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ENERGY PARAMETERS OF THE PROCESS OF ULTRAFINE GRINDING OF MEDICINAL AND COSMETIC COMPONENTS IN A BEAD MILL

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Abstract: *The process of ultrafine grinding of components of medicines and cosmetics in a laboratory bead mill is considered. The purpose of the study is (1) to determine the energy parameters of the process of ultrafine grinding of medicinal and cosmetic components in a bead mill, as well as (2) to determine the dependences of changes in technological parameters. A suspension of castor oil and cosmetic pigment in a ratio of 60% oil and 40% pigment was used as a model body. A series of experiments was carried out, where the degree of grinding was determined using a microscope with a built-in camera and software, the temperature was measured with ds18b20 temperature sensors, the power was measured with a three-phase CNFAJ Intelligent Power Meter. When grinding the pigment "red 120" for a period of time from 0 to 45 minutes, the power decreases, the temperature of the "beads-product" system increases, and the particle size decreases. This process occurs most intensively in the first 5 minutes. Most of the energy is spent on the work, which is spent on mixing the system "beads-product", and the work, that is spent on heating the structural components of the product and the parts of the mill interacting with them, which, in turn, depend on rheological properties of the suspension.*

Keywords: *grinding, beads, mill, energy, suspension*

INTRODUCTION

Soft drugs such as ointments, pastes, liniments, gels, as well as decorative cosmetics (lipsticks, lip glosses, nail polishes and gel polishes, correctors, etc.) are concentrated dispersed colloidal suspensions or liquid-like substances (Loh, Z.H., Samanta, A.K. et al., 2015; Schmidt, J., Romeis, S., 2015), in which a high degree of grinding of the feedstock for further use is of great importance (Loh, Z.H., Samanta, A.K. et al., 2015). One way of making high quality slurries is wet grinding in bead mills. (Mende, S., Rappl, M., 2014).

In the process of grinding, energy is expended, which includes mechanical energy, which is transferred from the working member (shaft with disks) to the working bodies (beads), turns into kinetic energy (the energy of the impact of working bodies), as a result of which the working bodies grind the product suspension (Mende, S., Rappl, M., 2014). As a result, energy is generated, which is spent on mixing the "beads-product" system, the energy of directly grinding the solid fraction of the product suspension, as well as excess energy that is released in the form of heat (Mende, S., Stenger, F., 2003)

Excess energy affects the properties of the suspension, especially in the case of grinding thermolabile and expensive components of the solid fraction of the product, leads to the destruction and loss of its desired properties (Schmidt, J., Romeis, S., 2015).

EXPOSITION

Research methodology

Materials

For the study were taken: pharmaceutical castor oil, red pigment 120 iron oxide.

Castor oil pharmaceutical - natural vegetable oil. Transparent, thick and viscous colorless or slightly yellowish liquid. Stable substance, practically insoluble in water and mineral oils (Loh, Z.H., Samanta, A.K. et al., 2015). Dynamic viscosity at 20 °C - 1000 mPa·s, relative density at 25

°C - 955-968 kg/m³. In pharmacy, it is most often used in creams and ointments in a concentration of 5-12.4% as a constituent substance and solvent for dermatological ointments, as well as a plasticizer in the manufacture of tablets and capsules (Loh, Z.H., Samanta, A.K. et al., 2015; Schmidt, J., Romeis, S., 2015).

The red iron oxide pigment 120 is a dispersed system of iron (III) oxide of hexagonal structure (hematite), a powdery material of red-brown color. Bulk density is 1.0-1.1 g/cm³, density is 5.0-5.1 g/cm³. The shape of the particles is spherical.

Methods

Research of ultrafine grinding of suspensions were performed in a laboratory bead mill of periodic action (Fig. 1) (Hrininh, K., Gubenia, O., Litovchenko, I., 2019).

The laboratory bead mill (Fig. 1) consists of a frame 1, an asynchronous engine 2, a engine shaft 3, to which a work glass 5 with a lid 6 is connected, and is fixed with a clamp 4 (Hrininh, K., Gubenia, O., 2020). Asynchronous engine 2 with a capacity of 0.37 kW operates at a voltage of 380 V, has an efficiency of 71.2% and a maximum speed of 1350 rpm.

The work glass 5 is equipped with a cooling jacket 8, as well as water supply and drainage pipes, through which the coolant (water) circulates from the circulation pump 7 through the tubes connected to the pipe clamps (Hrininh, K., Gubenia, O., 2020).

The lid of the glass 6 is equipped with a shaft 9, on which the disks 10 are fixed by means of a key connection 11 (Hrininh, K., Gubenia, O., Litovchenko, I., 2019). The discs have 4 symmetrically arranged holes. The whole structure is fixed with a washer 12, a Grover washer 13 and a bolt 14. The shaft 9 with disks 10 (working member) is attached to the glass cover through the bearing assembly and connected to the motor shaft 3 through a keyway (Fig. 2). The cover is equipped with a sampler with a sieve chuck, which is screwed onto the thread (Hrininh, K., Gubenia, O., 2020). Working bodies - glass beads with a diameter of 1.88 mm (Hrininh, K., Gubenia, O., Litovchenko, I., 2019; Weber, U., Langlois, D., 2010).

The heat capacity measuring unit consists of two temperature sensors ds18b20 for water inlet and outlet. Accuracy according to the passport $\pm 0,5$ °C, at temperatures in the range from -10 °C to +85 °C. Both sensors are connected in a parasitic circuit.

Flowmeter YF-S201. Measuring range 1-30 l/min. Operating temperatures from 0 °C to +80 °C. An additional study determined the maximum deviation from the real indicators – 3,2% (rms – 0,44%).

Three-phase wattmeter CNFAJ Intelligent Power Meter. Able to measure electrical parameters (three-phase voltage, three-phase current, active power, reactive power, apparent power, power factor, frequency, etc.). Accuracy class 0,5. Frequency 40-60 Hz, accuracy 0.1 Hz (Hrininh, K., Gubenia, O., 2020).

Particle size was determined using Konus Biorex 3 - a trinocular biological microscope with achromatic optics for scientific research. The model is adapted for photography: with the help of an adapter, a camera is installed in the 3rd optical port. In the mode of operation with the camera there is a complete deviation of light on a photoport thanks to what the picture turns out contrast and light. Data processing was performed using ImageJ software.

ImageJ is an open source program for analyzing and processing images. Written in Java by the National Institutes of Health and distributed without license restrictions as a public domain. It is widely used in research related to the analysis of images of different types of food.

Suspensions were prepared according to the following recipes: 1) iron oxide red pigment 120 - 40% and castor oil - 60% (Hrininh, K., Gubenia, O., 2020); 2) iron oxide red pigment 120 - 20% and castor oil - 80%. Grinding time - 33 minutes (1980 s). During grinding from the chamber of the bead mill every 60 seconds samples were taken to measure the particle size of the suspensions and every 30 seconds data were taken from measuring devices. The bead mill operated with water cooling in a circulating way, and the water consumption was $25-27 \times 10^{-6}$ m³/sec.

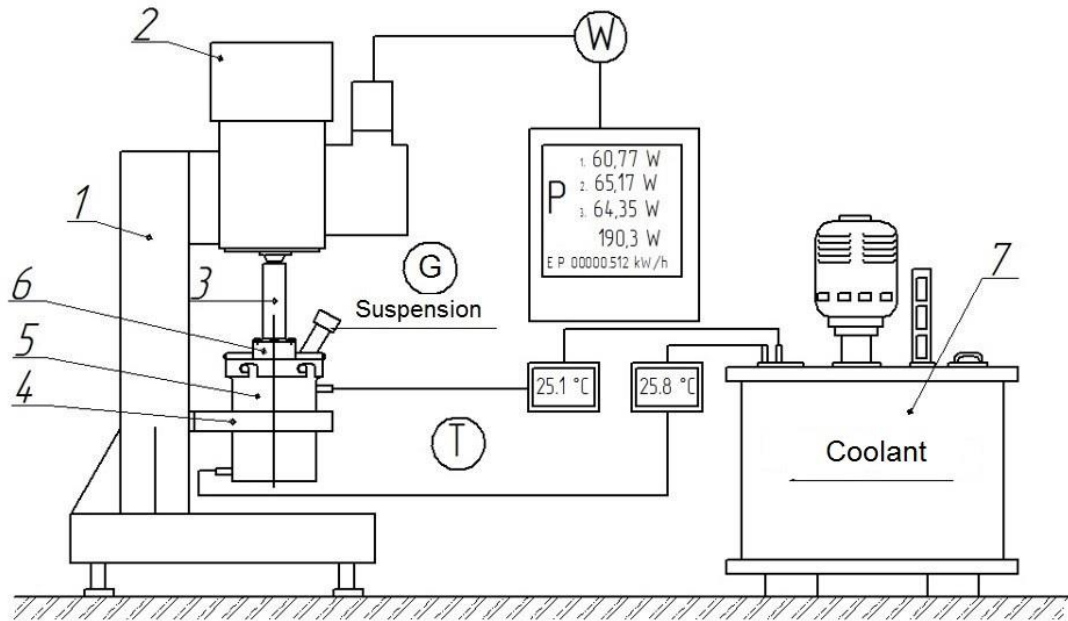


Fig.1. Experimental installation: (Hrininh K., Gubenia O., Litovchenko I., 2019): 1 - bed, 2 - engine, 3 - shaft of the engine, 4 - clamp, 5 - working glass, 6 – lid, 7 - circulation pump. W – power measurement by wattmeter; T - temperature measurement by thermometer; G – particle size was recognized by computer software.

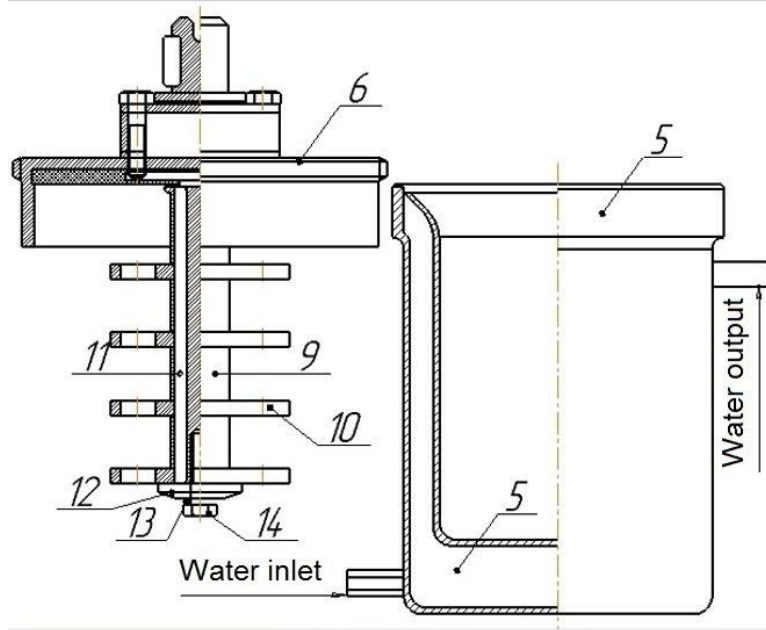


Fig.2. Experimental installation grinding capacity: (Hrininh K., Gubenia O., Litovchenko I., 2019): 5 - working glass; 6 - lid; 8 - cooling jacket; 9 - shaft; 10 - disks; 11 - key; 12 - washer; 13 - Grover's washer; 14 – bolt.

The total specific energy and thermal energy for the grinding process was considered as follows: the obtained process equation (function of power dependence) was substituted into an integral expression (formula (1) and formula (2)) (Hrininh, K., Gubenia, O., 2020):

$$E_{ts} = \int_{\tau_0}^{\tau_k} F(\tau) d\tau \quad (1)$$

$$E_H = \int_{\tau_0}^{\tau_k} t(\tau) d\tau \quad (2)$$

The amount of heat was calculated for each value obtained by the formula (3):

$$Q = C \times V \times \Delta t \quad (3)$$

Where C is the specific heat of water (taken as 4 200 kJ/(kg*K); V - fluid flow rate, m³/sec; Δt - temperature difference, °C.

RESULTS AND DISCUSSION

A series of experiments with suspensions with different concentrations of dry matter was conducted. The obtained results make it possible to construct curves of changes in power, heat and particle size during the grinding process.

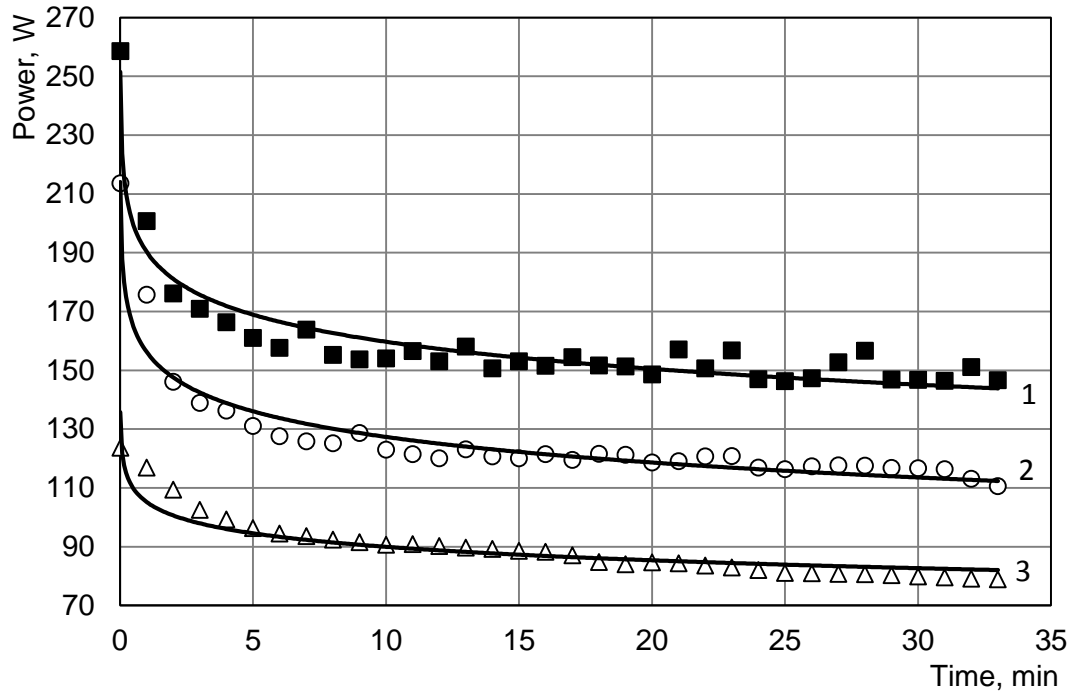


Fig.3. Power change in time, W: 1 – for a suspension concentration of 60/40; 2 - for a suspension concentration of 80/20; 3 - for castor oil.

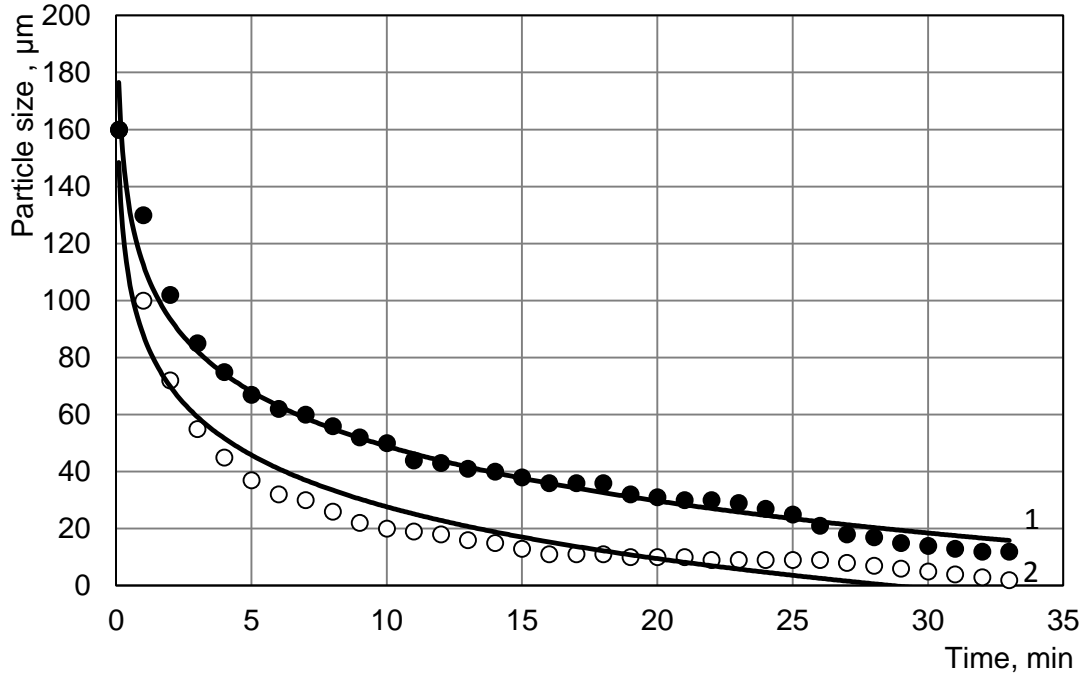


Fig.4. Effect of grinding time on the particle size of a suspension of iron oxide red 120, mkm: 1 – for a suspension concentration of 80/20; 2 - for a suspension concentration of 60/40.

A series of experiments with the concentration of dry matter shows that the most energy-intensive process increases when grinding to destroy the concentration of dry matter in suspension (60/40) due to greater resistance of the working member in the system "bead product", which has a higher density and viscosity, the presence of other grinding products.

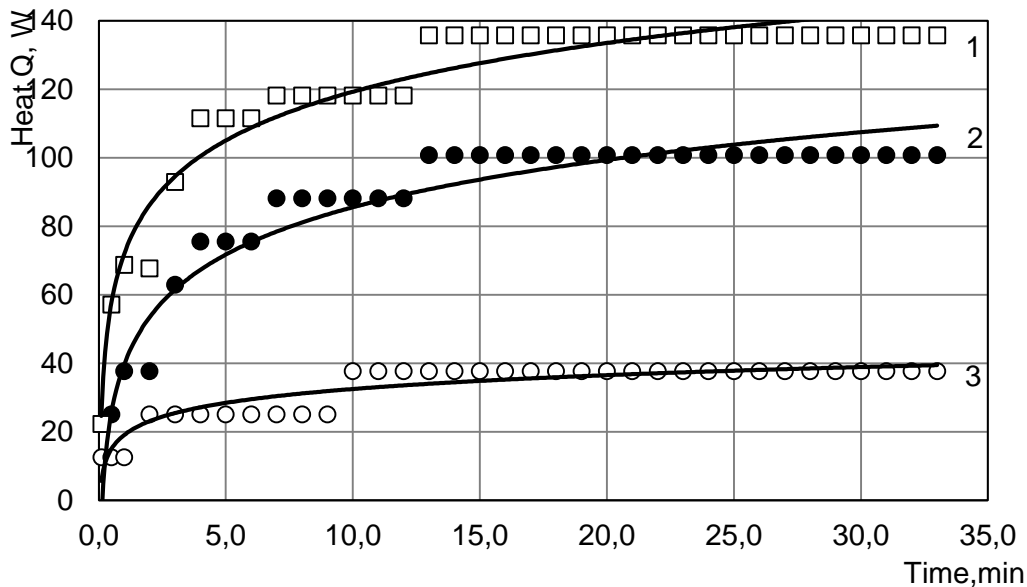


Fig.5. Dependence of thermal energy on time, joule: 1 – for a suspension concentration of 60/40; 2 - for a suspension concentration of 80/20; 3 - for castor oil.

The obtained graph of power versus time (Fig. 3) shows a rapid decline in energy spent on the process of ultrafine grinding in a bead mill. This is due to the high energy consumption for mixing the system "bead-product", the state of which depends on the rheological properties of the suspension. In addition, the level of energy consumed depends on overcoming the set of harmful resistances associated with the resistances of the product, system and moments of inertia (Kanda, Y., Kotake, N., 2007).

The graph of particle size versus time (Fig. 4) shows the most intense grinding in the first 10 minutes, especially for a suspension concentration of 60/40. Then the process slows down, but in the end we get the finished product with a particle size of 2-3 μm, which is optimal. This is due to the fact that at high concentrations of dry matter increases the so-called "packing density" of the pigment particles (Kanda, Y., Kotake, N., 2007), respectively increases the density of the suspension and its viscosity, respectively, the intensity of interaction of working bodies with particles of dry suspension will be higher. This interaction results in a finely divided product, but the side effect is high energy consumption and a large release of excess energy (energy not involved in the process) in the form of heat (Mende, S., Rappl, M., 2014; Weber, U., Langlois, D., 2010).

The graph of the dependence of thermal energy on time (Fig. 5) shows a rapid increase in energy, especially for a suspension with a concentration of 60/40. This is due to the fact that at the beginning of the process is the heating of the structural components of the product and the interacting parts of the mill by overcoming a set of harmful resistances. Moreover, the higher the concentration of dry matter in the suspension, the higher its density and viscosity (Weber, U., Langlois, D., 2010).

The obtained functions of the dependences of the total specific energy and thermal energy on time were substituted into integrated expressions (formula (1) and formula (2)) calculated and compared.

Tabl. 1. Comparative analysis of energy parameters of different products

Title	The resulting formula (specific energy)	The resulting formula (thermal energy)	Total specific energy, J.	Thermal energy, J.
Castor oil	$F = -6,623\ln(\tau)+185,34$	$t = 5,833\ln(\tau) + 19,084$	$5,571 \times 10^3$	$1,119 \times 10^3$
Suspension (80/20)	$F = -12,560\ln(\tau)+236,38$	$t = 19,939\ln(\tau) + 39,653$	$5,189 \times 10^3$	$2,951 \times 10^3$
Suspension (60/40)	$F = -13,450\ln(\tau) + 190,83$	$t = 20,544\ln(\tau) + 71,976$	$6,766 \times 10^3$	$4,061 \times 10^3$

Calculations (Table 1) of thermal energy and total specific energy show that the process of ultrafine grinding is a very high-energy process. Directly on the process of grinding the solid phase in suspension, energy is spent less than 1%. These costs are within the error of the devices and it is difficult to take into account separately. It is worth focusing on the energy that goes into driving the beads by the disk working body and dissipating the energy in the form of heat. It should be added that the bead mill has a rather low efficiency due to the fact that most of the energy goes to mixing the beads, and grinding takes place in the zones of the highest speed.

CONCLUSION

The process of ultra-fine grinding is a very high-energy process. Less than 1% of the total energy consumed in the whole process is consumed directly on the process of grinding the solid phase in the suspension. These costs are within the error of the devices and it is difficult to take into account separately.

Most of the energy is spent on mixing and overcoming the working body's resistance to the "bead-product" system and work on overcoming the set of harmful resistances associated with the resistances of the product, system and moments of inertia.

Ultrafine grinding in bead mills revealed the following pattern: the higher the concentration of dry matter in the slurry, the higher the energy consumption for the process, the higher the heat dissipation, the higher the density and viscosity of the slurry, the faster the required particle size in the product.

It is very important to take into account the properties of the suspension, the geometric parameters of the working chamber and the working body, the features of the working bodies and the operating parameters of the bead mill during modeling and process.

REFERENCES

- Hrininh, K., Gubenia, O. (2020). Energy parameters of ultrafine grinding of pharmaceutical and cosmetic ingredients in the bead mill. *Proceedings of University of Ruse*, 59(10.3), 40-44.
- Hrininh, K., Gubenia, O., Litovchenko, I. (2019). Review of the working bodies of vertical bead mills, *Proceedings of University of Ruse*, vol 58, book 10.2, 12-16.
- Kanda, Y., Kotake, N. (2007). *Handbook of Powder Technology*. Elsevier.
- Loh, Z.H., Samanta, A.K., Heng, P.W.S. (2015). Overview of milling techniques for improving the solubility of poorly water-soluble drugs. *Asian Journal of Pharmaceutical Sciences*, 10, 255-274.
- Mende, S., Rappl, M. (2014). Mill performance matched to the task. Throughput enhanced by optimising cooling and disc configuration // *European Coatings Journal*, 12, 88-91.
- Mende, S., Stenger, F., Peukert, W., Schweders, J. (2003), Mechanical production and stabilization of submicron particles in stirred media mills // *Powder Technology*, №132, 64–73.
- Schmidt, J., Romeis, S., Konnerth, C., Damm, C., Peukert, W. (2015). Formation of Nanoemulsions by Stirred Media Milling. *Procedia Engineering*, 105, 557-564.
- Weber, U., Langlois, D. (2010). The effect of grinding media performance on milling and operational behaviour. *The Journal of The Southern African Institute of Mining and Metallurgy*, 110, 147-152.