Selection of control system parameters for production of nanostructures concentrates

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Abstract. To control the production process of nanomodifiers, automated control and regulation of technological parameters, equipment protection, a process control system for the associated production of nanostructured concentrates is being developed. The optimal process for extracting nanostructures from technogenic wastes of silicon production is flotation.

1. Introduction
Nanostructures in the form of globular silicon dioxide and fullerene-like carbon have many promising fields of application for the production of materials with improved physic-chemical and operational characteristics [1-5], for example, for use in the production of metal alloys and composites for the aviation and automotive industry, catalysts and filters for the chemical industry, rubber products (in the form of special rubber, rubber-plastics, automobile and aircraft tires), paints, glass, ceramics, bitumen, mastic. Mass use of these nanostructures is limited by the methods of their production. At present, the known ways of producing nanostructures require special power, increased consumption of raw materials and energy [6-9].

Based on the results of exploratory studies, a technology for the enrichment and conditioning of concentrates from stale sludges of soda-bicarbonate gas-cleaning systems for ore-thermal electric arc furnaces during the production of silicon was proposed [10-13]. This technology is based on the isolation of not microparticles, but nanostructures in the form of fullerene-like carbon particles (CNTs) and spherical silicon dioxide.

To control the production process of nanomodifiers, automated control and regulation of technological parameters, equipment protection, a process control system for the associated production of nanostructured concentrates (ASC TPNC) is being developed. It is known that the quality of process control depends on correctly selected reference information.

2. Purpose of the study
To assess the quality of the initial settings of the automated flotation process control system for the extraction of carbon nanotubes using analytical calculation methods and their optimization using special software.

3. Description of the technological process
On the basis of laboratory studies, it has been established that flotation is an optimal process for extracting nanostructures from technogenic waste from the production of silicon. In this case, the
flotation of nano-sized particles and micron-sized particles should be carried out in a laminar flow of pulp with the least number of elementary flotation cycles.

Figure 1 shows the scheme of the flotation process, where the initial pulp (Gp) of a given density (ρp) and with the concentration of nanostructures (Cp) is fed to the flotation machine. Depending on the consumption of Gp in the flotation machine, kerosene and pine oil (Gf) in the amount of 0.002-0.003 m³ / h are added as surfactants. To activate the hydrophilized particles of the valuable component, pressure flotation is applied under the regime, which ensures the formation of air bubbles comparable to the sizes of the floated particles. Control over the size of air bubbles is carried out using a high-speed video camera with a frame rate of at least 1000 f / s at a resolution of 1024x1024. Aeration of the pulp is carried out with the help of an ejector, the air into which is injected by a compressor with a pressure of 2-4 bars from the atmosphere.

As a result of the flotation process, by controlling the mass flow rate of air (Ga), nanostructures are separated from the initial pulp. A chamber product (Gcp) with a concentration of silicon dioxide nanostructures (Ccp) emerges from the bottom of the flotation machine, and a foam product (Gfm) with a concentration of carbon nanostructures (Cfm) is on top of the flotation machine.

Figure 1. A structural scheme of the flotation process

Thus, Figure 1 shows that the indicator of the efficiency of the flotation process is the concentration of carbon nanostructures in a foam product (Cfp). The aim of the control is to ensure that the Cfp is equal to a given concentration at the maximum possible value for a given setting. The quality and quantity of the final product will depend on the correctly selected operating parameters of the flotation process.

4. Analysis of material balances of the process

To determine the parameters of the flotation process control system, we will first perform a material balance analysis that will make up a structural information scheme with the separation of input (control variables), perturbing effects (controlled and uncontrolled), output values and regime parameters.

The choice of parameters is carried out on the basis of the analysis of material balances in the static and dynamic modes of operation of equipment; taking into account the parameters of the technological process, not included in the balances, but characterizing the state of the process and equipment, and parameters that determine the state of occupational safety and health. The general expression for any balance in the analytical form is written as:

\[ \sum_{i=1}^{i=k} X_{IN} + \sum_{j=1}^{j=n} Y_{OUT} = 0 = K_0 \frac{dL}{dt} \left( at, L = \text{const}, \frac{dL}{dt} = 0 \right), \]  

(1)
where, \(X_{IN}\) - the input components of balance; 
\(Y_{OUT}\) - the output components of balance; 
\(K_0 \cdot dL / dt\) is a term characterizing the accumulation of matter or energy.

Equilibrium of balance to zero is observed only in the static mode of operation of the object. If there is a balance violation (the dynamic mode of the object), then on the right side of the balance there is a term containing the coefficient and derivative of the process parameter that characterizes the state of the given balance.

The process of flotation of raw materials is determined by the flow of five material balances.

4.1. Material balance by the target component in the process of flotation in a dynamic mode

The equation of dynamics:

\[
\rho_p \cdot V_p \cdot \frac{dC_p}{dt} = G_p \cdot C_p - G_{cp} \cdot C_{cp} - G_{fm} \cdot C_{fm}
\]

(2)

The equation of static with

\[
\frac{dC_p}{dt} = 0 \quad ; \quad G_p \cdot C_p = G_{cm} \cdot C_{cm} + G_{fm} \cdot C_{fm}
\]

(3)

where, \(\rho_p\) - pulp density; \(V_p\) - volume of the pulp; \(G_p\) - mass flow of the initial pulp; \(G_{cp}\) - mass consumption of the chamber product; \(G_{fm}\) - mass flow rate of the foam product; \(C_p\) - concentration of nanostructures in the initial pulp; \(C_{cp}\) - concentration of nanostructures in a chamber product; \(C_{fm}\) - concentration of nanostructures in the foam product.

4.2. Material balance by the concentration of CNTs in a chamber product

The equation of dynamics:

\[
\rho_{cp} \cdot V_{cp} \cdot \frac{dC_{cp}}{dt} = G_p \cdot C_p - G_{cp} \cdot C_{cp} - G_{fm} \cdot C_{fm},
\]

(4)

The equation of static with

\[
\frac{dC_{cp}}{dt} = 0 \quad ; \quad G_{cp} \cdot C_{cp} = G_{cp} \cdot C_{cp} - G_{fm} \cdot C_{fm}
\]

(5)

where: \(\rho_{cp}\) - density of the chamber product; \(V_{cp}\) - volume of the chamber product. 
On the basis of (5) and (6) we can take:

\[
C_{cp} = f(G_{cp}, G_p, G_{fm})
\]

(6)

4.3. Material balance by the concentration of CNTs in a foam product

The equation of dynamics:

\[
\rho_{fm} \cdot V_{fm} \cdot \frac{dC_{fm}}{dt} = G_p \cdot C_p - G_{cp} \cdot C_{cp} - G_{fm} \cdot C_{fm}
\]

(7)

The equation of static with
\[
\frac{dC_{fm}}{dt} = 0; \quad G_{fm} \cdot C_{fm} = G_p \cdot C_p - G_{cp} \cdot C_{cp}
\]  

(8)

where: \(\rho_{fm}\) – density of the foam product; \(V_{fm}\) – volume of the foam product.

**4.4. Material balance by the chamber product**

The equation of dynamics:

\[
\rho_{cp} \cdot S_{FM} \cdot \frac{dh_{cp}}{dt} = G_p + G_f - G_{cp}
\]

(9)

The equation of static with

\[
\frac{dh_{cp}}{dt} = 0
\]

(10)

where: \(S_{FM}\) - sectional area of a flotation machine; \(h_{cp}\) - the level of the chamber product in the flotation machine.

On the basis of (9) and (10) we can take:

\[
h_{cp} = f(G_p, G_{cp}, G_f)
\]

(11)

**4.5. Material balance by the chamber product**

The equation of dynamics:

\[
\rho_{fm} \cdot S_{FM} \cdot \frac{dh_{fm}}{dt} = G_p + G_a + G_f - G_{cp} - G_{fm}
\]

(12)

The equation of static with

\[
\frac{dh_{fm}}{dt} = 0; \quad G_{fm} = G_p + G_a + G_f - G_{cp}
\]

(13)

where: \(h_{fm}\) – the level of the foam product in the flotation machine.

From equations (12) and (13) we can take:

\[
h_{fm} = f(G_a, G_{fm}, G_f, G_{cp})
\]

(14)

It can be seen that the presented parameters refer to:

- **control actions:** \(G_p, G_{SAS}, G_{cp}, G_f\);
- **controlled disturbances:** \(\rho_{p}, G_{s}\);
- **uncontrolled disturbances:** \(C_{p}\);
- **controlled variables:** \(C_{cp}, C_{fm}, h_{cp}, h_{fm}\).

It has been established that for the technology under consideration during the process, the density of the pulp prepared in the repulpator can fluctuate either due to changes in the composition of the original dust, or by draining the content of the filter bucket into the repulpator when it overflows. The
discrepancy of the pulp density at the outlet from the repulpator to the desired range can significantly disrupt the normal operation of the entire process, since the quality of the flotation process directly depends on this value. It is necessary to maintain the density within the prescribed limits with the help of the regulating water supply to the repulpator.

5. Calculation of the regulating device
The technological operation for the preparation of pulp with a given density is carried out in the repulpator. Density is maintained within the prescribed limits by means of a water supply regulating device. The following initial data were used to calculate the regulating device (RD):

- medium - water;
- medium temperature - 25 °C;
- density of water $\rho = 997.07 kg/m^3$;
- dynamic viscosity of water $\mu = 0.8902 \times 10^{-3} Pa\cdot s$;
- maximum mass flow rate $G_{\text{max}} = 1333.33 kg/h$;
- minimum mass flow rate $G_{\text{min}} = 1000 kg/h$;
- height of the water column in the source tank $h_1 = 3m$, in the final tank, above the outlet of the line $- h_2 = 1m$;
- level difference: from the start of the settlement area to RD $h_s = 1.5m$, from the end of the settlement section to RD $h_e = 0m$;
- piping line data: one sharp turn to 90 degrees to the regulating device. The distance from the beginning of the line to RD $l_1 = 1.8m$, after RD and to the end of the pipeline is $l_2 = 0.2m$, $L = 2m$;
- flow characteristic - linear.

At the beginning of the calculation, we translate the mass flow into a volumetric flow:

$$Q_{\text{max}} = \frac{G_{\text{max}}}{\rho} = \frac{1333.33}{997.07} = 1.337 m^3/h$$

$$Q_{\text{min}} = \frac{G_{\text{min}}}{\rho} = \frac{1000}{997.07} = 1.003 m^3/h$$

The pipeline diameter ($D_p$) is then calculated, taking into account the maximum medium flow rate (1 - 2 m/s) and the permissible fluid flow rate: $w_{\text{per}} = 2 m/s$:

$$D = \sqrt{\frac{4 * Q_{\text{max}}}{\pi * w_{\text{per}}}} = \sqrt{\frac{4 * 1.337}{3600 \times 3.14159 \times 2}} = 0.01538 m = 15.38$$

Rounding $D$ to the nearest standard value, we obtain $D_p = 15 mm$, then the speed of the medium is specified in accordance with the chosen diameter:

$$w_{\text{max}} = \frac{353.4 * Q_{\text{max}}}{D_r^2} = \frac{353.4 * 1.337}{15^2} = 472.585$$

$$w_{\text{min}} = \frac{353.4 * Q_{\text{min}}}{D_r^2} = \frac{353.4 * 1}{15^2} = 353.4$$

Let's find the pressure at the beginning and end of the calculation section:
\[ P_s = \rho \cdot g \cdot h1 = 997.07 \cdot 9.81 \cdot 3 = 29343.7701 \text{ Pa} = 0.0293 \text{ MPa} \]  
(20)

\[ P_e = \rho \cdot g \cdot h2 = 997.07 \cdot 9.81 \cdot 1 = 9781.257 \text{ Pa} = 0.00978 \text{ MPa} \]  
(21)

Now you can determine the pressure drop in the system:

\[ \Delta P_s = P_s - P_e = 0.0293 - 0.00978 = 0.0196 \text{ MPa} \]  
(22)

Let us determine the mode of motion of the liquid:

\[ \text{Re} = \frac{\omega \cdot D_p \cdot \rho}{\mu} = \frac{1.1815 \cdot 0.02 \cdot 997.07}{0.8902 \cdot 10^{-3}} = 35287.95 \]  
(23)

The fluid motion regime is turbulent, since \( \text{Re} > 10000 \).

Let's find the coefficient of hydraulic friction (for seamless steel pipes in good condition, absolute roughness of the pipe surface is \( \Delta = 0.014 \text{ mm} \)):

\[ \frac{\Delta}{D_s} = \frac{0.014}{15} = 0.00093 < 0.01 \]  
(24)

Then the coefficient of hydraulic friction is calculated by the Altshul formula.

The obtained coefficient of hydraulic friction is used when finding the given resistance coefficients of pipeline sections to RD and after it:

\[ \xi_s = \frac{10^3 \cdot \lambda \cdot K_a \cdot l_s}{D_s^4} + \xi_s \]  
(25)

To simplify further calculations, \( D_p \) is used per unit mm. As a result, the resulted resistance coefficients of the sites will be:

\[ \xi_s = \frac{10^3 \cdot \lambda \cdot K_a \cdot l_s}{D_s^4} + \xi_s \]  
(26)

\[ \xi_s = \frac{10^3 \cdot \lambda \cdot K_a \cdot l_s}{D_s^4} + \xi_s \]  
(27)

It is now possible to determine the pressure drop in the pipeline to RD and after it:

\[ \Delta P_{p1} = \frac{0.0626 \cdot \xi_s \cdot Q_{max}^2 \cdot \rho}{D_s^4} = \frac{0.0626 \cdot 0.00009 \cdot 1.337^2 \cdot 997.07}{15^4} = 0.0000002 \text{ MPa} \]  
(28)

\[ \Delta P_{p2} = \frac{0.0626 \cdot \xi_s \cdot Q_{max}^2 \cdot \rho}{D_s^4} = \frac{0.0626 \cdot 0.000008 \cdot 1.337^2 \cdot 997.07}{15^4} = 0.0000002 \text{ MPa} \]  
(29)

Thus, the total pressure losses in the pipeline line are:

\[ \Delta P_{p} = \Delta P_{p1} + \Delta P_{p2} = 0.0000002 + 0.00000002 = 0.00000022 \text{ MPa} \]  
(30)

Now we can determine the pressure drop across the RD:

\[ \Delta P_{rd} = \Delta P_{s} - \Delta P_{p} = 0.0196 - 0.00000022 = 0.0195623 \]  
(31)
A check for the possibility of cavitation occurs according to the formula:

$$\Delta P_{\text{cav}} = K_c \cdot (P_1 - P_{ss})$$  \hspace{1cm} (32)$$

$P_{ss}$ – saturated steam pressure, (at 25 °C $\ P_{ss} = 0.003169 \ \text{MPa}$);

$P_1$ – pressure at the RD input.

$$P_2 = P_1 - P_{rd} = 0.029343567 - 0.0195623 = 0.00978127 \ \text{MPa}$$  \hspace{1cm} (33)$$

As a result:

$$\Delta P_{\text{cav}} = K_c \cdot (P_1 - P_{ss}) = 0.55 \cdot (0.029343567 - 0.003169) = 0.01308728 \ \text{MPa}$$  \hspace{1cm} (34)$$

Because $\Delta P_{\text{cav}} < \Delta P_{rd}, \ 0.01308728 \ \text{MPa} < 0.0195623 \ \text{MPa}$, then the appearance of cavitation is possible [19]. Hence, it is required to determine the effective pressure drop, at which the flow can still be regulated:

$$\Delta P_{m} = K_m \cdot (P_1 - r \cdot P_{ss})$$  \hspace{1cm} (35)$$

where $K_m$ is the coefficient of critical cavitation flow (at low flow rates);

$r$ - correction factor determined by the formula.

$$r = 0.96 - 0.28 \cdot \sqrt{\frac{P_{ss}}{P_{cr}}}$$  \hspace{1cm} (36)$$

where $P_{cr}$ – critical pressure (for water $P_{cr} = 22.12 \ \text{MPa}$):

$$r = 0.96 - 0.28 \cdot \sqrt{\frac{P_{ss}}{P_{cr}}} = 0.96 - 0.28 \cdot \sqrt{\frac{0.03169}{22.12}} = 0.957. $$  \hspace{1cm} (37)$$

$$\Delta P_{m} = K_m \cdot (P_1 - r \cdot P_{ss}) = 0.65 \cdot (0.029343567 - 0.957 \cdot 0.003169) = 0.0171 \ \text{MPa}$$  \hspace{1cm} (38)$$

Since $\Delta P_{m} < \Delta P_{rd}, \ 0.0171 \ \text{MPa} < 0.0195612 \ \text{MPa}$ - the maximum flow capacity of the RD calculated through the effective critical pressure drop according to the formula:

$$K_{v_{\text{max}}} = 0.01 \cdot Q_{\text{max}} \cdot \sqrt[3]{\frac{\rho}{\Delta P_{m}}} = 0.01 \cdot 1.337 \cdot \sqrt[3]{\frac{997.07}{0.0171}} = 0.01337 \cdot \sqrt{58300.95} = 3.229. $$  \hspace{1cm} (39)$$

The minimum throughput of RD will be:

$$K_{v_{\text{min}}} = 0.01 \cdot Q_{\text{min}} \cdot \sqrt[3]{\frac{\rho}{\Delta P_{m}}} = 0.01 \cdot 1.003 \cdot \sqrt[3]{\frac{997.07}{0.0171}} = 0.01003 \cdot \sqrt{58300.95} = 2.422. $$  \hspace{1cm} (40)$$

Conditional throughput of the RD is determined by the formula:

$$K_{vy} = K_{v_{\text{max}}} \cdot \eta$$  \hspace{1cm} (41)$$

where $\eta$ - safety factor ( $\eta = 1.1 \div 1.25$ with a linear flow characteristic);

$$K_{vy} = K_{v_{\text{max}}} \cdot \eta = 3.229 \cdot 1.25 = 4.04. $$  \hspace{1cm} (42)$$
Thus, the choice of RD size will be determined by the following conditions: 

\[ 0.25 \cdot D_y \leq D_y \leq D_z; \]
\[ 3.75 \leq D_y \leq 15. \]

At the same time, the permissible value of the velocity of the medium (for liquids) should not exceed 5 m/s:

\[ w_{per} = \frac{353.4 \cdot Q_{max}}{D_y^2} \leq \frac{353.4 \cdot 1.337}{15^2} = \frac{472.585}{225} = 2.1 \text{ m/s} \]  

(43)

The hydraulic module of the system is determined by the formula:

\[ n = \sqrt{\frac{\Delta P_{p}}{\Delta P_{rd}}} = \sqrt{\frac{0.00000022}{0.0195623}} = 0.0034. \]  

(44)

Depending on the set flow characteristic and the resulting hydraulic module, the shape of the flow characteristic is determined. With \( n < 1.2 \) the selected flow characteristic is linear.

6. Results of the study and their discussion

As a result, according to the calculations, the tuning parameters of the flotation process were obtained. To check the tuning parameters and optimize them, we use the Simulink package. In the Simulink environment, a transient process has been constructed based on the received tuning parameters. At the same time, analysis of the obtained transient process has shown that the calculated tuning parameters provide regulation, but they have a long regulation time. In this regard, to optimize the transient, internal settings of the PID controller were applied. As a result, the control time has decreased, and as a result, the quality of the new transient process has improved. So these parameters were adopted for the initial settings of the ASC TPNC of the flotation process.

7. Conclusion

To control the production process of nanomodifiers, automated control and regulation of technological parameters, equipment protection, a process control system for the associated production of nanostructured concentrates is being developed. The optimal process for extracting nanostructures from technogenic wastes of silicon production is flotation.

It is established that the quality of control of the technological process of flotation depends on the properly selected regulating body and its adjusting parameters. The presented results show that analytical methods of calculation do not give an accurate result, even optimization with the help of special software, does not guarantee getting correct settings of the regulator, because in real conditions, the control object is a dynamic system subjected to disturbing influences. In this regard, the results of calculations will be used as indicative and adjusted in the process of operation.

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