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FEATURES OF A ULTRA-FINE GRINDING BY WET METHOD IN BEAD MILLS

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***Abstract**: It was conducted an analytical review of the features of ultra-fine grinding in bead mills by wet method. The aim of the study was to reveal unexplored features of ultra-fine grinding processes by wet metod in bead mills with their subsequent study. As a result of the analysis, a relationship was found between mechanical energy and the degree of dispersion by grinding the suspension, the intensity of grinding over time, the temperature rise of the suspension, depending on the degree of dispersion. As a result of the analysis, no relatioship was found between the temperature rise of the grinding material and the power, the density of the dispersing suspension and its viscosity. In further studies, special attention should be paid to the relationship between the density of each of the dry bulk components and their wet mixes with energy that is consumed for grinding, the relationship of the degree of grinding to the time that is spent on achieving a given parameter, and what part of the mechanical energy is converted into heat in depending on the grinding time.*

***Key words**: Ultra-Fine Grinding, Relationship, Energy, Time, Temperature, Bead Mills.*

INTRODUCTION

When the technological process of the produce of the soft dosage forms is implemented, it is necessary to distribute and grind fine dispersed solids in a liquid medium, because during storage and transport of the starting material, the particles stick together forming the conglomerates (Rowe W.B., 2014). Therefore, the obligatory stage in the production of ointments, liniments, gels and pastes is dispersing. It is important that the process of fine grinding is divided in real grinding and dispersing (Drögemeier R., Leschonski K., 1994). This task perfectly solve the bead mills, which are used for grinding a wide range of different products, including pharmaceutical and cosmetic industries.

EXPOSITION

1. Comparative analysis of existing equipment for superfine grinding

For today, there are such type of machines for superfine grinding: bead mills, three-roller mills, colloid mills, jet mills, vibrating mills, planetary mills.

Bead mill. The bead mill consists of two parts: a grinding chamber, in which the beads are loaded together with the components of the mixture, and the rotor, which rotates the entire system. The grinding process proceeds between the individual bead particles which are in contact with each other, as well as between the beads and the rotor or the walls of the container. The size of the crushed product can reach less than 1 microns. In the cosmetic industry they are used for grinding of the pigmentary pastes, creams. In the pharmaceutical industry - for the production of the ointments, pastes, gels, liniments (Salenko YU., 2008; Ohnishi O., Suzuki H., Uhlmann E., Schröer N., 2015).

Table 1. Advantages and disadvantages of a bead mill
(Salenko YU., 2008; Ohnishi O., 2015; Nakach, M., Authelin, J., Agut, C. 2017)

Advantages	Disadvantages
Easy maintenance; Reliability and safety of work; Versatility; High performance; Invariably quality of grinding; Simplicity of construction; Possibility of embedding into an industrial continuous production line; Effective in the process of dispersing, homogenizing and grinding simultaneously.	Great power consumption; A small coefficient of useful action – 15%; Not economical consumption of water and detergent to prepare the equipment for work; Burning and "burning out" of the suspension in case of insufficient cooling.

Three-roller mills. Rollers that are rotated toward at different speeds ensure the product moves from a roller to a roller and increases friction between them. Rollers are made of porcelain, basalt or metal (EXAKT Technologies, Inc. Startseite). To maintain the optimum temperature of the product rolls are make hollow for serving the coolant inside. Degree of material grinding is to 3 µm (index taken from industrial working equipment). In the cosmetic industry it is used for grinding of the pigmentary pastes, creams. In the pharmaceutical industry - for the production of the ointments, liniments, gels, pastes (Salenko YU., 2008; EXAKT Technologies, Inc. Startseite; Grining K., Tarasenko M., 2017).

Table 2. Advantages and disadvantages of the three-roller mills
(Salenko YU., 2008)

Advantages	Disadvantages
Magnet-based safety devices; Possibility of embedding into an industrial continuous production line; Suitable for the processing of very common materials that are prone to sticking or containing sticky inclusions; Compactness.	Not intended for grinding dry ingredients; Intensive and uneven wear of working surfaces of rollers and a knife; Relatively low specific productivity; The probability of bias shaft due to uneven tightening springs; When the maximum fineness is reached, the productivity decreases.

Vibrating mills. The grinding is carried out using a special vibrating device, which is on the same axis with the electric motor, the ball drum vibrates. The balls are made from a porcelain, hard alloys or steel. The drum is full with balls for 80-90%. The size of the crushed product is 1-5 microns. They are used for fine-grained crushing of materials of low strength (Kanda Y. Kotake N., 2007; Ohnishi O., Suzuki H., Uhlmann E., Schröer N., 2015).

Table 3. Advantages and disadvantages of the vibrating mills (Kanda Y. Kotake N., 2007; Ohnishi O., Suzuki H., Uhlmann E., Schröer N., 2015; Wills, B.A. 2005)

Advantages	Disadvantages
High dispersion and homogeneity of the particle size of the product are achieved very quickly.	Not suitable for grinding powders sticky and viscous pastes and thermolabile materials; Low productivity (up to 1 t/h); Rapid wear of grinding bodies; Hard working conditions.

Colloidal mills. A lot of designs of the mills are offered. For preventing the adhesion of particles, the milling is done in the presence of a dispersing medium. The size of the crushed product is less than 0.4 microns. In the pharmaceutical industry they are used for the produce of liniments, ointments, pastes. In the chemical industry - for the crushing of some mineral pigment (Rowe W.B., 2014; Grining K., Tarasenko M., 2017).

Table 4. Advantages and disadvantages of the colloidal mills (Salenko YU., 2008)

Advantages	Disadvantages
Effective emulsification and dispersion process.	High wear of working elements; Little knowledge of the process.

Jet mills. A energy source (a compressed gas or a steam) at expansion in the nozzles acquires a high speed, which sometimes reaches several hundred meters per second. The material particles are crushed as a result of a collision between themselves when streams jets intersect, as well as strokes and abrasion on the wall of the chamber (Kanda Y. Kotake N., 2007). The size of the crushed product is 2-5 microns. In the pharmaceutical industry are used to obtain superfine powders from expensive components (Rowe W.B., 2014; Salenko YU., 2008).

Table 5. Advantages and disadvantages of the jet mills (Kanda Y. Kotake N., 2007; Rowe W.B., 2014)

Advantages	Disadvantages
The grinding elements of the mill practically do not wear out, and therefore don`t add impurities to the finished product; Longevity of equipment; Provides high purity powders with a predominantly oval form of particles; The material in the process of grinding doesn't change its initial temperature, which allows processing of thermolabile; Minimal product loss.	High energy consumption resulting in high energy consumption of the process; Necessary to uniform supply of material and maintenance of constant aerodynamic mode of operation; Necessary provision of additional equipment (air separator); Danger of work.

Planetary mills. The principle of the work is to rotate 3-4 drum around to the central axis and around its own axis in the opposite direction of rotation of the rotor (central axis) of the mill. The size of the crushed product is less than 4-6 microns. Used mainly in laboratories for scientific research, as well as in industries where small amounts of fine powders are required (Rowe W.B., 2014; Grining K., Tarasenko M., 2017).

Table 6. Advantages and disadvantages of the planetary mills (Rowe W.B., 2014; Grining K., 2017)

Advantages	Disadvantages
High specific productivity.	High wear of working elements; Difficulty with continuous loading and unloading of material

Among the recommended mills, the most versatile, compact, energy saving, capacity, with the possibility of embedding to an industrial continuous production line, with high grinding rates (up to 1 μm), effective in the process of dispersion and homogenization, have a simple constructive solution, are bead mills. Despite its disadvantages, the bead mill is one of the most popular mill using in the cosmetic industry to grind pigment pastes and creams, and in the pharmaceutical industry - for the production of ointments, gels, pastes, liniments.

2. Analysis of the features of the grinding process in a bead mill

The bead mill is an equipment for thin and superfine grinding, which occurs by a wet method. The mill consists of a vertical (or horizontal) grinding chamber with a shirt. The chamber contains a rotor which consist of a shaft with disks or rods attached to it. The camera is filled with 70-80% beads from a basalt, glass or steel. When the rotor rotates, the solid particles of the material are crushed as a result of the friction on the body that is grinding, and each other. An industrial bead mill works in periodic, continuous or circulating modes. The mill has a sieve cartridge or a slit to separate the beads from the suspension. The size of the crushed product can reach less than 1 μm (Rowe W.B., 2014; Salenko YU., 2008; Grining K., Tarasenko M., 2017).

The process of fine grinding in the bead mills depend to many operating parameters. The studies have shown that important factors are: 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; Rowe W.B., 2014). In addition, at grinding the desorption of gas and liquid residues from the surface of the substance is shattered, as a result the surface area of the solid changes (Drögemeier R., Leschonski K., 1994).

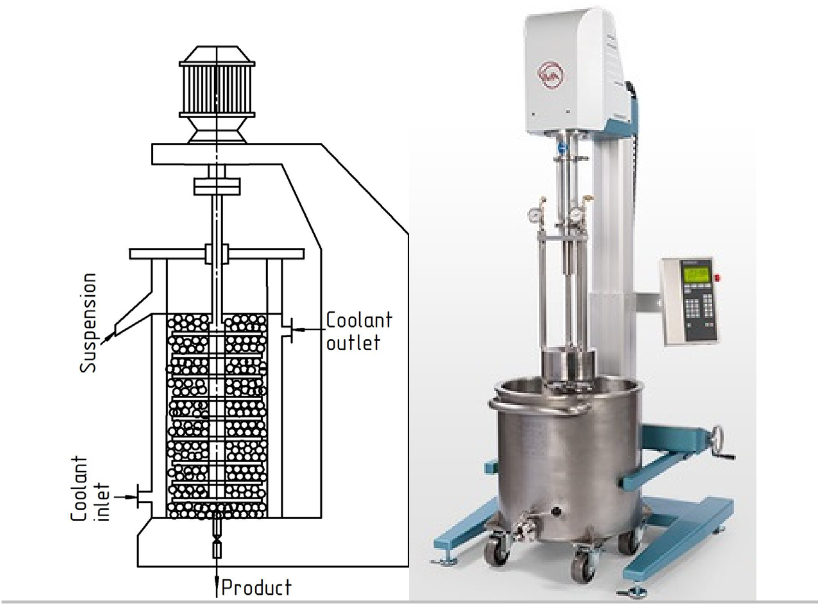

Fig. 1. Bead mill DISPERMAT®

Mechanical energy is the energy that is transmitted from the rotor through the beads to the suspension. The more energy is introduced into the system, the greater the likelihood of the destruction of strong agglomerates (Drögemeier R., Leschonski K., 1994). Energy can be introduced in several ways: with high rotor speed and low torque, or vice versa, at high torque and low rotational speed (Nakach, M., Authelin, J., Agut, C. 2017).

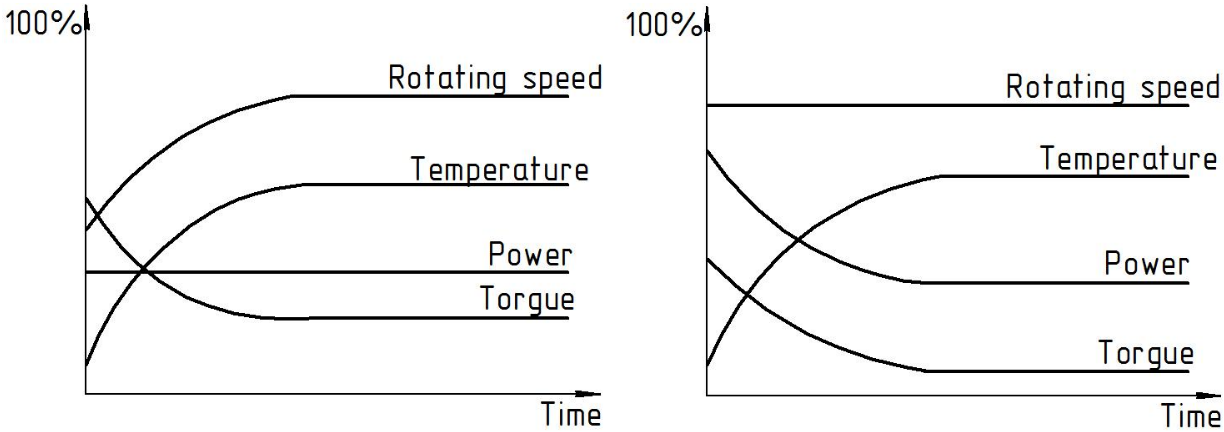

Fig. 2. Change of the parametres of grinding process depending at a constant power and rotate speed

Specific energy consumption is a value that is defined as the ratio of the mechanical energy transferred to the slurry and the volume or mass flow of the slurry for continuous or recirculation grinding processes (Cherevko O., Poperechniy A., 2014). For the periodic process, the specific energy consumption is determined based on the duration of the dispersion process and the transfer of mechanical energy to the system (Rowe W.B., 2014). For each product, the grinding process depends primarily on the consumed specific energy, as is proved by Steer and White (Mende S., Rappl M., 2014).

As the energy of collisions of grinding bodies is meant their kinetic energy, which is transmitted in the system from the grinding body to the substance, which is grinded. The kinetic energy is determined by the mass and velocity of grinding bodies (Cherevko O., Poperechniy A., 2014).

There is a direct relationship between the kinetic energy of grinding bodies, the number of their effective collisions and the specific energy consumption for a achieve of a result. The effectiveness of the contact is effective if the kinetic energy and energy from collision are more than energy of destruction or the binding energy so that the grinding process takes place. However, if the energy of colliding bodies is more than it needs, the excess is converted to heat, which leads to heating of the product and an increase of wear of the mill and its parts (Mende S., Rappl M., 2014; Nakach, M., Authelin, J., Agut, C. 2017).

The size of the grinding is determined solely by the specific consumption of energy: with any combination of technological parameters (capacity, residence time, degree of filling, grinding media), but with equal specific energy consumption, the particle size is the same (Mende S., Rappl M., 2014).

In the process of grinding, the properties of the suspension change: at a constant rotor speed its viscosity changes, and as a result, the power changes. As the viscosity decreases, the power decreases, and as the viscosity increases, the power increases. When the system is cooled intensively, the amount of power increases, and decreases at less intensive cooling (Nakach, M., Authelin, J., Agut, C. 2017). In addition, the greatest degree of grinding occurs at the beginning of the working time of the bead mill, at further grinding is not observed (Mende S., Rappl M., 2014).

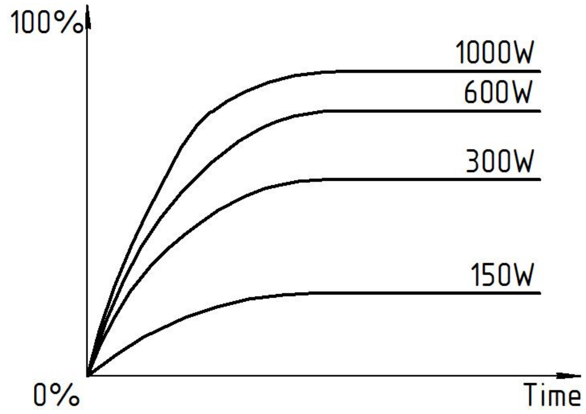

Fig. 3. Effect of the input power on the degree of dispersion

The torque depends on the configuration of the rotor, the viscosity of the suspension, the type, amount and size of the beads. A large number of beads increases the torque of the shaft and provides space for the dispersion process (Drögemeier R., Leschonski K., 1994).

Interdependencies between energy and time parameters allow to optimize and improve the dispersion process. It can increase the duration of the process until the desired result is achieved. The power increases with increasing speed, improving the dispersion result (Nakach, M., Authelin, J., Agut, C. 2017; Rowe W.B., 2014).

CONCLUSION

In the manufacture of soft dosage forms, there is a need for finer grinding and dispersing of loose components in a liquid medium. In the course of the literary review it was revealed that the bead mill satisfies the given requirements.

As a result of the analytical review of the scientific literature, it were studied the interactions between the specific energy consumed, the kinetic energy (collision energy) of grinding bodies, and the efficiency of the interconnection of the above energies; interaction between energy and time parameters; the relationship between the drive power, the rotational speed, the torque and the temperature of the entire system. In addition, the result of grinding is affected by the type, size and number of beads in the working container, as well as the shape and configuration of the guide discs (or rods). This gives a more complete picture of the process of ultrafine grinding in a bead mill, and makes it possible to increase the capacity of the production.

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