ISSN 1311-3321 (print)
ISSN 2535-1028 (CD-ROM)
ISSN 2603-4123 (on-line)

UNIVERSITY OF RUSE “Angel Kanchev”
РУСЕНСКИ УНИВЕРСИТЕТ “Ангел Кънчев”

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PROCEEDINGS

Volume 59, book 10.3.
Chemical technologies
&
Biotechnologies and food technologies

НАУЧНИ ТРУДОВЕ

Том 59, серия 10.3.
Химични технологии
&
Биотехнологии и хранителни технологии

Ruse
Русе
2020

ERI-SSS-CT(R)-01

ENERGY PARAMETERS OF ULTRAFINE GRINDING OF PHARMACEUTICAL AND COSMETIC INGREDIENTS IN THE BEAD MILL[8]

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Abstract: *The energy parameters of the grinding process of the components of pharmaceutical and cosmetic preparations in a bead mill are considered. The purpose of research - to determine the energy parameters of ultrafine grinding of pharmaceutical and cosmetic ingredients in the bead mill. A series of experimenters was conducted where the degree of grinding was measured using USB Digital Microscope and software, the temperature was measured with a TPM-10 electronic thermometer, and the power was measured with a CNFAJ Intelligent Power Meter three-phase wattmeter. As a result of the study, we can conclude that most of the energy is spent on the work of mixing the bead-product system and the work spent on heating the structural components of the product and the interacting parts of the mill, which, in turn, depend on the rheological properties of the suspension.*

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

INTRODUCTION

Fine and ultrafine grinding is necessary for materials that have an unsatisfactory dispersed and uniform composition and must provide the desired therapeutic or colorimetric effect in the pharmaceutical and cosmetic fields. Note that the finished product of the bead mill is a suspension. (Drögemeier, R., Leschonski, K. 1994).

The process of fine grinding in bead mills is influenced by many factors: mechanical energy, specific energy consumption, energy of collisions of grinding bodies, the number of effective collisions of grinding bodies with the product, as well as the residence time of particles in the mill (Mende, S., Rappl, M., 2014).

EXPOSITION

Research methodology

Materials

The following materials were taken for the study: pharmaceutical castor oil, red pigment 120 iron oxide.

Pharmaceutical castor oil is a natural product, vegetable oil from the fruits of castor oil bean. Transparent, thick and viscous, colorless or slightly yellowish liquid. It is freely soluble in ethanol (95%). It is practically insoluble in water and mineral oils. Stable substance. Dynamic viscosity at 20 ° C - 1000 MPa×s, at 40 °C - 200 MPa×s. Boiling point - 31 °C, melting point - 12 °C, freezing point -16 °C, relative density at 25 °C - 955-968 kg/m^3. In pharmacy, it is most often used in creams and ointments at a concentration of 5-12.4 % as a constituent substance and solvent for dermatological ointments, alcohol liquids, liniments, frostbite ointments) and oil injections, as well as a plasticizer in the production of tablets and capsules. It is part of many cosmetics because the

[8] Докладът е представен на студентската научна сесия на Русенски университет филиал - Разград на 28.05.2020г. в секция Химични технологии с оригиналното си заглавие на английски език.

suspension sedimentation process is significantly slowed down. In addition, the finished product has a reduced fusibility without the use of additional stabilizing impurities.

The pigment red 120 iron oxide is a dispersed system of iron oxide (III) of hexagonal structure (hematite), a powdery material of red-brown or dark red color. The chemical formula is Fe_2O_3. Humidity less than 1%. The pH of the aqueous extract is 3.5-7. Bulk density 1.0-1.1 g/cm^3. Density 5.0-5.1 g/cm^3. The shape of the particles is spherical. The predominant particle size is 0.11 mkm (according to the manufacturer's specification).

Methods

The following settings were used:

Laboratory bead mill. The working chamber is equipped with a shirt and a sampler with a sieve cartridge on the lid of the glass. The working body consists of a shaft on which 4 guide discs with 4 symmetrically located holes 10 mm in diameter are fixed. Working bodies are glass beads with a diameter of 2 mm. Grinding occurs wet (in the presence of a solvent) due to the interaction of the beads with each other, with the walls of the working chamber and the disks on the shaft and the entire contact area of the beads-product system. The three-phase engine rotates the working body with a frequency of 1350 rpm. Such a cooperation to produce, until we accept a finely tuned product, a great effect is that there is a great energy supply and a great wikid of excess energy in the view of heat (Mende S., Stenger F., Peukert W., Schweders J., 2003; Hrininh K., Hordeichuk R., Gubenia O., 2018).

Electronic thermometer TPM-10. Temperature measurement range from -50 °C to +100 °C. Measurement resolution 0.1 °C. Measurement error max 2 %. There are 2 sensors for input and output of water from the jacket of the working chamber.

Three-phase wattmeter CNFAJ Intelligent Power Meter. Able to measure electrical parameters (three-phase voltage, three-phase current, active power, reactive power, visible power, power factor, frequency, etc.). Accuracy class 0.5. Frequency 40-60 Hz, accuracy 0.1 Hz.

Particle size was determined using a USB Digital Microscope and software.

The bead mill worked with water cooling in a circulating way, the readings of the devises were taken every 30 seconds for 33 minutes. The mass of the product was 200 grams, the mass of the working bodies was 450 grams, and the water consumption was 0.03 kg/s (Hrininh K., Hordeichuk R., Gubenia O., 2018).

The installation scheme is presented in Fig.1 (Mende, S., Rappl, M., 2014; Rowe, W.B., 2014).

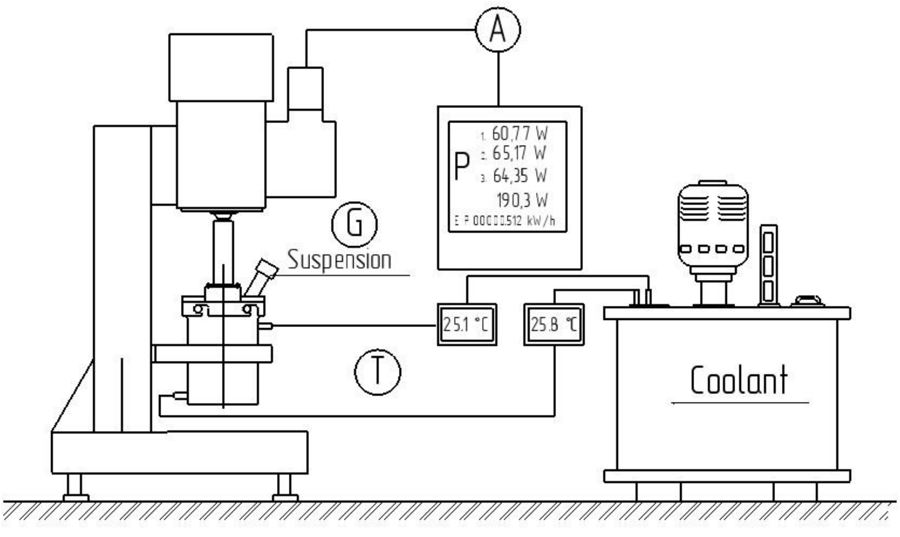

Fig. 1. Experimental installation: A - power measurement by wattmeter; T – temperature measurement by thermometer; G – particle size was recognized by computer software.

The total specific energy for the grinding process was calculated as follows: the obtained process equation is a function of power versus time, and is substituted into an integral expression.

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

Heat generation was calculated in a similar way, obtaining the function of the dependence of thermal energy on time.

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

RESULTS AND DISCUSSION

The results of the study are presented in the graphs below.

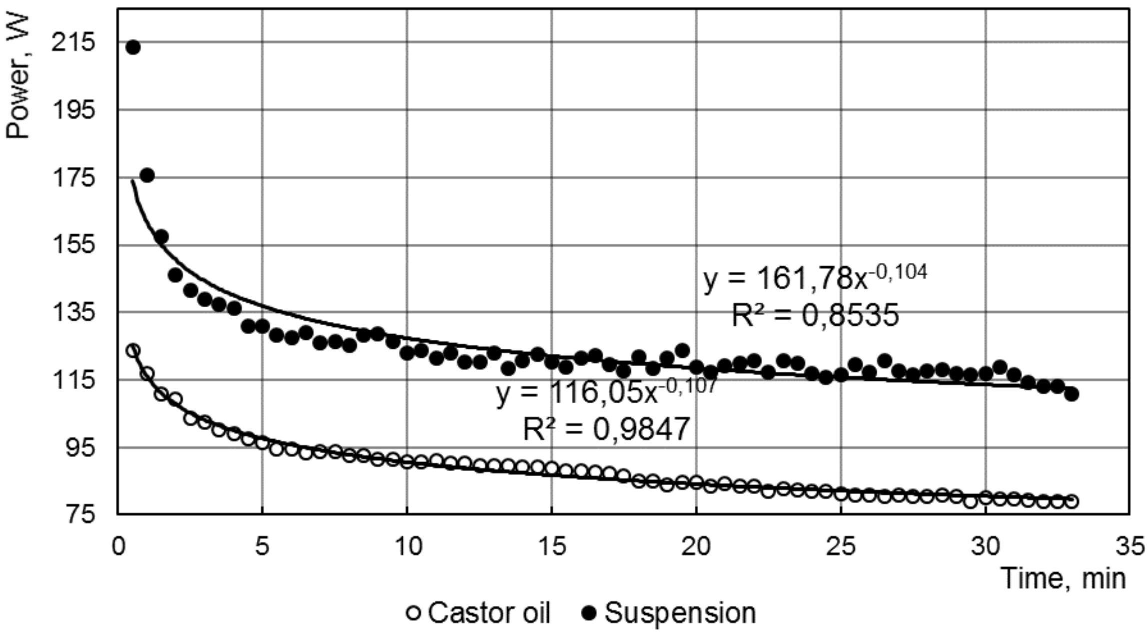

Fig. 2. Power change in time, W.

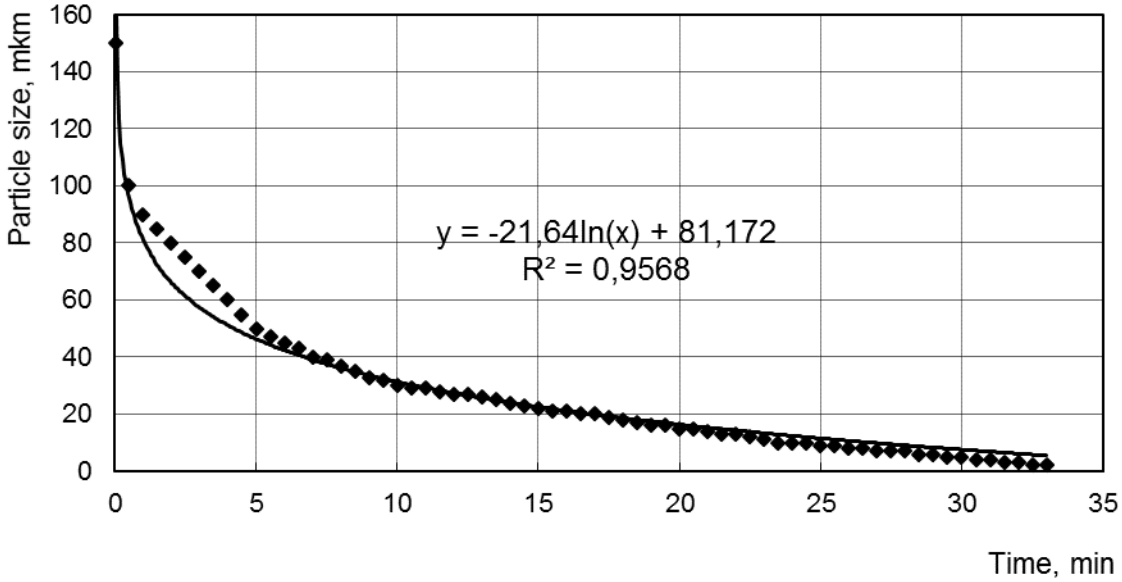

Fig. 3. Effect of grinding time on the particle size of a suspension of iron oxide red 120, mkm

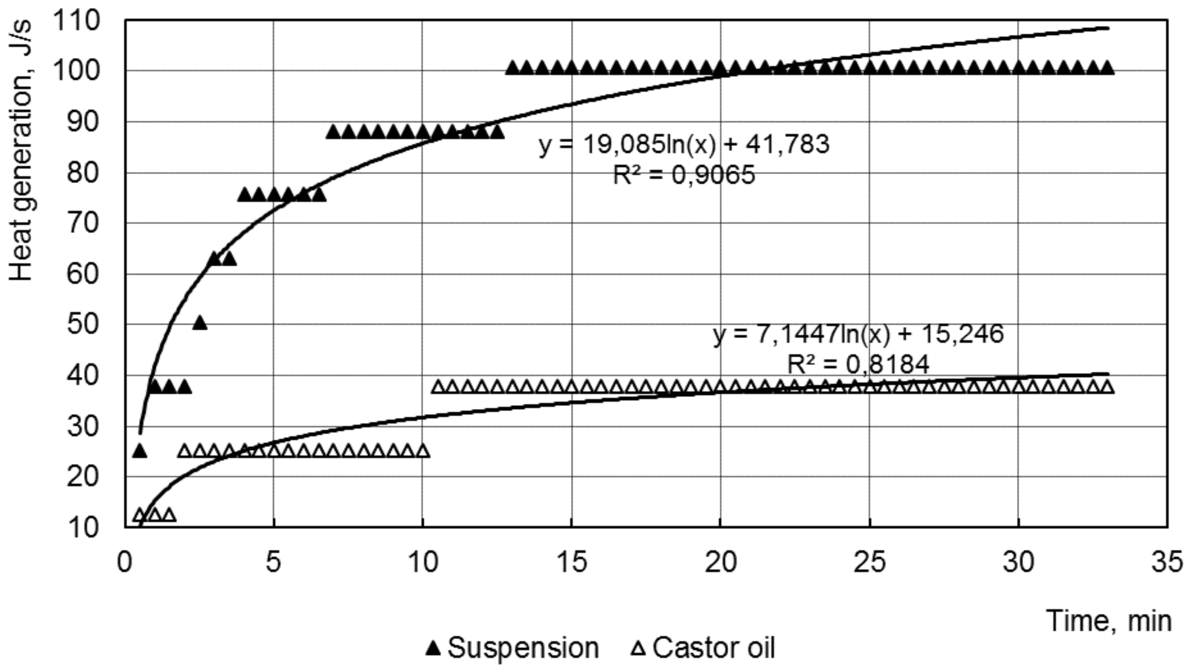

Fig.4. Dependence of heat generation on time,J/s.

Graphs of power versus time are obtained. We see a rapid drop in power in the first 6 minutes. This is due to the high energy consumption for mixing the "bead-product" system and the uniform distribution of the particle size distribution of the suspension (Kanda, Y., Kotake, N., 2007).

However, the heat schedule begins with a rapid increase in the first 10 minutes. This is due to the large increase in excess energy in the system, which is not involved in the grinding process of the dry fraction of the product.

The graph of the particle size distribution over time shows the most intense grinding in the first 10 minutes, where the particles of the dry fraction will decrease from more than 150 µm to 30 µm. 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 (Mende S., Stenger F., Peukert W., Schweders J., 2003).

Based on the equation of energy balance (Lisovenko A., 1982), the approximate energy balance can be represented as:

$$A = A_1 + A_2 + A_3 + A_4+A_5, \quad 3)$$

where A_1 - work spent on mixing the system "bead-product";

A_2 - work spent on moving the working body;

A_3 - work spent on heating of structural components of a product and the parts of a mill interacting with them, J/s;

A_4 - work spent directly on grinding, taking into account the energy expended on the absorption and wetting of dry components by the dispersed medium;

A_5 - work spent on the absorption and wetting of dry components by the dispersed medium (dispersing medium penetrates into the cracks of the solid particles of the suspension, preventing the closure of cracks and removing the formed particles from each other layer of molecules adsorbed on their surface) (Lisovenko A., 1982).

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 with a rotor with grinding discs and dissipating energy in the form of heat (A_1 + A_3). It should be added that the work A_1, which is spent on mixing the "bead-product" system, very much depends on the rheological properties of the suspension, which must be taken into account when modeling and conducting the process from one type of product to another (Lisovenko A., 1982).

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 (Mende, S., Rappl, M., 2014).

A series of experiments with different solvents and concentrations have been performed, resulting in the most energy-intensive process when grinding viscous products with a high dry matter content. We can say that the process very much depends on the initial properties of the suspension.

Table 1. Comparative analysis of energy parameters of different products

Title	Total specific energy, joule	Heat generation, joule
Water	2,254×10^5	1, 940×10^5
Castor oil	4,977×10^5	2, 160×10^5
Suspension castor oil 60% and pigment 40%	6,731×$\mathbf{10^5}$	2, 594×$\mathbf{10^5}$
Suspension castor oil 80% and pigment 20%	5,348×10^5	3, 826×10^5

CONCLUSION

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. Most of the energy is spent on the work of mixing the "bead-product" system and the work spent on heating the structural components of the product and the interacting parts of the mill, which, in turn, depend on the rheological properties of the suspension. This must be taken into account when modeling and conducting the process from one type of product to another. The smaller the particle size and higher viscosity of the suspension, which must be milled, the greater the energy required for the process, and the more heat will be released.

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