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# PROCEEDINGS

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## Chemical technologies
## &
## Biotechnologies and food technologies

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# INVESTIGATION THE PROCESS OF SUPERFINE GRINDING OF COMPONENTS OF PHARMACEUTICAL AND COSMETIC PRODUCTS ON THE BEAD MILL [7]

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*The process of grinding the components of pharmaceutical and cosmetic preparations in a bead mill is considered. The degree of grinding was measured with a "Klin" grindometer, the temperature was measured by a contactless pyrometer, the power was measured by current clamps. The smaller the particle size and the higher density of the suspension, the greater the energy required to conduct the process, and the more heat will be released. When grinding titanium dioxide over a period of 0 to 30 minutes, the power increases from 205 to 209 W, with the most intensive increase observed in the interval from 5 to 10 minutes at 2.33 W. In addition, the temperature of the suspension increases from 21.9 to 23.4 °C, the density of the suspension increases from 889 to 1176 kg/m$^3$, and the particle size decreases from more than 100 μm to 10 μm, the most intensive grinding in the first 5 minutes of the process. When the quinacridone red Red 122 is grinded, the power is increased from 205 to 210 W, the slurry temperature is from 22.4 to 24.3 °C, the density is from 870 to 952 kg/m$^3$, and the particle size decreases from more than 60 μm to 2 μm, while the most intensive grinding occurs in the first 5 minutes of the process.*

*Key words: grinding, beads, mill, pharmacy, cosmetics, suspension.*

## INTRODUCTION

In the modern production of pharmaceuticals and cosmetics, a high degree of grinding of the raw material for further use is of great importance. During the storage and transportation of raw materials, the particles get stick together, forming agglomerates. In addition, the powder particles have an irregular shape and a non-uniform particle size distribution. Such raw materials can`t give the product the necessary efficiency and quality. Therefore, the obligatory stage in the manufacture of cosmetics and pharmaceuticals is the grinding stage (Drögemeier, R., Leschonski, K. 1994).

## EXPOSITION

### Research Methodology

### Materials

The following materials were used for the research: vaseline oil perliquidum pharmaceutical, white pigment titanium dioxide (food additive E171) pharmaceutical, pigment quinacridone red Red 122.

[7] Докладът е представен на студентската научна сесия на филиал Разград, проведена на 15.05.2018г. в секция „Биотехнологии и хранителни технологии" с оригинално заглавие на английски език: Investigation the process of superfine grinding of components of pharmaceutical and cosmetic products on the bead mill.

**Vaseline oil perliquidum pharmaceutical** - mineral oil, a mixture of liquid hydrocarbons of petroleum distillate. The quality of vaseline oil is characterized by its color. It is determined by the Saybolt scale. The dark one is "- 16", the light one is 30 (Kanda, Y., Kotake, N., 2007).

Vaseline oil in pharmaceutics is used as a basis for creating multi-component ointments and creams, injectable suspensions, as a solvent for some medicines, and as a separate medicinal product. In the cosmetic industry, oil is used as a base for the preparation of a wide range of creams, but only if it has been multistage cleaned and completely cleared of foreign smell and impurities. Also, it is used in the production of decorative cosmetics (Kanda, Y., Kotake, N., 2007).

Appearance: colorless, clear, oily liquid. Virtually insoluble in water, slightly soluble in ethanol 96%, mixed with carbohydrates. Color (Saybolt) - +30. The kinematic viscosity at 40 °C is 11-15 cSt. Dynamic viscosity at 20 °C is 33-45 MPa·s. The relative density at 20 °C is 818-880 $kg/m^3$ (Kanda, Y., Kotake, N., 2007).

**Titanium dioxide (titanium white, E171)** is a white pigment. In the pharmaceutical industry, titanium dioxide is used for high chemical cleanliness to provide a high whitening and crusting effect. It is used for cosmetic products for giving white color and light tightness. In addition, it is used for the preparation of concentrated pigment pastes for various types of lipsticks, lip glosses, nail polishes, powders, tonal creams and other decorative products (Kanda, Y., Kotake, N., 2007).

Appearance: powder from white to slightly colored. The residue on the sieve with a mesh of 45 μm is 0.08%. Bulk density of 82 $kg/m^3$; Specific density 4 000 $kg/m^3$. Insoluble in water and organic solvents (Kanda, Y., Kotake, N., 2007).

**Quinacridone red Red 122**. Universal dry pigment, which has a stable purple hue among the violet-red pigments. It also has an increased stability of flocculation, good flowability. Used in the cosmetic industry for the preparation of concentrated pigment pastes for various types of lipsticks, lip glosses, nail polish (Kanda, Y., Kotake, N., 2007).

Appearance: powder of bright red color with a blue hue. Specific density is 1460 $kg/m^3$; bulk density 540 $kg/m^3$; The residue on the sieve with a mesh of 320 μm is 23,4%; the average particle size is 0,09 μm; specific surface area of 59 $m^2/g$. Resistant to acids, alkalis, water, organic solvents (Kanda, Y., Kotake, N., 2007).

## METHODS

**Expiremental instalations were used:**

**Bead laboratory mill**. Equipped with three capasity (glasses) with a shirt for water cooling and a sampler on the lid of a glass with a sieve cartridge. The rotor consists of a shaft on which 4 guide discs with 4 symmetrically located holes with a diameter of 1 cm are fixed. The working members are a glass bead that has a diameter of 2 mm. The motor operates at 380 V with a current of 1.0 A with a rotor speed of 1350 rpm (Salenko, YU., 2008; Rowe, W.B., 2014; Mende, S., Rappl, M., 2014).

**Pyrometer** is a non-contact digital thermometer. Infrared principle of action. The temperature range is -50 °C to + 330 °C (-58 °F to 626 °F). The accuracy is ±1.5% (Nakach, M., Authelin, J., Agut, C., 2017; Rowe, W.B., 2014).

**Grindometer "Klin"** is intended for the analysis of the particle size and agglomerates in determining the degree of grinding when testing pigmented suspension materials in different measuring ranges. The measuring range is from 0 to 100 μm. The scale division is 10 μm. The limit of the permissible absolute error is ±10 μm. Dimensions: measuring plate - 175×35×20; The scraper is 60×40×6 mm (Nakach, M., Authelin, J., Agut, C., 2017; Rowe, W.B., 2014).

**The current clamps** is a model of a multimeter with clamps, which opens up to a width of 5 cm. Clamps work on the principle of electromagnetic induction.

With it, you can measure AC and DC voltage, resistance, temperature, test diodes, ring connections, measure frequency. Equipped with a liquid crystal display with a resolution of 3½, which displays the results of all measurements (Nakach, M., Authelin, J., Agut, C., 2017; Rowe, W.B., 2014).

The scheme of the experimental setup is shown in Fig.1 (Mende, S., Rappl, M., 2014; Rowe, W.B., 2014).

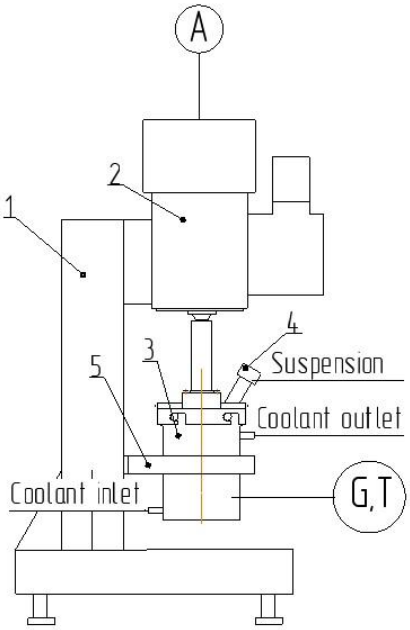


Fig.1. The scheme of the experimental setup: 1 – bedplate; 2 – motor; 3 – glass; 4 – sampler; 5 – clamp.

The preparation of the samples was as follows: in three clean glasses with a volume of 250 ml hung pigments and vaseline oil according to the following recipe (Nakach, M., Authelin, J., Agut, C., 2017; Rowe, W.B., 2014):

Composition A: vaseline oil - 250 g;

Composition B: titanium dioxide 50 g, vaseline oil 200 g;

Composition C: Red 122 - 30 g, vaseline oil - 200 g.

There were weighed on an analytical scale with a precision of 0.01 g. The compositions B and B were mixed on a agitator.

Composition A was tested for temperature only. Compositions B and C were tested for the temperature of the suspension by means of a pyrometer, the particle size with a "Klin" grindometer, the density before and after the test was determined by means of formula:

$$\rho = \frac{m}{V}, \mathrm{kg/m^3} \tag{1.1}$$

After testing, the compositions took turns taking part in the experiment (Nakach, M., Authelin, J., Agut, C., 2017; Rowe, W.B., 2014).

The bead mill operated in a batch mode with cooling, after each experiment the installation was washed. The samples were analyzed and the instruments readings were taken every 5 minutes for 30 minutes. The obtained samples were analyzed for the degree of grinding by a grindometer, the pyrometer measured the temperature of the suspension, the amperage at three phases was measured with a current clamps (Salenko, YU., 2008; Drögemeier, R., Leschonski, K. 1994).

## RESULTS AND DISCUSSION

The results of the research are shown in the graphs below. The graphs show that the greatest power is consumed for grinding the suspension of titanium dioxide (Fig. 1), which may be due to the fact that the suspension has the highest density after grinding - 1,176 kg/m$^3$ (Fig. 5), as well as the largest size of agglomerates at the beginning of the experiment (more than 100 μm). In addition, less heat is produced by grinding a suspension of titanium dioxide than the grinding of a suspension of quinacridone red Red 122 (Figure 4), although Red 122 has a greater bulk density (540 kg/m$^3$) than titanium dioxide (82 kg/m$^3$) , which can also be related to the density of the suspension (Hrininh, K., Gubenia, O., Dimitrov TS., 2018).

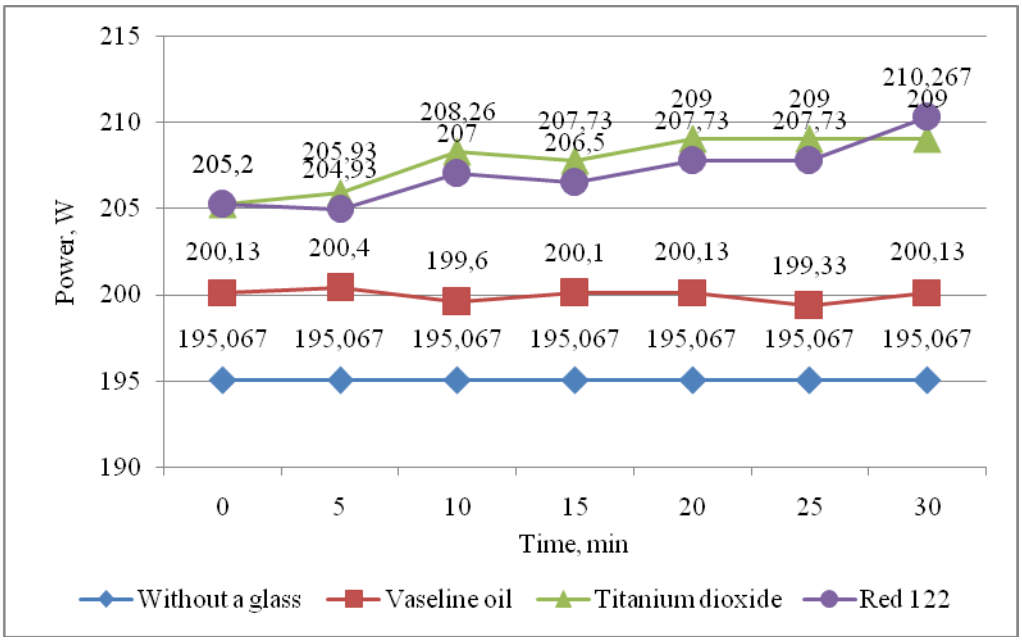


Fig. 2. Change in power, W.

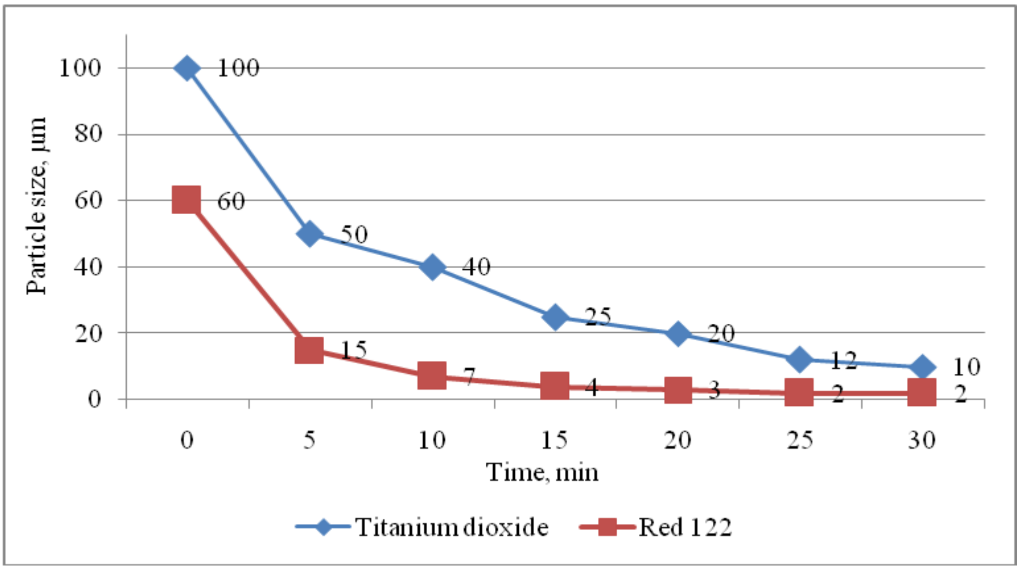


Fig.3. Change in particle size, µm.

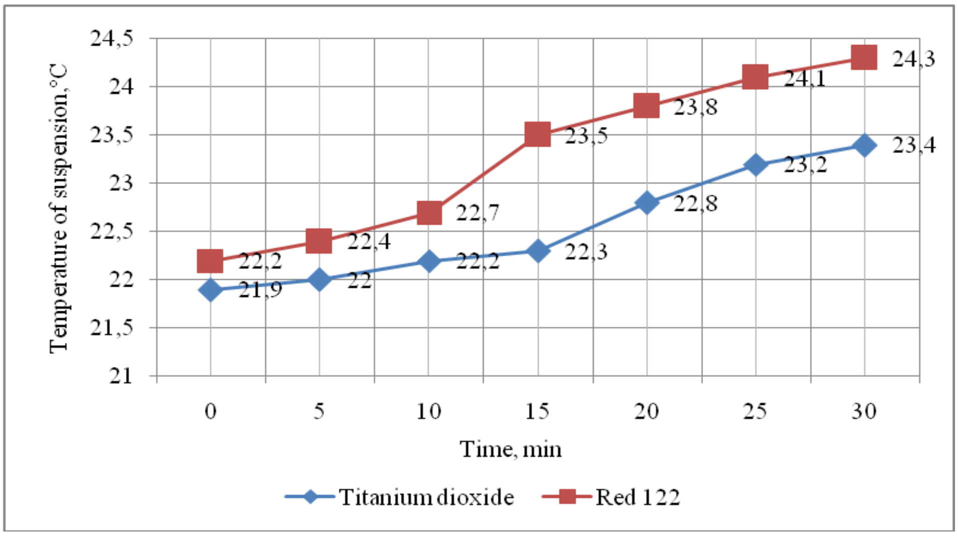


Fig.4. Change in temperature of the suspension, °C.

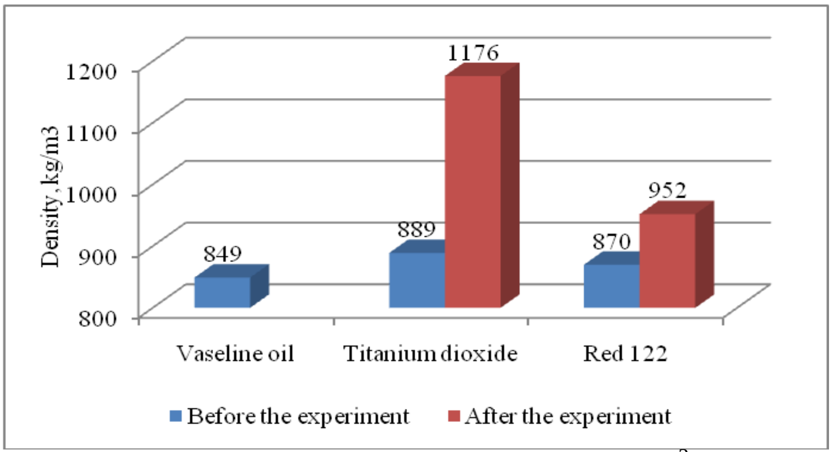


Fig.5. Change in suspension density, kg/m$^3$.

**CONCLUSION**

When grinding titanium dioxide over a period of 0 to 30 minutes, the power increases from 205 to 209 W, with the most intensive increase observed in the interval from 5 to 10 minutes at 2.33 W. In addition, the temperature of the suspension increases from 21.9 to 23.4 °C, the density of the suspension increases from 889 to 1176 kg/m$^3$, and the particle size decreases from more than 100 µm to 10 µm,the most intensive grinding in the first 5 minutes of the process from 100µm to 50µm.

When the quinacridone red Red 122 is grinding for 30 minutes, the power increases from 205 to 210 W, the suspension temperature increases from 22.4 to 24.3 °C, the density of the suspension increases from 870 to 952 kg/m$^3$, and the particle size decreases from more than 60 µm to 2 µm, with the most intensive grinding occurring in the first 5 minutes of the process - from 60 µm to 15 µm.

When the pure pharmaceutical vaseline oil is dispersed, the power remains practically unchanged (199-200 W).

In results, the smaller the particle size and the higher density of the suspension that needs to be ground, the more energy is needed to conduct the process and the more heat will be released.

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