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FORMATION OF SUSPENSION STRUCTURE IN THE PROCESS OF GRINDING IN BEAD MILLS

Kateryna Hrininh, PhD student
Department of Machines and apparatus of food and pharmaceutical productions
National University of Food Technology, Ukraine
E-mail: neackriss@gmail.com

Assoc. Prof. Oleksii Gubena, PhD
Department of Machines and apparatus of food and pharmaceutical productions
National University of Food Technology, Ukraine
E-mail: gubena@meta.ua

Assoc. Prof. Yuliya Telychkun, PhD
Department Machines and apparatus of food and pharmaceutical productions,
National University of Food Technology, Kyiv, Ukraine
E-mail: tvill@meta.ua

Abstract: *It has been investigated the rheological properties of the suspension of farmaceutical and cosmetics preparates during grinding at an experimental laboratory bead mill of periodic action. The rheological characteristics have been investigated on a rotary viscometer Reotest 2. as a result of the research, the flow curve of the suspension during grinding has been constructed and it has been found that within the investigated range the product does not change the character of flow regardless of measurement time and temperature. Analysis of the curve of the suspension during grinding revealed features which explain formation of the structure. The dependence of the effective viscosity on the shear rate during grinding of the suspension is polynomial and confirms the non Newtonian character of the product and is defined as a linear plastic body. The investigated finished product has 6.5 time's higher viscosity in relation to pure castor oil, which is explained by the high dry matter content (40 %) and the increase in the newly formed area. The viscosity of the suspension varies depending on the temperature: cooled to 20 °C, the finished product has a viscosity of 22.5% higher than immediately after the grinding process with a temperature of 34 °C.*

Keywords: *grinding, suspension, shear stress, shear rate, effective viscosity.*

INTRODUCTION

Liquid and soft dosage forms and cosmetics widely use various suspensions of high concentrations based on vegetable fats. The use of the latter requires the provision of such indicators as stability, ease of use and dosage and rheological properties of the dispersion, which meet the requirements of their specific application, namely: to have long-term resistance to delamination and aggregation of particles (Ahmed J., Ptaszek P., Basu S., 2016; Hrininh K., Hordeichuk R., Gubenia O., 2018).

The scientific literature is not enough data to change the rheological properties of suspensions based on vegetable fats during their treatment in the bead mill. Note that the values of rheological parameters of suspensions are required for modeling and calculation of processes occurring in the bead mill and next processes of manufacturing pharmaceutical and cosmetic products (Mende S., Stenger F., Peukert W., Schweders J., 2003).

The purpose of the research is to establish the influence of the regime parameters of processing suspensions based on vegetable fats in a bead mill on their rheological properties.

EXROSITION

Materials and methods. Research of ultrafine grinding of suspensions were performed in a laboratory bead mill of periodic action (Fig. 1) (Hrininh K., Hordeichuk R., Gubenia O., 2018). 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. 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).

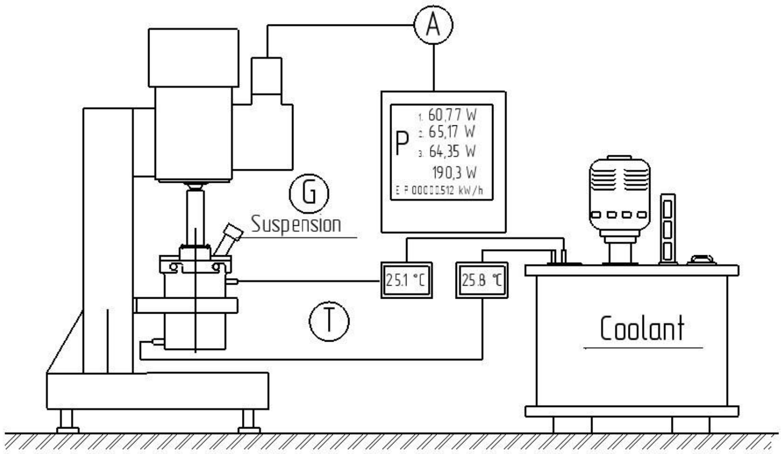

Fig.1 Experimental installation:
A - power measurement by wattmeter; T - temperature measurement by thermometer;
G – particle size was recognized by computer software.

The suspension was prepared according to the recipe: iron oxide red pigment 120 - 40% and castor oil - 60%. Grinding time - 45 minutes (2700 s). During grinding from the chamber of the bead mill every 5 minutes (300 s) samples were taken to measure the shear stress at different values of the shear rate.

Research of rheological characteristics were performed on a rotary viscometer Reotest 2 (Fig. 2) (Rachok V., Hudzenko V., Telechkun YU., Telechkun V., 2018). The main elements of the viscometer are the drive and the measuring mechanism with the cylindrical measuring device. The viscometer is driven through a 12-speed reversible gearbox from a synchronous motor with pole switching and a choice of 24 different speeds of rotation of the cylinder shaft or shear rate.

Research of the material at a certain temperature is achieved by directly maintaining the temperature by supplying liquid to the circulating thermostat.

Research the suspension mass, we use a cylindrical measuring device H, in which the material is placed in a gap created between a fixed plate and a cylinder of radius R, rotating with a constant ω.

A sample of the suspension was placed in the measuring container of a cylindrical device, which was mounted on a viscometer and fixed with a tension ring.

To measure the values of rheological parameters on a rotary viscometer, the following relations are valid:

– shear stress:

$$\tau = c \cdot \alpha, \text{ Pa}, \quad (1)$$

where c is the constant value of the cylinder, 10^{-1} Pa/scale divisions; α is the value of the indicators to the indicator device/scale divisions /.

– effective viscosity:

$$\eta = \frac{\tau}{\gamma}, Pa \cdot s, \quad (2)$$

де γ - shear rate, s^{-1}.

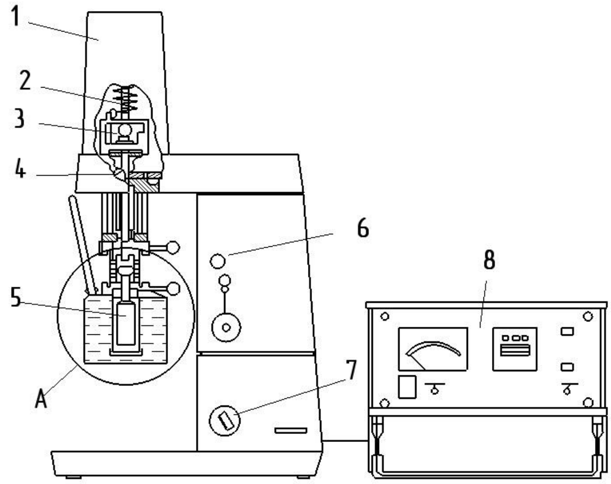

Fig. 2a The scheme of the rotary viscometer REOTEST 2:
1 – the case of the viscometer; 2 – dynamometer; 3 – potentiometer; 4 – measuring mechanism; 5 – cylindrical device; 6 – speed scale, 7 – speed switch (a – b), 8 – measurement unit

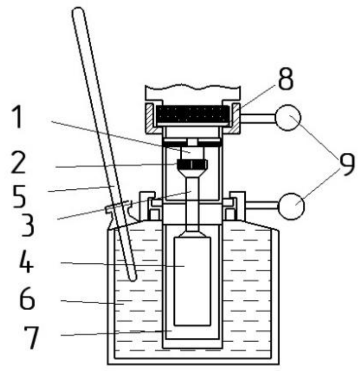

Fig. 2b Cylindrical measuring device:
1 – coupling; 2 – bushing; 3 – shaft; 4 – cylinder; 5 – thermometer; 6 – thermostatic chamber; 7 – measuring capacity; 8 – tension ring; 9 – tension lever.

RESULTS AND DISCUSSION

The obtained results make it possible to construct a flow curve of the suspension during grinding (Rachok V., Hudzenko V., Telechkun YU., Telechkun V., 2018). Research have shown that within the research range, the suspension does not change the nature of the flow depending on the time interval of grinding and temperature change.

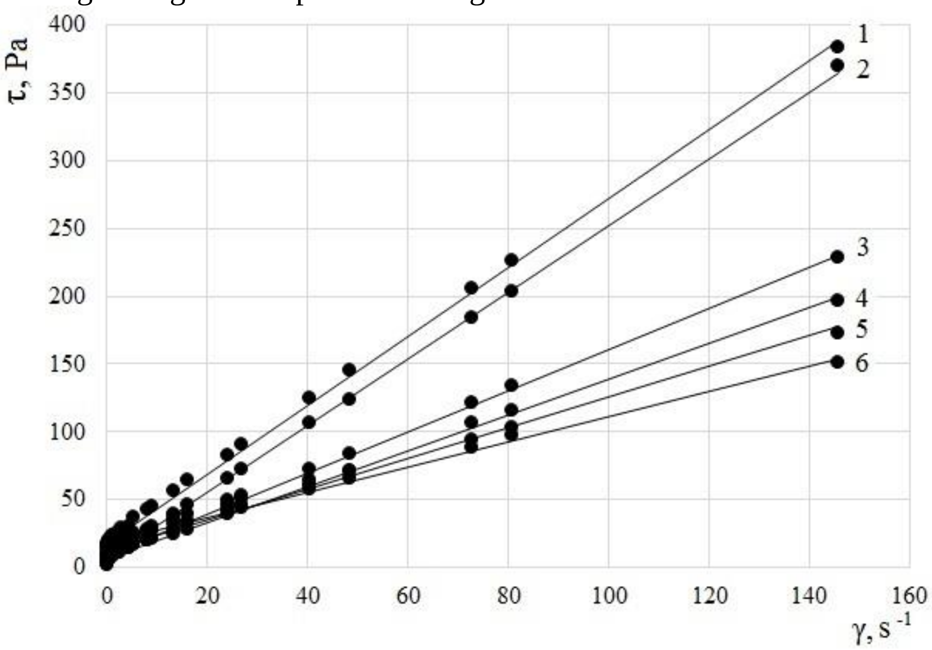

Fig. 3 Experimental curve of the flow of the suspension depending on the temperature change in the working chamber: 1 – 0 min (20 ° C); 2– 5 min (24 ° C); 3–10 min (26 ° C);
4 – 15 min (28 ° C); 5 – 35 min (32 ° C); 6 – 45 min (34 ° C).

A set of straight lines with different angles of inclination is obtained, which are described by the equation of the dependence of the shear stress on the shear rate during the grinding of the suspension:

$$\tau = (-0,015t + 1,62)\gamma + 0,27t + 4,7 \quad (3)$$

This dependence is linear.

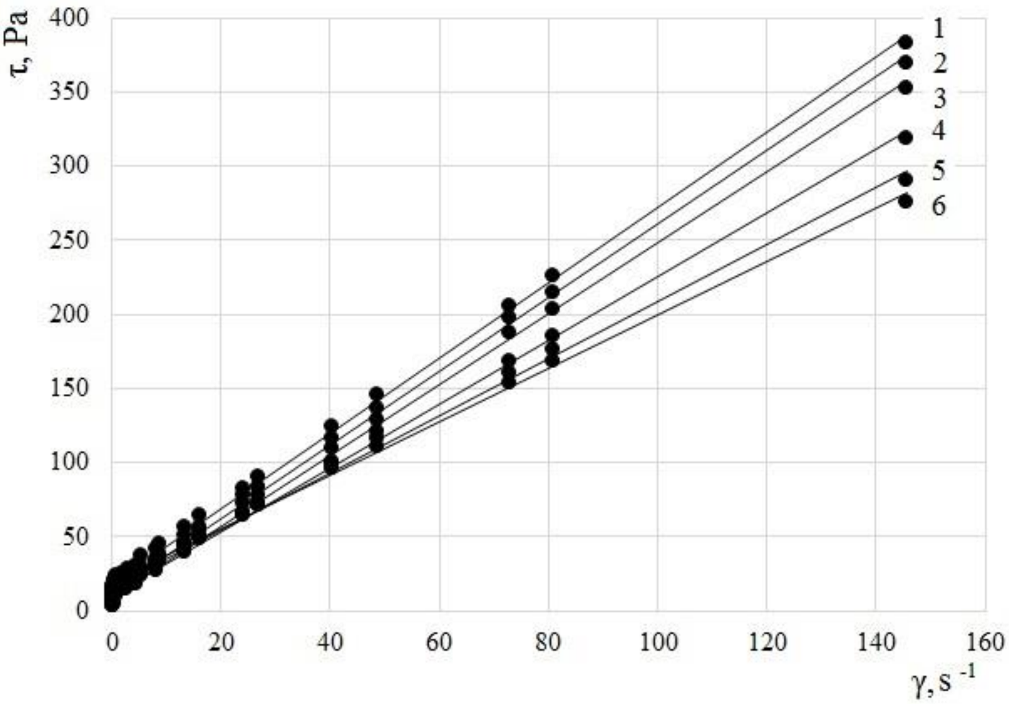

Fig. 4 Experimental curve of the flow of the suspension under standard conditions (20 ° C) in the working chamber:
1 – 0 min; 2–5 minutes; 3 – 10 minutes; 4 – 15 minutes; 5 – 35 minutes; 6 – 45 minutes

A set of straight lines with different angles of inclination is obtained, which are described by the equation of the dependence of the shear stress on the shear rate during the grinding of the suspension:

$$\tau = (-0{,}015t + 2{,}5)\gamma + 0{,}3t + 5{,}8 \quad (4)$$

The flow curve of the suspension (Figs. 3 and 4) during grinding has features, which is explained by the formation and formation of the structure. The constant value in the formula is the ultimate shear stress of the formed suspension structure, which is achieved at a shear rate within 150 s^{-1}.

The investigated finished product has 6.5 times higher viscosity in relation to pure castor oil, which is explained by the high dry matter content (40%) and the increase in the newly formed area.

The viscosity of the suspension varies with temperature: when cooled to 20 °C, the finished product has a viscosity of 22.5 % higher than immediately after the grinding process with a temperature of 34 °C.

The viscosity of the suspension varies depending on the concentration of dry matter: the higher the concentration of dry matter in the suspension, the higher the viscosity of the latter.

Changing the temperature and concentration of dry matter in the suspension based on castor oil can increase the viscosity of the product and ensure its stability from delamination and coagulation, without using surfactants as stabilizers, but increasing the viscosity leads to deterioration of consumer properties: difficult dosing, reemulgulation or resuspension, so this approach is possible for optimal storage of the product as a semi–finished product.

The obtained experimental data make it possible to obtain a change in the effective viscosity of the suspension during grinding and its dependence on the shear rate, which confirms its non–Newtonian character (Fig. 5).

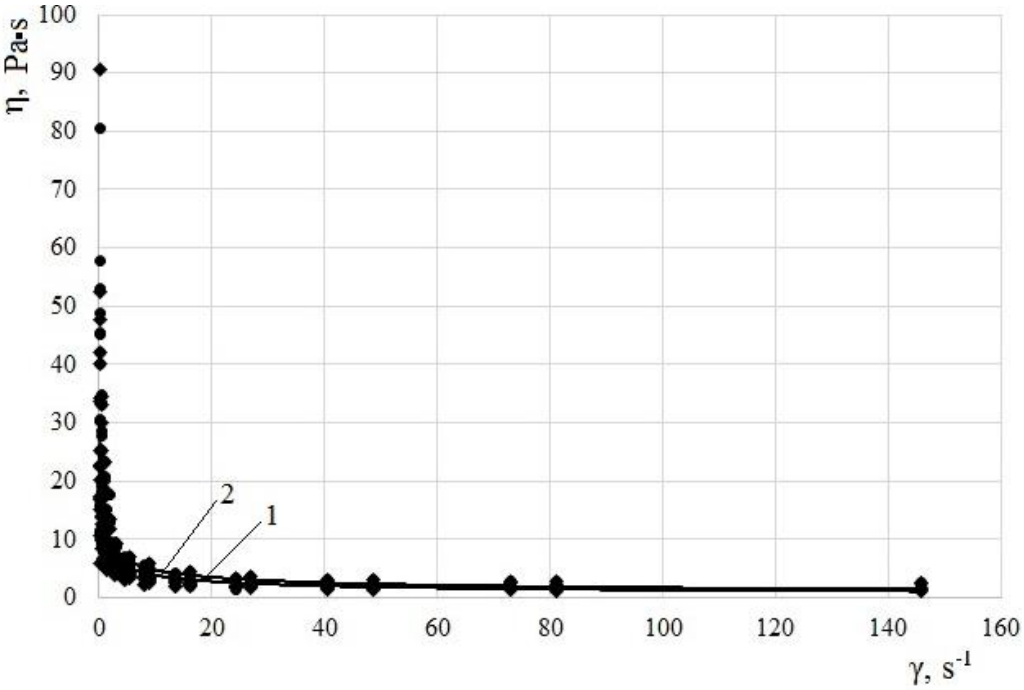

Fig. 5 The dependence of the effective viscosity on the shear rate during grinding depending on the temperature change in the working chamber

As a result of mathematical processing of experimental data the equation of elasticity of effective viscosity from shear rate during grinding of suspension is received:

$$\eta = 13{,}325\gamma^{-0{,}443} + 11{,}023\gamma^{-0{,}456} \quad (5)$$

This dependence is of a power nature.

Increasing the grinding temperature and reducing the concentration of dry matter in the suspension, although it reduces the instantaneous energy consumption, but increases the residence time of the product in the grinding chamber, and, accordingly, increases the overall energy consumption of the process.

CUNCLUSION

1. The suspension within the investigated range does not change the nature of the flow depending on the time interval of measurements and the change in temperature.
2. The constant value in the formula is probably the ultimate shear stress of the formed structure, which is explained by the formation and formation of the structure.
3. The effective viscosity during grinding and its dependence on the shear rate confirms the non–Newtonian character of the suspension and is defined as a linear plastic body.
4. The finished product has 6.5 times higher viscosity than pure castor oil, which is due to the high dry matter content (40%) and the increase in the newly formed area.
5. The viscosity of the suspension varies depending on the temperature: cooled to 20 ° C, the finished product has a viscosity of 22.5% higher than immediately after the grinding process with a temperature of 34 ° C. The viscosity varies depending on the dry matter concentration: the higher the dry matter concentration in the suspension, the higher the viscosity of the latter.

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