Tonkie Khimicheskie Tekhnologii = Fine Chemical Technologies.
2024;19(2):95–103 ISSN 2686-7575 (Online)

Theoretical bases of chemical technology
Теоретические основы химической технологии

UDC 66.03
https://doi.org/10.32362/2410-6593-2024-19-2-95-103

RESEARCH ARTICLE

Preparation of fine suspensions using stirred media bead mill

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Abstract

Objectives. To determine the change patterns for the main physical properties of suspensions after their grinding in bead mills, with the prospect of optimizing the preparation technology and extending the results obtained to other dispersed phases.

Methods. The study used the Fraunhofer laser diffraction method to determine particle size. The obtained data on the particle size distribution of suspensions were qualitatively verified by optical microscopy. The Brookfield relative viscosity method was used to evaluate the rheological properties of the resulting suspensions. The density of the resulting suspensions was measured by the hanging method using a calibrated pycnometer.

Results. The dependencies of the change in the particle size distribution after grinding in a bead mill were established. The viscosity of the suspensions was observed to increase following grinding. Common regularities of changes in the density of the considered suspensions were established.

Conclusions. The conducted studies showed that the physical and mechanical properties of suspensions are affected by the type and the filling ratio of the grinding media; the residence time of the suspension in the grinding chamber; the number of grinding operations; mill designs.

Keywords

bead mill, stirred media mill, grinding, particle size distribution, suspensions, disperse systems, Fraunhofer diffraction, suspension viscosity

Submitted: 01.03.2023
Revised: 12.05.2023
Accepted: 14.03.2024

For citation

Elinevskaya L.S., Dzardanov D.V., Ulybina O.V., Ivanov R.N. Preparation of fine suspensions using stirred media bead mill. *Tonk. Khim. Tekhnol. = Fine Chem. Technol*. 2024;19(2):95–103. https://doi.org/10.32362/2410-6593-2024-19-2-95-103

НАУЧНАЯ СТАТЬЯ

Приготовление тонкодисперсных суспензий с использованием бисерных мельниц

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Аннотация

Цели. Изучение закономерностей изменения основных физических свойств суспензий после их измельчения в бисерных мельницах с перспективой оптимизации технологии приготовления и распространения полученных результатов на другие дисперсионные фазы.

Методы. Размеры частиц определяли с помощью лазерной дифракции Фраунгофера. Полученные данные по дисперсному составу суспензий качественно проверяли оптической микроскопией. Для оценки реологических свойств полученных суспензий использовали метод определения кажущейся динамической вязкости по Брукфильду. Плотность полученных суспензий измеряли навесным методом с помощью калиброванного пикнометра.

Результаты. Установлены зависимости изменения дисперсного состава после измельчения суспензий в бисерной мельнице. Было обнаружено увеличение вязкости суспензий после процесса размола. Установлены общие закономерности изменения плотности рассматриваемых суспензий.

Выводы. Проведенные исследования показали, что на физико-механические свойства суспензий влияют вид и степень загрузки используемого бисера, время пребывания суспензии в размольной камере, количество операций измельчения, конструкции мельницы.

Ключевые слова

бисерная мельница, диспергирование, размер частиц, суспензии, функции распределения частиц, микрогетерогенные дисперсные системы, дифракция Фраунгофера, вязкость суспензий

Поступила: 01.03.2023
Доработана: 12.05.2023
Принята в печать: 14.03.2024

Для цитирования

Елиневская Л.С., Дзарданов Д.В., Улыбина О.В., Иванов Р.Н. Приготовление тонкодисперсных суспензий с использованием бисерных мельниц. *Тонкие химические технологии*. 2024;19(2):95–103. https://doi.org/10.32362/2410-6593-2024-19-2-95-103

INTRODUCTION

Heterogeneous systems in the form of suspensions are widely applicable and convenient products in many areas of production and human activity. Depending on the application and the physicochemical properties of their constituent components, suspensions vary greatly in terms of their quality characteristics and the methods used to produce them. This present work sets out to examine the process of obtaining finely dispersed products using bead mills.

Bead mills have become widespread in recent years in such areas as the production of plant protection chemicals, paints, pharmaceuticals and food products, as well as in the production of building materials, extraction of natural resources, etc. Compared with other dispersion devices, bead mills typically have lower energy costs for the grinding process [1]. However, despite the fairly wide distribution of such devices, the regularities of their operation have so far been poorly studied [2]. This, in particular, can be due to the complexity of the processes occurring in them during grinding, which involve a significant number of parameters [3] that affect the actual grinding of dispersion phase particles. In addition to purely technological characteristics, such as the size, hardness and degree of filling of the grinding bodies, suspension flow rate, mill design, etc., it is also worth noting the ongoing increase in the active surface area of the ground components. This, in turn, can be associated with various surface phenomena affecting the final product. In addition, the very concept of quality is determined by a different combination of physical and chemical characteristics depending on its intended purpose. Among these characteristics, in addition to particle size before and after grinding, it is also worth noting the rheological behavior of suspensions, their aggregation and sedimentation stability, as well as the uniformity of distribution of particles of the dispersion phase, etc.

The construction of a single algorithm based on available theories and capable of including all of the

above parameters for working with a bead mill often appears as a more labor-intensive and complex than the grinding itself. For this reason, the operating modes of such devices are typically selected on an *ad hoc* basis [2, 4]. However, this results in a lack of numerical data on the process of grinding suspensions in bead mills. Approaches to the theoretical description of the operating patterns of bead mills are presented in [1, 3, 5–14] together with some data obtained from experimental studies.

The purpose of the present work is to refine the above-mentioned theoretical basis, experimentally study the influence of the main technological parameters of bead mills on the physical and mechanical properties of the resulting suspensions (disperse composition, viscosity, and density), as well as to identify possible regularities for their further extension to other systems and practical application in production processes.

MATERIALS AND METHODS

The objects of study were aqueous suspensions of chalk and kaolin, which are used in the production of a number of products: plant protection agents, fine fillers, building materials and building mixtures, paper and glass products, paints, cosmetics, etc. The process of grinding suspensions in bead mills is directly associated with colloidal phenomena. In particular, the surface area of the solid phase, which increases during grinding, can interact differently with the dispersion medium, which does not exclude the formation of aggregates from the resulting crushed particles. The studied water suspensions of chalk and kaolin have sufficient aggregative stability, which makes it possible to neglect the influence of surface phenomena on the grinding process.

Experiments were carried out on LabStar (*Netzsch*, Germany) and MultiLab (*WAB*, Switzerland) laboratory mills. These machines differ in terms of the size of the grinding chambers, the types of mixing devices on the rotor, and the systems for separating beads from the product. The LabStar mill grinding chamber has a volume of 0.9 L, an internal diameter of 90 mm, and a length of 187.9 mm. A mesh cartridge with a centrifugal bead ejection system is used as a system for separating beads from the product. In the LabStar mill, ZetaBeads (*Netzsch*, Germany) 0.6 ceramic beads with a load of 61.7 vol % were used as grinding media.

The MultiLab grinding chamber has a volume of 0.561 L, an internal diameter of 77 mm, and a length of 150 mm. A slot classifier (*WAB*, Switzerland) was used as a system for separating beads from the product. Glass beads SL 7505 (*Sigmund Lindner GmbH*, Germany) with a load of 80 vol % were used as grinding media in this mill.

During the experiments, chalk of MTD-2 grade (*MelStrom*, Russia), dry enriched kaolin from the Chekmakul deposit (GOST 19608-84[1], *Novokaolinovyi GOK*, Russia) was used with distilled water according to GOST R 58144-2018[2]. The solid content phase in the chalk suspension was 40 wt %, while in the aqueous suspension of kaolin, it was 25 wt %. The experiments were carried out on a laboratory installation as depicted in Fig. 1. The suspension was prepared in beaker B_1. A paddle mixer was used for mixing. A predetermined amount of chalk or kaolin was added into a given amount of water with continuous stirring. After loading all the components, the resulting suspension was additionally stirred for 10–15 min. Then a sample of the resulting suspension was taken to measure its initial dispersion, viscosity, and density.

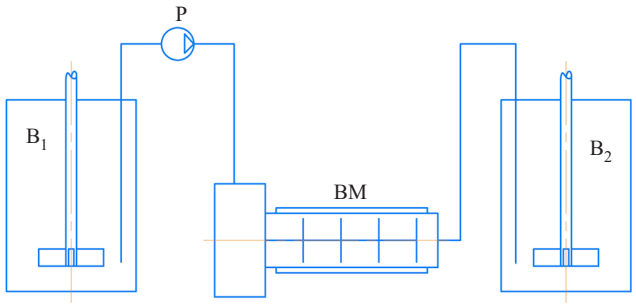

Fig. 1. Scheme of the laboratory assembly: B_1 and B_2 are chemical beakers with an overhead stirrer; P is a peristaltic pump; BM is an agitator bead mill

After preparing the initial suspension and setting certain experimental parameters, the suspension was ground in a continuous mode. The initial suspension was fed into a bead mill (BM) using a peristaltic pump (H) equipped with a supply hose with a diameter of 13 × 2.5 mm. Filling of the mills was typically performed at a pump rotor speed of 15 rpm. A mill rotor speed for the LabStar mill was 1000 rpm, while for the MultiLab mill, filling was carried out in rotor-off mode. In all the experiments, the pump rotor speed during the grinding process was 50 rpm, which corresponds to a volumetric flow rate of V = 350 mL/min for the LabStar mill and V = 195 mL/min for the MultiLab mill. In all the experiments, the rotor speed ω of the LabStar mill was 3000 rpm, while that of the MultiLab was 2986 rpm. Immediately after the suspension in B_1 ran out, the grinding process was stopped. Next, in order

[1] GOST 19608-84. State Standard of the USSR. Enriched kaolin for rubber and plastic products, artificial leather and fabrics. Technical conditions. Moscow: USSR State Committee for Standards; 1984.

[2] GOST R 58144-2018. National Standard of the Russian Federation. Distilled water. Technical conditions. Moscow: Russian Institute of Standardization; 2022.

to average the properties, the resulting suspension was stirred in beaker B_2 for 10–15 min. Then a sample was taken to measure the dispersion, viscosity, and density of the finished product. Finally, beakers B_2 and B_1 were swapped, and additional sampling passes were carried out.

The mill jackets were connected to a liquid thermostat. The latter was set at 3°C and filled with a water-glycol solution. During each experiment, technological parameters were recorded, and analysis of the dispersed composition, viscosity and density of the resulting suspensions—the most general characteristic properties of suspensions—was carried out.

The dispersed composition of the suspensions under study was measured using a Mastersizer 2000 laser particle analyzer (*Malvern Instruments*, UK). A sample of the suspension in an amount of 1 g was added to 30 g of distilled water and stirred for 2 min by a glass rod with a rubber tip. After placing the resulting suspension in a measuring cell, the particle size was measured. To assess the dispersed composition of the crushed materials, the following characteristics were selected [15]:

- percentage of particles with the size less than 5 μm, a_5;
- weighted average volume diameter of particles, $d(4,3)$[3].

The density of the initial suspensions was measured by the hanging method using a pycnometer according to GOST 319992.1[4]. The apparent dynamic viscosity of suspensions η was measured on a Brookfield viscometer (*AMETEK Brookfield*, USA) in accordance with GOST 25271-93[5] at three different spindle speeds, n: 20, 60 and 100 rpm.

RESULTS AND DISCUSSION

From the data presented in Figs. 2 and 3, the distribution peak for the suspensions of chalk and kaolin shifts to the left after one pass—to the region of lower particle sizes. In this case, the distribution of particles has become more monomodal.

The $d(4.3)$ value for chalk suspensions in one pass changed from 9.76 to 2.25 μm, for kaolin—from 23.55 to 17.09 μm. The change in the viscosity of the resulting suspensions associated with a decrease in the particle size of the solid phase can be seen in the graphs shown in Fig. 4. According to the dependencies, the apparent dynamic viscosity at low rotation speeds of the viscometer spindle after one pass increases about 200-fold for chalk suspensions and almost 4-fold for kaolin suspensions. Here it is worth noting that the viscosity of the finished suspensions depends not only on the particle size, but also on the physicochemical properties of the dispersed phase and the dispersion medium. The main substance in kaolin suspensions—kaolinite—can swell in water, thus forming fragile structures. Thus, despite the higher $d(4.3)$, these dispersed systems have a higher viscosity than more loaded chalk suspensions. At increased rotation speed of the viscometer spindle, the shear rate in the test sample also increases. Thus, it can be argued on the basis of the dependencies shown in Fig. 4 that all the suspensions under consideration are pseudoplastic fluids.

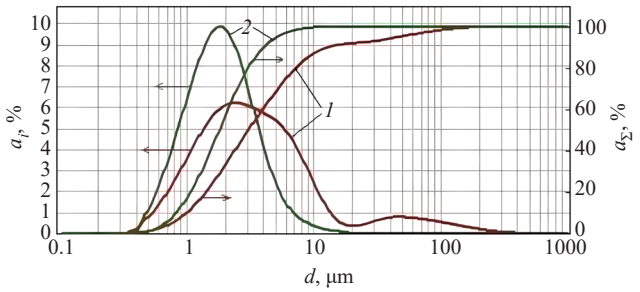

Fig. 2. Differential a_i and integral a_Σ size distribution functions of the chalk suspension particles in water for different number of passes:
(*1*) initial suspension;
(*2*) one pass

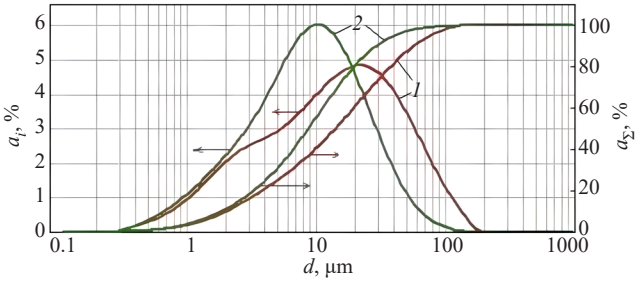

Fig. 3. Differential a_i and integral a_Σ size distribution functions of the kaolin suspension particles in water for different number of passes:
(*1*) initial suspension;
(*2*) one pass

From the data given in Table 1 it can be seen that the density of chalk and kaolin suspensions after grinding increases and is close to the density calculated using the formula for the additivity of specific volumes of solid and liquid phases. Apparently, this is due to the fact that

[3] $d(4,3)$—De Broecker or Harden average diameter—weighted average by mass or volume (average diameter of a sphere of equivalent volume), is the center of mass for density distribution functions in volume/mass units.

[4] GOST 319992.1. Interstate Standard. Paint and varnish materials. Method for determining density. Part 1. Pycnometric method. Moscow: Standartinform; 2013.

[5] GOST 25271-93. Interstate Standard. Plastics. Liquid resins, emulsions or dispersions. Determination of apparent viscosity according to Brookfield. Minsk: Interstate Council for Standardization, Metrology and Certification; 1993.

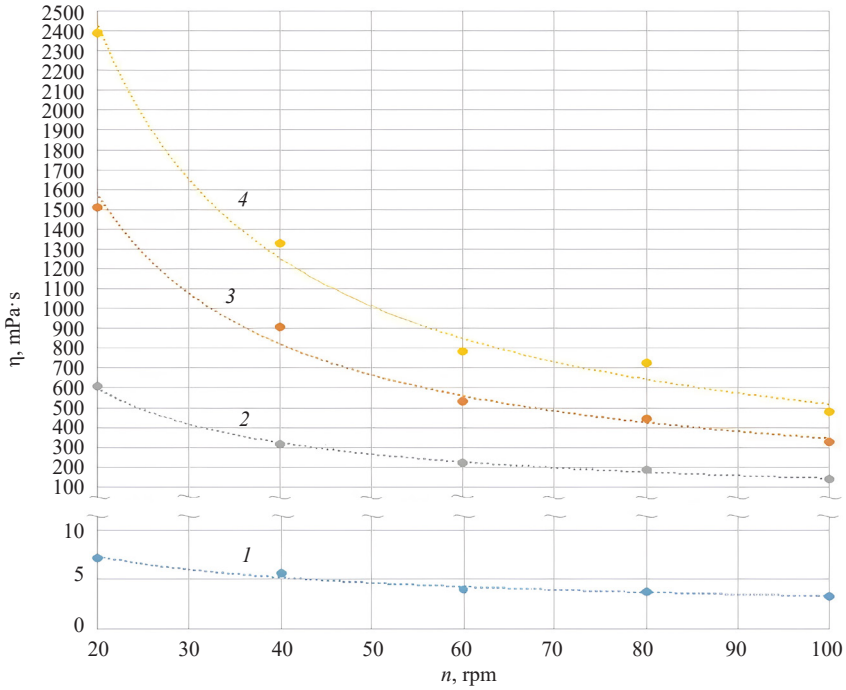

Fig. 4. Dependence of the relative viscosity of suspensions on the frequency of rotation of the viscometer spindle (LabStar mill):
(*1*) the initial suspension of chalk;
(*2*) the initial suspension of kaolin;
(*3*) the suspension of chalk after one pass;
(*4*) the suspension of kaolin after one pass

Table 1. Density of the studied suspensions depending on the degree of grinding

Suspension	Density of suspension after grinding, kg/m^3	Density of suspension after one pass grinding, kg/m^3	Calculated density of suspension, kg/m^3
40% chalk in water	1316	1333	1341
25% kaolin in water	1162	1180	1182

during the grinding process, air contained in aggregates and agglomerates of solid phase particles is released.

When studying the multi-pass grinding mode using chalk suspensions as an example, the distribution peak was observed to shift to the left and the suspension become monodisperse as the number of passes increases (Fig. 5). After three passes, the value of the weighted average volumetric diameter $d(4.3)$ changed from 9.76 to 1.77 μm. It is clear from the dependencies shown in Fig. 6 that the viscosity of the resulting suspensions increases with the number of passes. Over three grinding cycles, the viscosity index at a viscometer spindle speed of 20 rpm increased by approximately 500 times.

An optical microscope (*Olympus*, Japan) was additionally used for studying the dispersed composition of the initial and resulting suspensions. Figure 7 depicts samples of such suspensions. In particular, the photo of the initial suspensions (Fig. 7a) reveals rather large crystalline particles of the solid phase. Following three

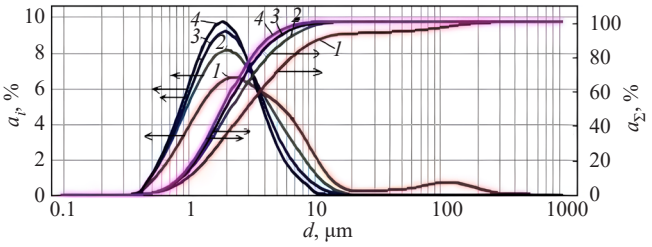

Fig. 5. Particle size distribution of chalk suspension particles in water with different number of passes (LabStar mill):
(*1*) initial suspension;
(*2*) one pass;
(*3*) two passes;
(*4*) three passes

grinding operations, all dispersed phase particles are smaller and more uniform in size (Fig. 7b).

The results show that, at the same number of passes, the particles with the size less than 5 μm for a chalk

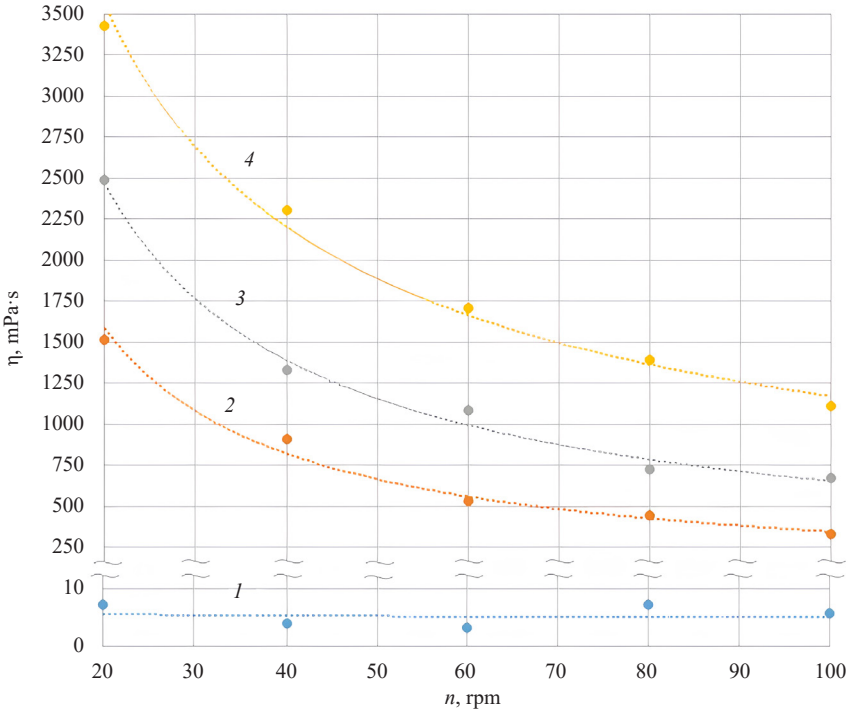

Fig. 6. Dependence of the relative viscosity of chalk suspensions on the speed of rotation of the viscometer spindle:
(*1*) initial suspension;
(*2*) one pass;
(*3*) two passes;
(*4*) three passes

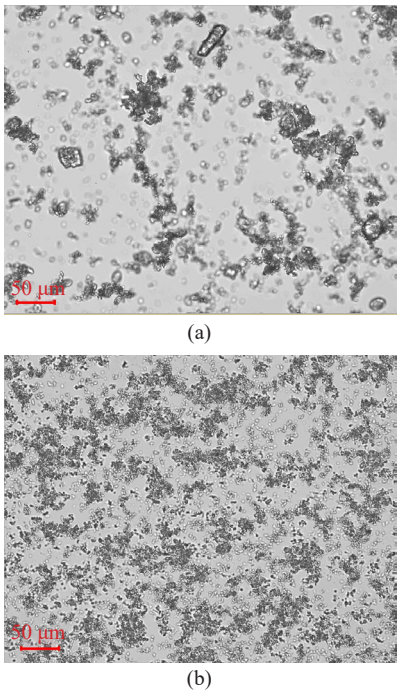

Fig. 7. Image of the initial suspension of chalk (a)
and the suspension after three passes (b)
(optical microscope)

suspension under experimental conditions is better ground in a LabStar mill than in a MultiLab mill (Fig. 8). Parameter a_5 changed in three passes from 68 to 94.1% for MultiLab and from 68 to 99.9% for LabStar. The weighted average volumetric diameter $d(4.3)$ changed in 3 passes from 9.76 to 2.27 μm for MultiLab and from 9.76 to 1.77 μm for LabStar.

As noted in [3], the average residence time of suspension particles in bead mills can be calculated using the formula (1):

$$\overline{\tau} = \frac{V_{fr} - V_b}{V}, \quad (1)$$

where V_{fr} is the free volume of the grinding chamber without beads, m^3; V_b is the total volume of bead particles, m^3; V is the volumetric flow rate of the suspension, m^3/s.

Calculation using formula (1) showed that the average residence time is 98 s for the LabStar mill and 87 s for the MultiLab mill. Thus, it will take a few seconds longer to process the solid particles in the grinding chamber of the LabStar mill.

It was proposed in [5] to introduce two parameters in order to estimate the specific energy E_m expended on the grinding process in bead mills: *SN* (number of stress events)—the number of grinding events; and *SI* (stress intensity)—collision intensity (formulas (2) and (3)):

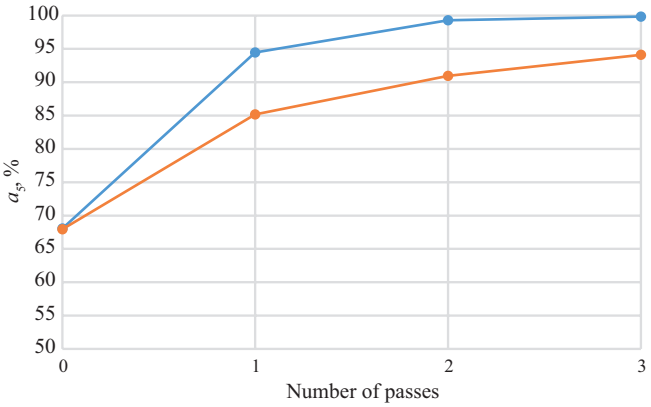

Fig. 8. Dependence of parameter a_5 for chalk suspension on the number of passes:
(*1*) MultiLab mill;
(*2*) LabStar mill

$$SN \propto \frac{\varphi_b(1-\varepsilon)}{(1-\varphi_b(1-\varepsilon))x_V} \cdot \frac{\omega\tau}{d_b^2}, \quad (2)$$

where ω is the rotation speed of the mill rotor, rpm; τ is mill operating time, s; φ_b is the volume fraction of beads in the grinding chamber; ε is bead porosity; d_b is the diameter of beads, m; x_V is the volume fraction of the solid phase in the suspension.

$$SI \propto d_b^3 \rho_b \upsilon_r^2 , \quad (3)$$

where d_b is the diameter of the beads, m; ρ_b is the bead density, kg/m^3; υ_r is the maximum linear speed of the mill rotor, m/s.

When calculating the *SN* parameters using dependence (2), it was found that the number of grinding events for the LabStar mill is approximately 3.6 times higher than for the MultiLab mill (Table 2). When calculating the *SI* parameters using dependence (3), the intensity of bead collision in the MultiLab mill was found to be approximately 5.6 times higher than in the LabStar mill (Table 2), although the peripheral rotation speeds of the mill rotors υ are quite close: for MultiLab, υ was 10.00 m/s, and for LabStar, 9.73 m/s.

When estimating the expended specific energy E_m, the following expression can also be used [5] (formula (4)):

$$E_m \propto SI \cdot SN , \quad (4)$$

Calculations using dependence (4) showed that the specific energy spent on the grinding process in the MultiLab mill is approximately 1.5 times higher than in the LabStar mill (Table 2).

Table 2. Results of the analysis of experimental data

Agitator bead mill	$\bar{\tau}$, s	*SN*	*SI*	*SI* · *SN*
LabStar (Netzsch)	98	$3.41 \cdot 10^{10}$	$1.56 \cdot 10^{-4}$	$5.32 \cdot 10^{6}$
MultiLab (WAB)	87	$0.947 \cdot 10^{10}$	$8.74 \cdot 10^{-4}$	$8.28 \cdot 10^{6}$

CONCLUSIONS

The dependencies of the basic physicochemical properties of aqueous suspensions of chalk and kaolin on the parameters of their grinding in bead mills were studied. It was established that the type of differential and integral curves following the grinding process changes in the same manner for different suspensions: both the weighted average volume diameter and the dispersion of particle sizes decrease. The viscosity of suspensions was also found to increase after grinding; this occurred regardless of the nature of the dispersion phase and its ability to form associates. The density of the suspensions under study tends to increase during the preparation process and with approaches that are calculated by the additivity rule. It was shown that changes in the main parameters of suspensions after grinding can be influenced by the properties and degree of filling of the beads and the residence time of the suspension in the grinding chamber. According to the results of a numerical assessment of the energy consumed, grinding in the LabStar mill turned out to be more energy efficient under experimental conditions than in the MultiLab mill.

Acknowledgments

The work was carried out using raw materials and equipment of the R&D department of *Firma August*. The authors thank the company's management for supporting scientific and research activities in the field of wet grinding technologies.

Authors' contributions

L.S. Elinevskaya—research idea, planning experiments, analyzing and discussing the results, and editing the text of the article.

D.V. Dzardanov—development of a methodology for conducting an experiment, participation in experimental work, and discussion of the results.

O.V. Ulybina—participation in experiments, processing of the obtained data, and participation in editing the text of the article.

R.N. Ivanov—search for scientific publications on the topic of the article, formation of a list of references, setting up and conducting experiments, processing the data obtained, and writing and formatting the text of the article.

The authors declare no conflict of interest.

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Translated from Russian into English by M. Povorin
Edited for English language and spelling by Thomas A. Beavitt