ISSN 1561-8331 (Print)
ISSN 2524-2342 (Online)

ТЭХНІЧНАЯ ХІМІЯ І ХІМІЧНАЯ ТЭХНАЛОГІЯ

TECHNICAL CHEMISTRY AND CHEMICAL ENGINEERING

УДК 661.833+66.096.5
https://doi.org/10.29235/1561-8331-2026-62-4-344-352

Поступила в редакцию 09.02.2026
Received 09.02.2026

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THERMAL DECOMPOSITION OF SODIUM HEXAFLUOROSILICATE IN A FLUIDIZED BED

Abstract. We studied the thermal decomposition of sodium hexafluorosilicate Na_2SiF_6, carried out under isothermal conditions at temperatures of 773, 873, 973, and 1 073 K in a fluidized bed. A mathematical model of sodium hexafluorosilicate fluidization is proposed, taking into account the kinetics of chemical transformations and changes in the physicochemical properties of the fluidized bed material and the gaseous medium. The results of calculations show that the height and porosity of the fluidized bed change during the conversion of sodium hexafluorosilicate. The specific consumption of silicon tetrafluoride during a single loading of sodium hexafluorosilicate varies with time and depends on the bed temperature. Proposed analytical expression for the time-dependent mass flow rate of Na_2SiF_6 allows obtaining a constant mass yield of silicon tetrafluoride SiF_4. The use of fluidized bed technology enables the thermal conversion of sodium hexafluorosilicate in a fluidized bed in an air environment with continuous removal of the reacted dispersed material and supply of the initial dispersed material. This reduces the equipment cycle time without regular reactor shutdowns and startups, thereby increasing the reliability of the Na_2SiF_6 thermal decomposition equipment and the SiF_4 production productivity.

Keywords: sodium hexafluorosilicate, silicon tetrafluoride, fluidized bed, decomposition kinetics, rate constant, reactor scheme

For citation. Vasilevich S. V., Stoiko S. O., Yukhnevich S. D. Thermal decomposition of sodium hexafluorosilicate in a fluidized bed. *Vestsi Natsyyanal'nai akademii navuk Belarusi. Seryya khimichnykh navuk = Proceedings of the National Academy of Sciences of Belarus. Chemical Series*, 2026, vol. 62, no. 4, pp. 344–352 (in Russian). https://doi.org/10.29235/1561-8331-2026-62-4-344-352

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ТЕРМИЧЕСКОЕ РАЗЛОЖЕНИЕ ГЕКСАФТОРСИЛИКАТА НАТРИЯ В ПСЕВДООЖИЖЕННОМ СЛОЕ

Аннотация. Исследовано термическое разложение гексафторсиликата натрия Na_2SiF_6, проводимое в изотермических условиях при температурах 773, 873, 973 и 1 073 К в псевдоожиженном слое. Предложена математическая модель псевдоожижения гексафторсиликата натрия, учитывающая кинетику химических превращений и изменение физико-химических свойств материала псевдоожиженного слоя и газовой среды. Результаты расчетов показывают, что высота и порозность псевдоожиженного слоя изменяются в процессе конверсии гексафторсиликата натрия. Удельный выход тетрафторида кремния при однократной загрузке гексафторсиликата натрия изменяется со временем и зависит от температуры слоя. Предложенное аналитическое выражение для зависящего от времени массового расхода Na_2SiF_6 позволяет получать постоянный массовый выход тетрафторида кремния SiF_4. Использование технологии псевдоожиженного слоя позволяет осуществлять термическую конверсию гексафторсиликата натрия в псевдоожиженном слое в воздушной среде с непрерывным удалением прореагировавшего диспергированного материала и подачей исходного диспергированного материала. Это сокращает время цикла работы оборудования без регулярных остановок и запусков реактора, тем самым повышая надежность оборудования термического разложения Na_2SiF_6 и производительность производства SiF_4.

Ключевые слова: гексафторсиликат натрия, тетрафторид кремния, псевдоожиженный слой, кинетика разложения, константа скорости, схема реактора

Для цитирования. Василевич, С. В. Термическое разложение гексафторсиликата натрия в псевдоожиженном слое / С. В. Василевич, С. О. Стойко, С. Д. Юхневич // Весці Нацыянальнай акадэміі навук Беларусі. Серыя хімічных навук. – 2026. – Т. 62, № 4. – С. 344–352. https://doi.org/10.29235/1561-8331-2026-62-4-344-352

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Introduction. Sodium hexafluorosilicate Na_2SiF_6 is a valuable product, the production of which has been the subject of a number of studies [1–3]. Na_2SiF_6 is currently widely used to produce silicon, which is utilized in various industries. The demand for pure silicon on the global market is growing due to its growing use in industries such as solar energy and electronics.

Sodium hexafluorosilicate can be used as a silicon source to form silicon nitride Si_3N_4 coatings using a hybrid precursor system – chemical vapor deposition [1, 4].

In the production of high-purity ^{28}Si, the starting product is silicon tetrafluoride SiF4, which is obtained by the thermal decomposition of sodium hexafluorosilicate: $Na_2SiF_6 \rightarrow 2NaF + SiF_4$ [5, 6]. Thermal decomposition of sodium hexafluorosilicate begins at a temperature of 300 °C. The process of obtaining SiF_4 is carried out at temperatures of 540–710 °C.

A description of a method and a device for processing (thermal decomposition) Na_2SiF_6 is given in [7]. This device is intended for the recycling of secondary products of apatite processing during the production of phosphate fertilizers, in particular, sodium hexafluorosilicate Na_2SiF_6, to produce silicon tetrafluoride SiF_4 and sodium fluoride NaF.

In this device, the reaction occurs in a dense layer, which causes passivation of the reaction as the upper layers of sodium hexafluorosilicate react, thereby preventing a continuous process of its thermal decomposition.

This paper presents the results of studies on the features of fluidization of Na_2SiF_6 during its thermal decomposition under isothermal conditions in an air atmosphere in a fluidized bed.

The aim of the study is to determine the influence of various factors on the process of fluidization of sodium hexafluorosilicate during its thermal conversion.

Fluidized bed model. Sodium hexafluorosilicate Na_2SiF_6, a powder with a particle size $d_{Na_2SiF_6}$ of 0.317 mm, was used as the study object. The density of Na_2SiF_6 is 2.68 g/cm³ [8, 9]. Air was used as the fluidizing agent.

The thermal decomposition of Na_2SiF_6 in air can be written as a formal reaction [1–5]:

$$Na_2SiF_6 \rightarrow SiF_4 + 2NaF \quad (1)$$

Since the thermal decomposition of Na_2SiF_6 is not instantaneous and is accompanied by a shift of the reaction front from the periphery to the center of the particle, the composition and density of sodium hexafluorosilicate change gradually during the conversion process. The particle density of the layer can be determined as:

$$\rho_p = \rho_{Na_2SiF_6}(1-\alpha) + \rho_{2NaF}\alpha,$$

where α is the degree of decomposition of Na_2SiF_6; $\rho_{Na_2SiF_6}$ is the density Na_2SiF_6, kg/m³; ρ_{NaF} is the density of NaF, kg/m³.

The density of sodium fluoride is 2.79 g/cm³ [8, 10]. The volume of NaF particles is 2.33 times smaller than that of the original Na_2SiF_6. The decrease in size occurs due to the compaction of the crystal lattice. If the dimensions of the unit cell of Na_2SiF_6 are 0.886 nm and 0.504 nm (the crystal has the form of a trigonal syngony with two sodium ions in the lattice) [9], then for NaF they are 0.463 nm (the crystal has the form of a cube with four sodium ions in the lattice) [10]. Then the particle size (equivalent particle diameter) will be determined as:

$$d_p = d_{Na_2SiF_6}\sqrt[3]{1-\alpha\left(1-\frac{2\rho_{Na_2SiF_6}M_{NaF}}{\rho_{NaF}M_{Na_2SiF_6}}\right)} = d_{Na_2SiF_6}\sqrt[3]{1-0.5708\alpha}. \quad (2)$$

Here d_p is the equivalent particle diameter, m; $M_{Na_2SiF_6}$ – molecular weight of sodium hexafluorosilicate, kg/mol; M_{NaF} – molecular weight of sodium fluoride, kg/mol:

$$M_{Na_2SiF_6} = 188 \cdot 10^{-3} \text{ kg/mol}; \; M_{NaF} = 42 \cdot 10^{-3} \text{ kg/mol}.$$

Previously [11], the authors conducted studies on the thermal conversion of this material in a dense layer. It was determined that the kinetics of the thermal decomposition of sodium hexafluorosilicate under the conditions of the studies performed is described by the Avrami–Erofeev equation:

$$\left(-\ln\left(1-\alpha\right)\right)^{n} = kt\,, \tag{3}$$

where n – is a certain indicator; k – is the rate constant of the formal reaction of decomposition of the substance, min^{-1}; t – is the time of holding the sample at the decomposition temperature, min.

Analysis of the obtained data revealed that the range of n-values ranged from 0.541 to 1.291. The average n-value calculated across all study variants was 0.761.

The rate constant k is determined from the Arrhenius equation:

$$k = A\exp\left(-\frac{E}{RT}\right), \tag{4}$$

where A is the pre-exponential factor, s^{-1}; E – activation energy, J/mol; R – universal gas constant (8.314 J/mol·K); T – temperature, K.

The decomposition rate of Na_2SiF_6 increases with increasing temperature [11]. The average activation energy was 63.23 kJ/mol. The average pre-exponential factor A was $1.807 \cdot 10^2$ min^{-1}.

Since the Na_2SiF_6 density changes during the thermal conversion of the fluidized bed (due to changes in the composition), the bed height also changes. It is known that the bed height depends on its porosity:

$$H = H_0\frac{\left(1-\varepsilon_0\right)}{\left(1-\varepsilon\right)}, \tag{5}$$

where H_0 – the height of the dense layer, m; ε – the fluidized bed porosity; ε_0 – the porosity of the dense layer.

The porosity ε depends on the Reynolds number Re and the Archimedes number Ar, and therefore depends on the particle density, velocity, density and viscosity of the fluidizing gas.

Clearly, during the thermal conversion of the fluidized bed material, a gas component (SiF_4) is released and entrained in the gas flow, increasing the mass flow rate of the resulting gas mixture compared to the initial fluidizing gas. Since this process (gas release and its "entrainment" in the gas flow) occurs throughout the entire height of the fluidized bed, the mass flow rate, and therefore the gas velocity, are not uniform across the height. Therefore, equation (5) is transformed as follows:

$$H = \int_0^{H_0}\frac{\left(1-\varepsilon_0\right)}{\left(1-\varepsilon\right)}dh. \tag{6}$$

The porosity of the fluidized bed is determined by the equation [12]:

$$\varepsilon = \left(\frac{18\,\mathrm{Re} + 0.36\,\mathrm{Re}^2}{\mathrm{Ar}}\right)^{0,21}. \tag{7}$$

The Reynolds and Archimedes numbers are defined as:

$$\mathrm{Re} = \frac{v d_p\,\rho_g}{\mu_g},$$

$$\mathrm{Ar} = \frac{d_p^3\rho_g^2 g}{\mu_g^2}\;\frac{\rho_p - \rho_g}{\rho_g}, \tag{8}$$

where v is the velocity of the fluidizing gas, m/s; d_p – particle size, m; ρ_p – particle density, kg/m^3; ρ_g – gas density, kg/m^3; μ_g – dynamic viscosity of gas, Pa · s.

The particle density is determined by equation (2).

The viscosity of gas mixtures does not obey the additivity rule. The dynamic viscosity μ_g of a gas mixture is determined by the Wilke formula [13, 14]:

$$\mu_g = \frac{\mu_{air}}{1+\left(\frac{X_{SiF_4}}{X_{air}}\right)f_1} + \frac{\mu_{SiF_4}}{1+\left(\frac{X_{air}}{X_{SiF_4}}\right)f_2}, \tag{9}$$

$$f_1 = \frac{\left[1+\left(\frac{\mu_{air}}{\mu_{SiF_4}}\right)^{\frac{1}{2}}\left(\frac{M_{SiF_4}}{M_{air}}\right)^{\frac{1}{4}}\right]^2}{4\sqrt{2}\left[1+\left(\frac{M_{air}}{M_{SiF_4}}\right)\right]^{\frac{1}{2}}}, \quad f_2 = \frac{\left[1+\left(\frac{\mu_{SiF_4}}{\mu_{air}}\right)^{\frac{1}{2}}\left(\frac{M_{air}}{M_{SiF_4}}\right)^{\frac{1}{4}}\right]^2}{4\sqrt{2}\left[1+\left(\frac{M_{SiF_4}}{M_{air}}\right)\right]^{\frac{1}{2}}}, \tag{10}$$

where X_{air}, X_{SiF4} – volume fractions of air and SiF_4 in the gas mixture; M_{air}, M_{SiF4} – molecular masses of air and SiF_4, kg/mol; $\mu_в$, μ_{SiF4} – dynamic viscosities of air and SiF_4, Pa · s:

$$M_{air} = 28.98 \cdot 10^{-3} \text{ kg/mol}; \; M_{SiF4} = 104.08 \cdot 10^{-3} \text{ kg/mol}.$$

The dynamic viscosities of air and SiF_4 depend on temperature and can be determined using the Sutherland equation:

$$\mu_{air} = \mu_{air}^{273K}\frac{273+C_{air}}{T+C_{air}}\left(\frac{T}{273}\right)^{\frac{1}{2}},$$

$$\mu_{SiF_4} = \mu_{SiF_4}^{273K}\frac{273+C_{SiF_4}}{T+C_{SiF_4}}\left(\frac{T}{273}\right)^{\frac{1}{2}}, \tag{11}$$

where μ_{air}^{273K} – dynamic viscosity of air at a temperature of 273 K, Pa · s; $\mu_{SiF_4}^{273K}$ – dynamic viscosity of SiF_4 at a temperature of 273 K, Pa · s; C_{air} – Sutherland constant for air, K; C_{SiF4} – Sutherland constant for SiF_4, K.

The velocity of the fluidizing gas is defined as:

$$v = \frac{4G}{\rho_g \pi D^2}, \tag{12}$$

where G – gas mass flow rate, kg/s; D – reactor diameter in the reaction zone, m.

The mass flow rate of gas can be defined as:

$$G = G_0 + \int_0^h dG = G_0 + \frac{d\alpha}{dt}\frac{\pi D^2}{4}\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)\int_0^h (1-\varepsilon)\,dh, \tag{13}$$

where G_0 – gas flow rate at the entrance to the reaction zone of the fluidized bed, kg/s; h – height of the considered area of the layer above the grate, m; $\frac{d\alpha}{dt}$ – rate of change of the degree of conversion of sodium hexafluorosilicate, s^{-1}.

In formula (13), the density of sodium fluoride is divided by 2.33, since during reaction (1) the volume occupied by the solid substance decreases by a factor of 2.33, which must be taken into account when determining the mass of gaseous SiF_4 released during the reaction from a unit volume of the reaction zone.

Then the flow rate of the gas mixture is determined as:

$$v = \frac{T}{293}\left(\frac{4G_0}{\rho_{air}^{nc}\pi D^2} + \frac{d\alpha}{dt}\frac{\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)}{\rho_{SiF_4}^{nc}}\int_0^h (1-\varepsilon)\,dh\right), \tag{14}$$

where ρ_{air}^{nc} – air density under normal conditions (at a temperature of 293 K), kg/m^3; $\rho_{SiF_4}^{nc}$ – density of SiF_4 under normal conditions (at a temperature of 293 K), kg/m^3.

The density of a gas mixture is defined as the ratio of the mass flow rate to the product of the velocity and the cross-sectional area of the reaction zone:

$$\rho_g = \frac{4G}{v\pi D^2} = \frac{293\rho_{air}^{nc}\left(v_0 + \frac{d\alpha}{dt}\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)\int_0^h (1-\varepsilon)\,dh\right)}{T\left(v_0 + \frac{d\alpha}{dt}\frac{\rho_{air}^{nc}\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)}{\rho_{SiF_4}^{nc}}\int_0^h (1-\varepsilon)\,dh\right)}, \quad (15)$$

where v_0 – air velocity at the reactor inlet, m/s.

The volume fractions of air and SiF_4 are determined as:

$$X_{air} = \frac{T}{293}\frac{v_0}{v} = \frac{1}{1 + \frac{d\alpha}{dt}\frac{\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)}{v_0\rho_{SiF_4}^{nc}}\int_0^h (1-\varepsilon)\,dh}, \quad (16)$$

$$X_{SiF_4} = 1 - \frac{T}{293}\frac{v_0}{v} = \frac{\frac{d\alpha}{dt}\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)\int_0^h (1-\varepsilon)\,dh}{v_0\rho_{SiF_4}^{nc} + \frac{d\alpha}{dt}\left(\rho_{Na_2SiF_6} - \frac{\rho_{NaF}}{2.33}\right)\int_0^h (1-\varepsilon)\,dh}. \quad (17)$$

Based on expression (3), we can write a formula for the rate of change of the conversion degree:

$$\frac{d\alpha}{dt} = \frac{k^{\frac{1}{n}}\,t^{\frac{1}{n}-1}}{n}\exp\left(-(kt)^{\frac{1}{n}}\right). \quad (18)$$

A program was developed for calculating layer parameters using a numerical method.

The following initial data were adopted for the calculation: reactor diameter D equal to 0.05 m; dense bed height H_0 equal to 0.1 m; dense bed porosity ε_0 equal to 0.3; air dynamic viscosity μ_{air}^{273K} at 273 K equal to $18.2 \cdot 10^{-6}$ Pa · s; dynamic viscosity $\mu_{SiF_4}^{273K}$ of SiF_4 at 273 K equal to $14.5 \cdot 10^{-6}$ Pa · s; Sutherland constant C_{air} for air equal to 124 K; Sutherland constant C_{SiF4} for SiF_4 equal to 300 K; air mass flow rate G_0 equal to $1.6 \cdot 10^{-4}$ kg/s; air density ρ_{air}^{nc} under normal conditions equal to 1.22 kg/m^3; sodium hexafluorosilicate density $\rho_{Na_2SiF_6}$ equal to 2 680 kg/m^3; sodium fluoride density ρ_{NaF} equal to 2 790 kg/m^3; SiF_4 density $\rho_{SiF_4}^{nc}$ under normal conditions equal to 4.64 kg/m^3.

Figure 1 shows graphs of layer porosity versus height and temperature. The graphs are presented for temperatures of 773, 873, 973, and 1 073 K and conversion levels of 0.1, 0.5, 0.7, and 0.9.

The graphs show that the porosity of the fluidized bed increases with height. This is due to the addition of the thermal conversion products of the bed material to the fluidizing gas. Furthermore, with increasing temperature, the bed porosity increases, which is associated with an increased Na_2SiF_6 conversion rate. Once a conversion rate of 0.9 is reached, the bed porosity increases significantly more slowly with height due to a rapid decrease in the conversion rate and, consequently, a decrease in the amount of gaseous products replenishing the fluidizing agent.

Clearly, as the bed porosity increases, so does the height of the fluidized bed. Figure 2 shows graphs of the relative bed height (relative to the dense bed height) as a function of the Na_2SiF_6 conversion rate and temperature.

The figure shows that at all temperatures considered, the conversion process is accompanied by an increase in the height of the fluidized bed. At temperatures of 973 and 1 073 K, the height initially increases, followed by a slow decrease. This is due to the fact that the thermal conversion of sodium hexafluorosilicate releases gaseous silicon tetrafluoride SiF_4. The specific consumption of silicon tetrafluoride (SiF_4) during a single Na_2SiF_6 feed is variable and depends on time and temperature (initially, the specific consumption increases, then decreases after reaching its maximum value).

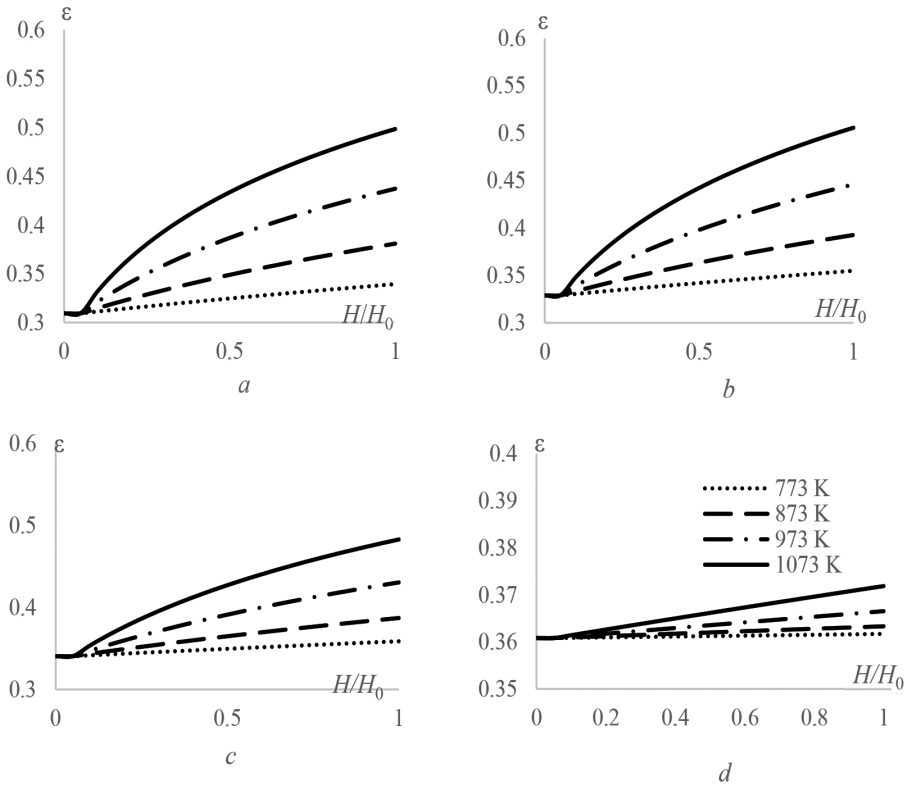


Fig. 1. Calculated dependences of porosity on temperature and relative height of the fluidized bed at a conversion degree of Na_2SiF_6 of 0.1 (*a*), 0.5 (*b*), 0.7 (*c*) and 0.9 (*d*)

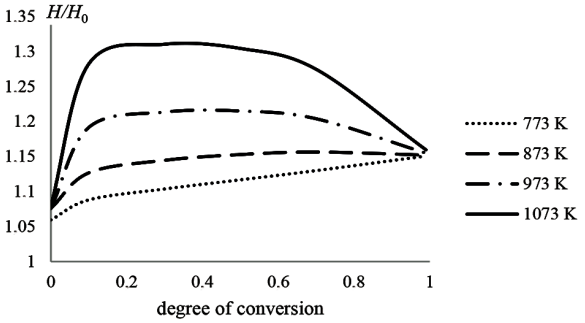


Fig. 2. Calculated dependences of the relative layer height (related to the dense layer height) on the degree of conversion of Na_2SiF_6 and on temperature

The specific consumption of SiF_4 during a single load is determined by the equation

$$W_{SiF_4} = \frac{d\alpha}{dt}\left(1-\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right) = \frac{k^{\frac{1}{n}}\,t^{\frac{1}{n}-1}}{n}\left(1-\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right)\exp\left(-(kt)^{\frac{1}{n}}\right). \quad (19)$$

Figure 3 shows graphs of the dependence of the specific consumption of silicon tetrafluoride on time at conversion temperatures of 773, 873, 973 and 1 073 K.

To ensure a constant specific SiF_4 consumption, it is necessary to continuously feed Na_2SiF_6 (replenish the bed) with continuous removal of the reacted solid residue. Assuming that the conversion degree of the reacted material should be at least 0.9, the following condition must be met to ensure a constant specific SiF_4 consumption:

$$\frac{d\alpha}{dt}M_{Na_2SiF_6}\left(1-\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right)+0{,}9\left(1+\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right)\int_{t_0}^{t}\left(G_{Na_2SiF_6}\int_{0}^{t-t_0}\frac{d\alpha}{dt}dt\right)dt = \text{const},$$

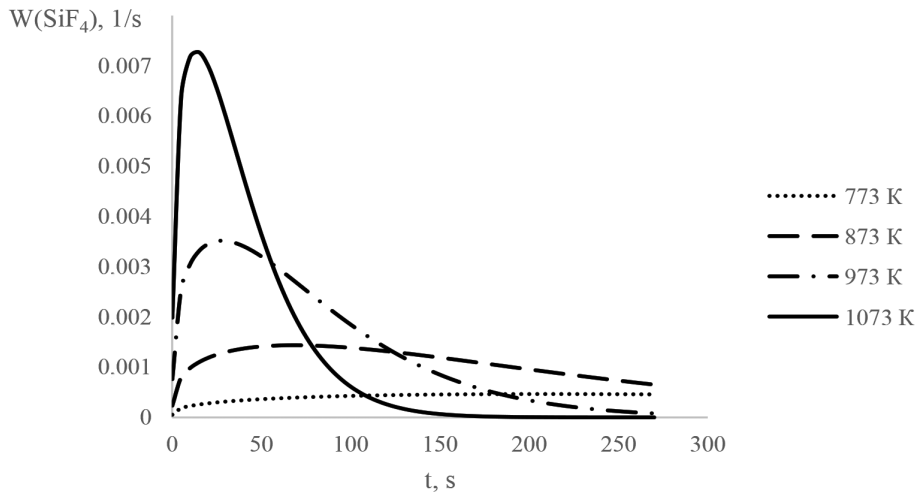


Fig. 3. Calculated dependences of the specific consumption of silicon tetrafluoride on time at conversion temperatures of 773, 873, 973 and 1 073 K

$$\frac{d^2\alpha}{dt^2} M_{Na_2SiF_6}\left(1-\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right)+0,9\left(1+\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right)G_{Na_2SiF_6}\int_0^{t-t_0}\frac{d\alpha}{dt}\,dt=0, \tag{20}$$

where $M_{Na2SiF6}$ – initial loading weight of sodium hexafluorosilicate, kg; $G_{Na2SiF6}$ – mass flow rate of sodium hexafluorosilicate, kg/s; t_0 – time of start of layer replenishment, s.

By solving equation (20) taking into account expression (18), we can obtain an analytical expression for the mass flow rate of sodium hexafluorosilicate

$$G_{Na_2SiF_6}=\frac{M_{Na_2SiF_6}}{0,9}\frac{(kt)^{\frac{1}{n}}}{(nt)^2}\left(\frac{2.33\,\rho_{Na_2SiF_6}-\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}+\rho_{NaF}}\right)\frac{(kt)^{\frac{1}{n}}+n-1}{1-\exp\left(1-n-(kt)^{\frac{1}{n}}\right)}. \tag{21}$$

Figure 4 shows graphs of the Na_2SiF_6 mass flow rate versus time for temperatures of 773, 873, 973, and 1 073 K. It is evident that to ensure a constant SiF_4 flow rate, the Na_2SiF_6 flow rate must be increased. During the first half hour, the consumption of sodium hexafluorosilicate should be increased by approximately 4.2 times, during the second half hour – by 1.3 times, etc.

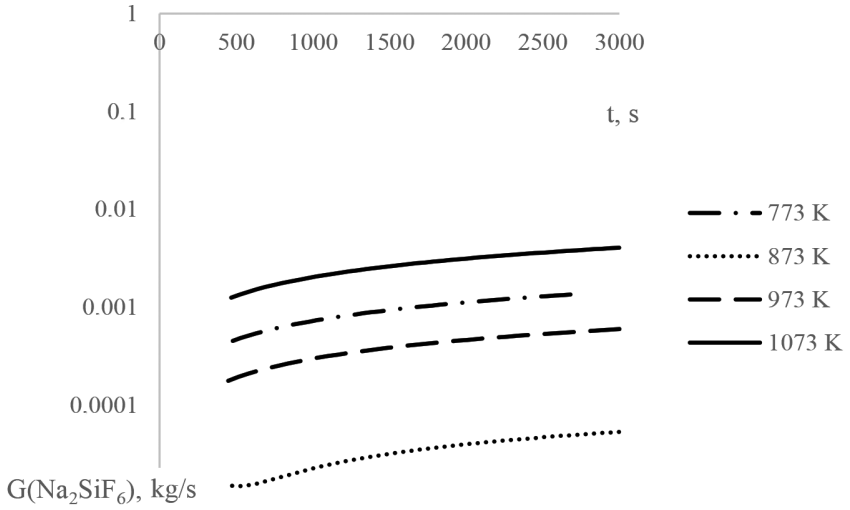


Fig. 4. Calculated dependences of the mass flow rate of Na_2SiF_6 on time for temperatures of 773, 873, 973 and 1 073 K

The mass flow rate of the discharged waste material is determined as:

$$G_{NaF} = G_{Na_2SiF_6}\left(0.1 + 0.9\frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right). \quad (22)$$

Obviously, it is necessary to replenish the layer with "fresh" sodium hexafluorosilicate not at the beginning, but after a period of time equal to $t_0 = \frac{(1-n)^n}{k}$. At this point in time, the specific consumption of silicon tetrafluoride will be at its maximum (see Fig. 3). Further, with a continuous supply of Na_2SiF_6 and removal of NaF, the specific consumption of SiF_4 will be constant and equal to:

$$W_{SiF_4}^{max} = \frac{k(1-n)^{1-n}}{n}\left(1 - \frac{\rho_{NaF}}{2.33\,\rho_{Na_2SiF_6}}\right)\exp\left(-(1-n)\right). \quad (23)$$

Implementation of the presented model in a fluidized bed reactor will enable the thermal conversion of sodium hexafluorosilicate Na_2SiF_6 in a fluidized bed in an air environment with continuous removal of the reacted dispersed material and supply of the initial dispersed material to replenish the fluidized bed. This will reduce the equipment cycle time without regular reactor shutdowns and startups, thereby increasing the reliability of the sodium hexafluorosilicate Na_2SiF_6 thermal decomposition equipment (by ensuring constant equipment operation) and increasing the productivity of silicon tetrafluoride SiF_4 production (by reducing the cycle time).

Acknowledgements. This work was supported by the Belarusian Republican Foundation for Fundamental Research (grant T26M-001).

Благодарности. Работа выполнена при поддержке Белорусского фонда фундаментальных исследований (грант T26M-001).

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