## Materials of Conferences

# EFFICIENCY EVALUATION OF VIBRATION MILLING OF MINERAL MATERIALS IN THE CONTEXT OF SOLID WASTES FROM HYDROGEN FLUORIDE PRODUCTION

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In this article, the results of performance studies on a laboratory vibration milling device are provided for the process of treatment of calcium sulfate-containing waste of hydrofluoric production in chemical industry. The obtained findings have allowed determining the operational parameters of semi-industrial vibration milling device using calculation analogies method. These parameters are the basis for design and construction of the experimental prototype. Pilot testing of the experimental prototype have demonstrated essentially high performance characteristics of fluoroanhydrite milling process in comparison with traditionally used equipment such as ball mills.

One of the actual ecological problems is a recycling of permanently accumulated industrial wastes. It is reasonable to search for the solution from the point of view of energy- and resource-efficient technologies.

$$CaF_2 + H_2SO_4 = 2HF + CaSO_4. \qquad (1)$$

**The main aim of this work** is to study the efficiency of vibration milling of one of the solid industrial wastes – fluoroanhydrite, the analogue of natural mineral material, anhydrite. Secondly, it is necessary to develop the scaling model for laboratory vibration equipment with the given industrial performance for one of ways of recycling calcium sulfate-containing wastes of hydrofluoric production in chemical industry.

By the reaction of hydrogen fluoride formation (1), the solid waste of anhydrous calcium sulfate affords as by-product. In technical and scientific literature, this product is referred as 'fluoroanhydrite'. Some properties of fluoroanhydrite from OJSC "Siberian chemical combine" (Seversk) are given in Table 1.

As it seen from Table 1, the chemical basis of fluoroanhydrite is a solid anhydrous calcium sulfate (up to 98,2 % wt.) in a form of pelletized material with various grain composition (from 30 mm to 0,1 mm), bulk mass in range of 1,37–1,57 t/m$^3$ and actual mass of 2,57 t/m$^3$. Previously, it was established that the most perspective direction of the neutralized fluoroanhydrite usage is the exploitation of binding properties of this waste in production of construction materials and products [1].

In Western countries, the top priority direction of fluoroanhydrite usage is the production of anhydrite-based construction materials [2]. In this case, it might be assumed that pelletized fluoroanhydrite was not grinded during the preparation of the fusion mixture, but only the excess of sulfuric acid was neutralized. Even the leader in Western hydrogen fluoride technology, "Buss A" Company does not recycle solid wastes of hydrogen fluoride production and dumps the neutralized pulp is in the nearby canyons [3–5].

It is known from the theory of binding materials that the higher the active surface of the binder, the more durable the building material. Therefore, besides neutralization of acid components in fluoroanhydrite, grinding and averaging of the raw material is required for construction industry. The process of grinding of this mineral technogenic material is a necessary stage of energy- and resource-saving technology of anhydrite binder production.

**Materials and methods.** Previously, processes of neutralization and grinding were performed in ball mill by staff of the Tomsk Polytechnic University. During this operation, relatively low volumetric efficiency of ball mill (about 0,04 t/m$^3$·hour) appeared as well as insufficient rate of mechanical activation (the maximum registered size of grain was about 0,6 mm). Respectively, when grinding rate was increased, the hour performance declined. Under these circumstances, it was necessary to improve the existent technology of grinding, thus it was proposed to use vibration-milling method

**Table 1**

Composition of the initial fluoroanhydrite from OJSC "SCC"

| Temperature of the waste, °C | Chemical composition of fluoroanhydrite, % wt. | | | | Angle of slope, degrees | Grain size (mm) and composition, % wt. | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | $CaSO_4$ | $CaF_2$ | $H_2SO_4$ | HF | | +5 | 5–2,5 | 2,5–2 | 2–1 | –1 |
| 150–230 | 88,5–98,2 | 0,5–1,8 | 0,5–10,0 | 0,01–0,2 | 31–41 | 6,7–20,2 | 8,7–20,0 | 4,2–7,2 | 14,5–46,2 | 29,4–39,2 |

for mechanical activation of fluoroanhydrite [7]. Vibration methods are reputed to have an activation impact on the material properties; due to this fact, they provide the most effective solution for a number of problems [8].

Laboratory vibration milling device (LVM) is represented as grinding chamber made from metal pipe internal diameter of 80 mm and height of 80 mm. In the bottom, it is constrained with metal perforated diaphragm (Fig. 1, 2).

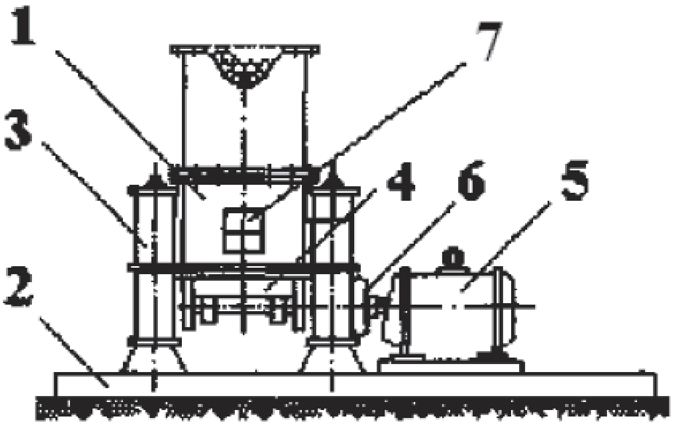

Fig. 1. Laboratory vibration milling device:
1 – grinding chamber volume of 3,2l; 2 – supporting plate; 3 – steel shock absorbers;
4–6 – electromechanical vibrator; 7 – unloading gate

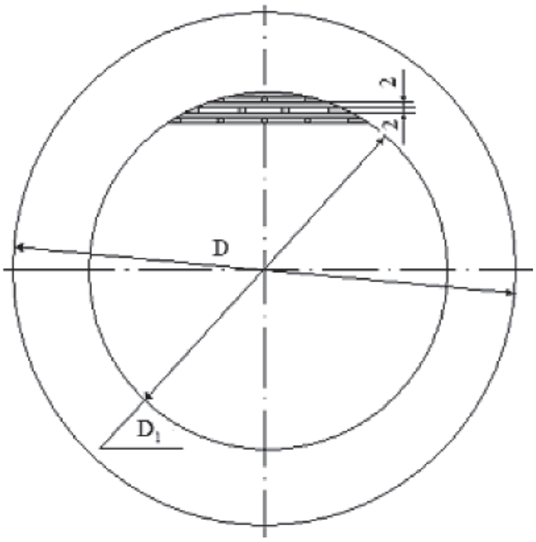

Fig. 2. Grate bar of vibration grinder

The internal chamber is filled with uniform-sized steel balls diameter of 15 mm to the height of 100 to 200 mm. Vibration milling device is equipped with steel shock absorbers, eccentric electromechanical vibrator and bunker with dosing auger conveyor used to supply the grinding chamber with pre-dozed raw material. Using this milling device, operational parameters of fluoroanhydrite milling were established to achieve the desired grain size and maximum effective performance.

As a result of numerous practical experiments, high performance characteristics of LVM were established. These data give ground for design of a pilot device with similar specific characteristics as in industrial scale. Therefore, the analysis of performance characteristics of LVM is necessary to provide recommendations for design and construction of the mentioned device.

Principal scheme of LVM could be demonstrated as a vibrating system (Fig. 3) with parameters *M*, *C* and *Fd*(*t*):

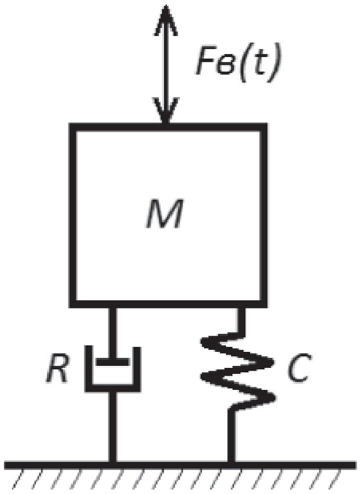

Fig. 3. Principal scheme of LVM

Where M determines the total mass of LVM elements, which oscillate, ***C*** characterizes stiffness of the suspension structure of LVM, and $Fd(t)$ is the disturbing force which affects the LVM. The total mass of oscillating elements of LVM is the sum of masses of the grinding chamber, steel balls, mill feed material, supporting plate with the chamber and eccentric vibrator attached, and the mass of electromechanical vibrator. Stiffness of the vertical suspension of LVM is formed by four springs is $523\cdot10^3$ N/m. Disturbing force of the vibrator $Fd(t)$ has amplitude $F_0$ of 2500 N and frequency of 50 Hz.

Taking into account the fact that LVM oscillates only in vertical dimension, we can formulate the equation of LVM movement with specific presumptions as:

$$M\frac{d^2x}{dt^2}+R\frac{dx}{dt}+Cx=F_{0\text{в}}\sin\omega t, \qquad (2)$$

where $M$ – the total mass of oscillating LVM elements, kg; $R$ – resistance of viscous losses, N·cm/m; $C$ – suspension stiffness, N/m; $x$ – amplitude of vertical oscillations, m; $F_0$ – amplitude of the disturbing force, N; $\omega_\text{в}$ – frequency of the disturbing force, $\text{sec}^{-1}$.

In case of oscillations in air, losses of viscous friction ($R$) can be neglected, thus equation (2) can be formulated as:

$$M\frac{d^2x}{dt^2}+Cx=F_0\sin\omega t. \qquad (3)$$

Amplitude of the oscillating chamber loaded with balls and mill feed material can be calculated by the following expression:

$$X_0=\frac{F_0}{M\sqrt{\left(\omega_{0\text{в}}^2-\omega^2\right)^2}}, \qquad (4)$$

where $X_0$ – amplitude of LVM oscillations, m; $\omega_0$ – natural oscillation frequency of LVM, $\text{sec}^{-1}$.

During practical tests on LVM, we have varied height of steel balls filling in the grinding chamber, which influenced the total mass M and its natural oscillation frequency, respectively. The mentioned operational parameters of LVM are presented in Table 2. As seen, changes of the total mass M lead to relevant changes of natural oscillation frequency of LVM.

In Table 3, calculated values of operational parameters for oscillating elements of LVM are demonstrated. These calculations are based on the equation (4), where mass of single steel ball is 13,8 gram.

The maximum obtained performance of LVM corresponded the height of steel balls in the grinding chamber of 200 mm. In this operation mode, natural oscillation frequency of LVM equals to 176 $\text{sec}^{-1}$ and oscillating amplitude of balls is 2,2 mm. Steel balls are located on mechanically unbound layers and in some periods of time can oscillate with phase shifting of π. In this case, the maximum distance between balls reaches 4,4 mm. Consequently, for ensuring the optimal operation parameters, it is necessary to perform pre-treatment procedures of the initial material in order to achieve certain grain size of particles. In the current case, the maximal grain size should not excess 4 mm. During the experiment, it was revealed that the maximal performance of grinding stage achieved with the grain size of 3,5 mm, and calculated value of the interaction force between balls of 5,94 N. Moreover, the maximal pressure in space between balls can be evaluated taking into account size of balls and force generated by every single ball in LVM. When the diameter of the single ball is 15 mm, the minimal space between balls is characterized by the diameter of 2,31 mm. In this case, calculated pressure in the inter-ball space in LVM ($P_T$) that ensures enough performance of grinding of the initial material is $14{,}2\cdot10^5$ Pa. Above mentioned studies and the modeling method are the basis for design of industrial-scale vibration milling device.

**Table 2**

Operational parameters of LVM

| Height of filling with steel balls, mm | 100 | 150 | 180 | 200 | 220 |
|---|---|---|---|---|---|
| Number of balls, pcs | 160 | 240 | 290 | 320 | 355 |
| Total mass of oscillating elements, kg | 14,7 | 15,8 | 16,5 | 16,9 | 17,3 |
| Suspension stiffness, N/m | $523\cdot10^3$ | $523\cdot10^3$ | $523\cdot10^3$ | $523\cdot10^3$ | $523\cdot10^3$ |
| Natural oscillation frequency, $\text{sec}^{-1}$ | 188,8 | 182 | 178,3 | 176 | 173,7 |

**Table 3**

Operational parameters of LVM (part II)

| Height of filling with steel balls, mm | 100 | 150 | 180 | 200 | 220 |
|---|---|---|---|---|---|
| Oscillation amplitude of balls, $\text{m}\cdot10^{-3}$ | 2,7 | 2,4 | 2,3 | 2,2 | 2,1 |
| Speed of balls, m/sec | 0,85 | 0,76 | 0,71 | 0,69 | 0,66 |
| Ball acceleration, $\text{m/sec}^2$ | 266,9 | 238,5 | 224,3 | 215,6 | 207,7 |
| Interaction force between balls, N | 7,35 | 6,57 | 6,18 | 5,94 | 5,72 |

Data obtained from the practical studies and the known size of steel balls allows to determine the value of the force that is required for milling of the initial material ($F_N$):

$$F_N \geq P_T S_M, \quad (5)$$

where $P_T$ – required pressure in the inter-ball space, Pa; $S_M$ – surface of the inter-ball space, m$^2$.

The oscillation amplitude ($x_T$) of the industrial-scale vibration milling device with the equatable performance can be evaluated if the force $F_N$ and ball mass are known:

$$x_T = \frac{F_N}{2\omega_в^2 m_b}, \quad (6)$$

where $m_b$ – mass of the single ball, kg.

The following properties were set to achieve the suitable uploading volume of material and sufficient number of steel ball layers: diameter of balls is 30 mm, the total mass of device is 700 kg, and the inter-ball space is 4,68 mm.

Basing on expressions (5), (6) and the above-mentioned properties it was revealed that calculated value of the oscillation amplitude of the device should be more than 1,12 mm. In these conditions, the task of designing an industrial-scale device is to choose suspension stiffness and amplitude of the disturbing force.

As it appears from the equation (4), the required oscillation amplitude of the device can be achieved by three possible ways, by changing one of the three parameters while the two others are constant. Adjustment of the suspension stiffness is restricted at the industrial unit by the ability to manufacture springs of enough capacity. That is why standard cylindrical compression springs “К-КТ2ШТ” were chosen for industrial unit design. The spring has rod diameter of 12,7 mm, internal spring diameter of 127 mm and 9 spring turns. The stiffness of single spring is $18{,}14 \cdot 10^3$ N/m and the total stiffness of 4 springs is $72{,}56 \cdot 10^3$ N/m, respectively. The frequency of the disturbing force is determined by characteristics of the utilized standard electromechanical vibrator. As a rule, the frequency is 50 Hz. Calculated value of the natural frequency of 700 kg industrial unit equipped with four springs is 10,18 sec$^{-1}$.

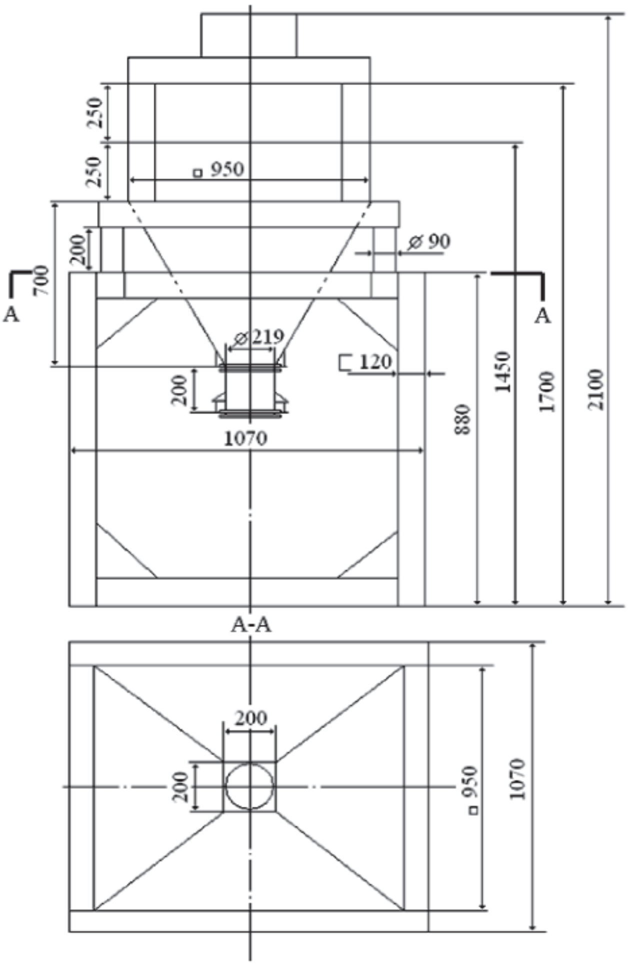

Fig. 4. Dimensions of the industrial vibration milling unit performance of 1500 kg/hour

Then, according to the equation (4), theoretically sufficient value of disturbing force should be at least 77,329 N for obtaining the required oscillation amplitude of the industrial unit of no more than 1,12 mm.

Based on the above calculations, pilotdevice with the following characteristics was constructed (Table 3, Fig. 4).

**Table 3**

Operational parameters of the pilot device

| | |
|---|---|
| Total mass of oscillating elements, kg | 700 |
| Steel balls diameter, mm | 30 |
| Suspension stiffness, $N/m \cdot 10^3$ | 72,56 |
| Disturbing force, N | 85,000 |
| Vibrator frequency, Hz | 50 |

**Results and discussions.** During pilot testing of the designed and constructed vibration milling device performance of 1500 kg/h (as presented in Fig. 4), it was revealed that it has volumetric efficiency at the level of 4000 $kg \cdot m^3/h$, what is two-order higher than volumetric efficiency of equatable ball mill. The mass of the device as well as the power of electric motor of the vibrator is one-order lower than it is required for ball mill with equatable hour performance.

**In conclusion**, it should be stressed that vibration milling process has considerable advantages in terms of resource- and energy efficiency in comparison with milling in ball mills. In this case, metal intensity of ball mill (BM) performance of 1,6 tones/hour is 13,8 tones, while metal intensity of vibration milling device (VM) is 0,7 tones; volumetric efficiency is 0,04 $t/m^3 \cdot$hour and 4,0 $t/m^3 \cdot$hour for BM and VM, respectively. In regards to energy consumption, the power of electric motor of the vibrator for BM is 55 or 34,375 kW per ton of raw material, and 5,5 or 3,67 kW for VM motor, respectively.

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