 °C the process takes place with higher intensity, and titanium tetrafluoride is removed within 8–10 min.

Within the temperature range 400–700 °C, the dependence of transformation degree (α) on fluorination time (τ) was studied (Fig. 7). The obtained dependences exhibit S-shaped structure, which is characteristic of interactions in the system solid – gas [30].

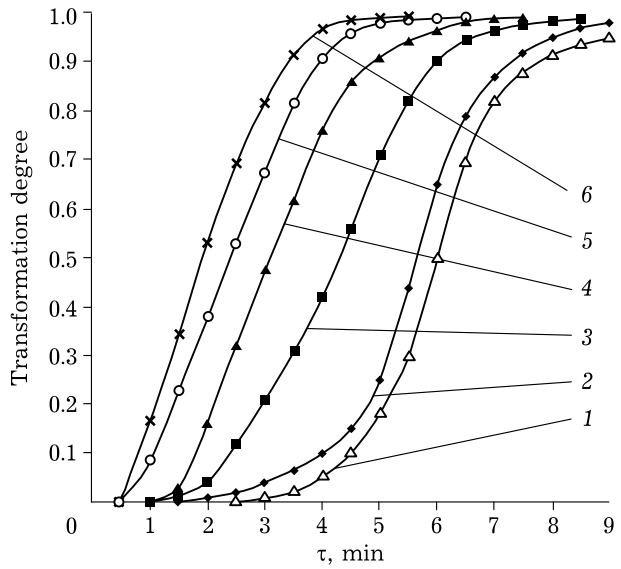

Fig. 7. Dependence of transformation degree in the system ilmenite concentrate – fluorine on fluorination time (τ) and temperature: 400 (*1*), 500 (*2*), 550 (*3*), 600 (*4*), 650 (*5*), 700 °C (*6*).

During IC fluorination, the amount of the solid product increases due to the formation of FeF_3, so the transformation degree α was calculated according to equation

$$\alpha = \frac{m_i - m_0}{m_{max} - m_0}$$

where m_0 is the initial sample mass, g; m_i is sample mass during fluorination at moment of time t_i, g; m_{max} is sample mass at the maximal fluorination time t_{max}, g.

The kinetic dependences are characterized by three regions. The first region relates to the initial period, at which the process rate is very low, and fluorination occurs due to the formation of intermediate oxyfluorides of the elements that are present in the IC [31]. During this period, the interface is formed, which is composed of partially fluorinated components of the initial concentrate. Due to fluorine diffusion, the formation of the intermediate components of the initial concentrate starts inside the particles. The second region is characterized by a gradual increase in fluorination rate to the maximal value. As a result, the kind of kinetic curves depends on changes in the position of interface; that is, on the thickness of the layer of solid particles in which their interaction with the gas phase proceeds. At the third region, process rate decreases due to evaporation of the gaseous fluorides and the formation of nonvolatile fluorides in the form of new particles [32]. Fluorination proceeding at 400 °C is characterized by

TABLE 2

The values of E_a and K_0 of ilmenite concentrate fluorination with molecular fluorine, calculated using different methods

Model	Equation	E_a, kJ	K_0	Approximation confidence (R^2)
Gistling	$1 - (1 - \alpha)^{2/3} = k\tau$	45.74	0.43	0.8988
Jander	$\mathbf{(1 - (1 - \alpha)^{1/3})^2 = k\tau}$	**37.60**	**0.81**	**0.9383**
Shrinking cylinder	$1 - (1 - \alpha)^{1/2} = k\tau$	35.80	0.01	0.9211
Prout–Tompkins	$\ln(\alpha/(1 - \alpha)) = k\tau + C$	94.79	17.29	0.9129
Kazeev–Erofeev	$\ln(-\ln(1 - \alpha)) = n\ln\tau + \ln k$	108.91	$1.62 \cdot 10^6$	0.9306
Shrinking sphere	$1 - (1 - \alpha)^{1/3} = k\tau$	34.65	0.90	0.9345

Note. Here and in Table 3: E_a is apparent activation energy; K_0 is preexponential factor; α is the degree of ilmenite concentrate transformation; k is reaction rate constant; τ is fluorination time; T is process temperature.

longer reaction time. With temperatures rising to 550–700 °C, the rate increases substantially due to the transition to the combustion phase, proceeding with the release of a large amount of heat.

The obtained results were described using kinetic equations, where k is the rate constant of fluorination: Gistling $1 - (1 - \alpha)^{2/3} = k\tau$; Jander $(1 - (1 - \alpha)^{1/3})^2 = k\tau$; shrinking cylinder $1 - (1 - \alpha)^{1/2} = k\tau$; Prout–Tompkins $\ln(\alpha/(1 - \alpha)) = k\tau + C$; Kazeev–Erofeev $\ln(-\ln(1 - \alpha)) = n\ln\tau + \ln k$; shrinking sphere $1 - (1 - \alpha)^{1/3} = k\tau$. As a result, activation energies (E_a) and preexponential factors (K_0) were calculated; the results are presented in Table 2.

The confidence of approximation (R^2 = 0.9383), closest to unity, was obtained by process description using Jander equation. The linear dependence in the coordinates $\ln k - (1000/T)$, where T is process temperature, is characteristic of the whole fluorination process except the initial and final stages. Deviation from the straight-line dependence at the very start of the process is due to filling the system with argon, which is necessary to remove air from the system; at the final stage of the process, the deviation is due to the formation of solid non-volatile fluorides, in particular iron trifloride. The proposed model of IC particle fluorination fully corresponds to the obtained experimental kinetic results.

Deviation from linearity in the system time – $(1 - (1 - \alpha)^{1/3})^2$, occurring after fluorination is over, is explained by the fact that IC particles reacting with fluorine at different rates form both volatile and non-volatile fluorides. In addition, the transformation of iron oxyfluoride into trifluoride determines the process kinetics because it is the limiting stage of the process. The rate constants k of IC fluorination at different temperatures T were calculated from the data presented in Fig. 8 and are shown in Table 3.

The apparent activation energy E_a and preexponential factor K_0 of IC fluorination with molecular fluorine were determined from the slope of the straight line in Arrhenius coordinates using Jander equation (Fig. 9).

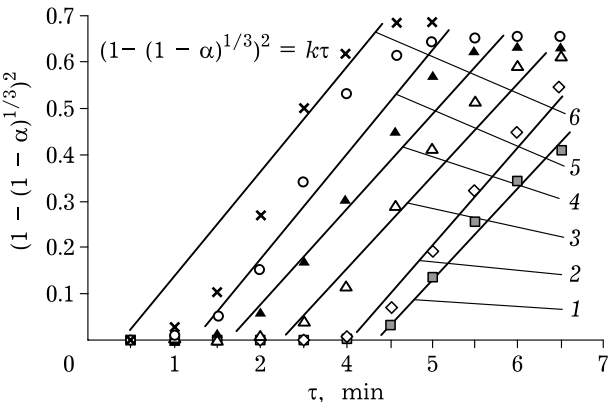

Fig. 8. Dependence of $(1-(1-\alpha)^{1/3})^2$ on time (τ) at fluorination temperature: 400 (*1*), 500 (*2*), 550 (*3*), 600 (*4*), 650 (*5*), 700 °C (*6*).

TABLE 3

Calculated kinetic values (according to Jander equation) for ilmenite concentrate fluorination with molecular fluorine

Temperature		$1000/T$, 1/K	k, min^{-1}	ln k
°C	K			
400	673	1.486	0.042	−3.169
500	773	1.294	0.056	−2.884
550	823	1.215	0.072	−2.625
600	873	1.145	0.074	−2.607
650	923	1.083	0.076	−2.576
700	973	1.028	0.081	−2.515

Note. For designations, see Table 2.

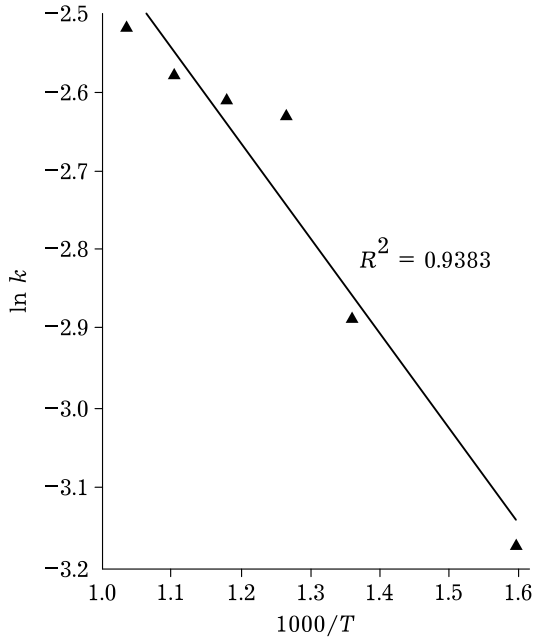

Fig. 9. Dependence of ln k (k − rate constant of fluorination) on reciprocal temperature (T) as calculated according to Jander equation $(1-(1-\alpha)^{1/3})^2 = k\tau$.

The kinetic equation of IC fluorination is

$$(1 - (1 - \alpha)^{1/3})^2 = 0.81e^{-37.6/RT}$$

The value of apparent activation energy E_a = 37.6 kJ provides evidence that the limiting stage of fluorination is diffusion (input of gaseous fluorine to the surface of IC particles and output of the formed gaseous fluorides from the volume and from the surface of the particles obtained in the process), rather than fluorination kinetics (that is, the rate of interaction of the solid material with fluorinating agent).

CONCLUSION

Thus, the efficiency of electromagnetic and electrostatic separation, as well as the methods of flotation concentrating, are demonstrated for ilmenite ore concentrating. Electromagnetic separation allows providing the complete separation of magnetic fraction from non-magnetic one through three sequential concentrating processes (in three stages). The major advantage of electrostatic separation is the provision of high degree of ilmenite concentrating (TiO_2 concentration is ~40 %) by means of a single-shot process. This is the feature of electrostatic concentrating of ilmenite ores under investigation.

Subsequent flotation concentrating using sodium oleate as a collector (foam-forming agent) allows providing both further increase in ilmenite content in the target product and separation of rutile from the dead rock composed of non-magnetic fraction. As a result of concentrating according to the proposed procedure, IC with TiO_2 content exceeding 50 % is obtained. These concentrates may compete in their quality even with concentrates manufactured in Australia, with an ilmenite content somewhat lower than 50 % [33].

The interaction of the obtained IC with fluorine involved spontaneous heating of the system and transformation of oxides in these concentrates into volatile and nonvolatile fluorides.

As a result of the kinetic studies of IC fluorination with molecular fluorine, the dependences of transformation degree on time were studied within the temperature range 400−700 °C. It is demonstrated that the best results are obtained at 700 °C. Further temperature rise is unreasonable because in this case the interaction of molecular fluorine with the material of fluorination reactor walls (monel metal) becomes significant. Mathematical processing of the quantitative data on the interaction of the components of concentrate was carried out using the kinetic equations: Gistling, Jander, shrinking cylinder, Prout−Tompkins, Kazeev−Erofeev and shrinking sphere. It is determined that the process is described most accurately with Jander equation.

The values of apparent activation energy (37.6 J/mol) and preexponential factor (k = 0.81 min^{-1}) of IC fluorination with molecular fluorine were determined.

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