Effect of compression modes on tablet strength and friability

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Abstract

Introduction. The aim of study was to determine the effect of the number of compression cycles and rotary table speed on the strength and friability of tablets.

Materials and methods. Two tablet samples manufactured on an industrial rotary pressing machine with a rotary table speed of 30 to 80 rpm were studied. Tablet strength was assessed by the breaking stress in the diametrical direction, and abrasion was measured by the percentage of mass loss during processing in a rotating drum.

Results and discussion. With an increase in the number of pressing cycles from 28,000 to 280,000, which corresponds to approximately 8 to 80 hours of machine operation, the tablet strength decreases to 10% of the initial value of 1.02–1.52 MPa. The decrease in strength occurs according to a parabolic dependence - during the first 100,000–150,000 cycles (30–35 hours of press operation) it is insignificant, and then accelerates. Under the conditions of this study, the process should be stopped after 200,000–250,000 pressing cycles, that is, after approximately 60–70 hours of press operation.

An increase in the friability value was observed with increasing the compressing cycles: at the beginning of the experiment, the percentage of friability was 0.18–0.30%, at the end of the experiment it increased to 1.19–1.24%. When the rotation speed of the rotary table increases from 35 to 80 rpm, the percentage of friability of tablets increases from 0.15 to 1.24%, and the strength was decreases from initial 2.3–3.5 to 10% with linear dependence. The value of tablets friability was higher than pharmacopoeia recommendation (1%). The decrease in tablet quality is explained by product sticking to the press tool surface, increased surface roughness, friction coefficient, and adhesive strength. As a result, the outer layer of the tablet had lower strength properties and was subject to rapid destruction. The reduction in strength at a higher rotation speed of the rotary table is explained by a decrease in the holding time in the die and a higher speed of pushing out of the tablet, which reduces the strength of its outer layers. Increased friability of the tablet affects subsequent technological operations: dust accumulates in the tablet feed channels for blister packaging, making it difficult to move them, and the tablets lose weight when applying a coating on drum machines. A decrease in the strength of the tablet leads to its fragmentation during various technological operations and storage.

Conclusions. For the tablets used in the study, the optimum table rotor speed was 50 rpm. Increased rotation speed and productivity can be achieved by more frequent polishing of the die and punch surfaces.

Introduction

When pressing on rotary machines, deviations in the strength and friability of tablets may be observed, which leads to a violation of their technological parameters (Siiriä et al., 2011), namely, in their integrity during subsequent coating, packaging, transportation and storage operations (Herasymenko et al., 2024; Natoli et al., 2017), can affect product safety (Gordiienko et al., 2015), and affect the profitability of production.

It is obvious that the strength and friability of tablets are primarily influenced by the technology and degree (effort) of tablet pressing. However, these indicators can also be affected by the operating parameters of the pressing equipment, in particular, the rotation frequency of the rotary table of the pressing machine, and the number of pressing cycles, i.e. the number of tablets produced in one press pair "die-punch" (Herasimenko et al., 2024). The influence of these factors is not sufficiently described in the scientific literature, so the issue requires additional research.

There is disagreement about the terminology characterizing the strength properties of a tablet. The terms "hardness", "strength", "tensile strength", "breaking stress" are used. Most researchers consider the term "strength" to be correct, characterizing the stress at which a tablet breaks under lateral (diametrical) compression. Note that "strength" cannot be called "hardness": from a mechanical point of view, these are different quantities measured by different methods.

Usually, at determining the strength, the shape of the tablet is not paid attention to, however, it has a scientific interest the determination of stresses in tablets of different shapes – from the usual flat (cylindrical) to more complex ones: convex, capsule-shaped, oval, and others. These problems are solved by mathematical modelling, and more recently by using software packages based on the finite element method (Pitt et al., 2016; Siiriä et al, 2011).

To characterize such a phenomenon as the separation of small dust-like particles from a tablet, the terms "scraping", "friability", "abrasion", “wearability” are found, and the incorrect terms "fragility" and “brittleness” is occurred. Brittleness is the property of a product to fracture with little elastic deformation and without significant plastic deformation (Goots et al., 2016; Rösler et al., 2007), this term is relevant for ceramic products, glass, concrete, etc., but it cannot characterize the loss of tablet mass due to the separation of its small particles. The term "friability" is commonly used and corrected.

The value of tablet strength is usually not regulated by external regulatory documents and the Pharmacopoeias (Pharmaeducation, 2024), and is set by the drug manufacturer (CDER, 2015). However, the deviation from the accepted strength should not exceed 5%. Usually, the lateral force at which the tablet breaks is between 40 and 100 N, and the strength derived from it depends on the size of the tablet (Pharmaeducation, 2024).

According to the harmonized European Pharmacopoeia, the value of friability should not exceed 1% (European pharmacopoeia, 2024), however, some researchers consider this value to be overestimated and recommend a friability value of 0.3–0.5% (Podczeck, 2007).

The strength and friability of tablets are influenced by the technology, degree of pressing, type and modes of operation of the equipment, the geometrical parameters and state of the pressing tool surface. Taking into account a significant number of random factors, the strength and friability of tablets are supported technologically, and instrumentally – by setting the pressing tool and the operating modes of the equipment.

Most researchers pay attention to the dependence of strength and friability only on the technology of tablets, compression force or degree of pressing. For example, with an increase in compressing force (and, accordingly, the degree of pressing) from 8 to 30 kN, the strength of the tablet increases from 0.2 to 1.4 kPa; with high compressing force, the strength of the tablet changes slightly (Pitt et al., 2013). However, the experience of engineers and adjusters of compressing equipment indicates that strength, friability and other quality indicators are affected by the kinematic parameters of the press and its operation time. During several workdays of the compression, the tablet strength may decrease by more than 10%, and the friability may exceed 1% (Herasymenko et al., 2024). Similar indicators of failure occur at high speeds of the rotary table. Therefore, the issue of regulating the structural-mechanical and quality indicators of tablets by changing the operating parameters of the equipment and the condition of the surface of the pressing tool requires additional research.

The aim of the study was to determine the effect of the number of compressing cycles and the rotational speed of rotary table of the press on the strength and friability of tablets.

Materials and methods

The strength and friability of tablets were studied. This study is not medical one, does not make any medical claims, and is intended solely to determine the effect of pressing process parameters on tablet strength and friability.

Materials

Strength of two well-known medicines in the form of tablets were studied:

- Tablets 1 were made on the basis of: citric acid monohydrate, potato starch, povidone, cocoa, calcium stearate, and API: acetylsalicylic acid, paracetamol, and caffeine.
- Tablets 2 were made on the basis of: potato starch, talc, calcium stearate, and AFI sodium metamizole.

Equipment

The tablets were produced under industrial conditions on a Korsch XL 400 tablet compression machine. The machine provides the possibility of working with a value of preliminary pressing of 20 kN and main pressing of 100 kN, a maximum productivity of 338,000 tablets per hour at a maximum rotary table speed of 120 rpm.

The schematic diagram of the tablet compression machine is shown in Figure 1. The main operations – filling the die with granulate, preliminary and main compression of the tablet, pushing out the tablet.

During the study of tablets for strength, the tablets were taken every eight hours of continuous operation of the press (or every 28.2 thousand finished tablets per one die). For each study, 10 tablets were selected.

During the study of tablets for friability, the rotary table speed was 35, 50, 65, and 80 rpm. 12 tablets were used for each friability test.

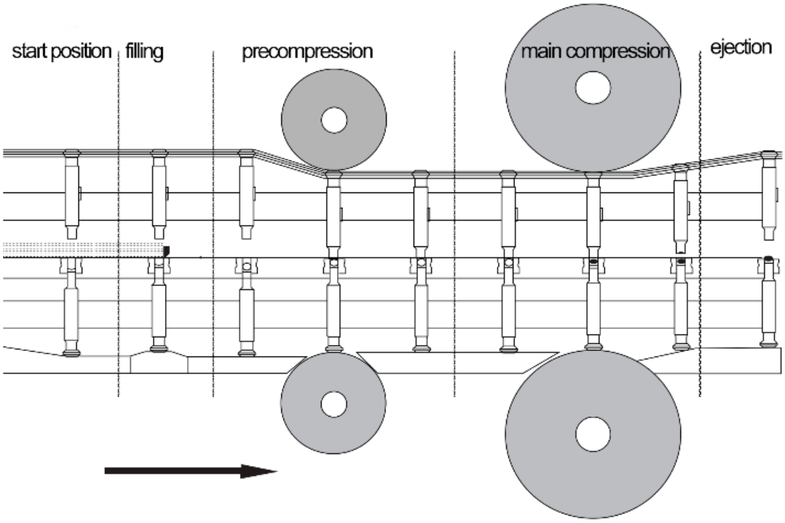

Figure 1. Schematic diagram of Korsch XL 400 tablet compression machine

Determination of tablet strength

The strength (or ultimate strength) (European pharmacopoeia, 2024) of a tablet was determined by a method generally accepted in pharmaceutical activity: the tablet was compressed in the diametrical direction, the destructive force P at which the tablet was destroyed was determined, and according to the destructive force, the ultimate strength σ_p was determined.

The strength (ultimate strength) of the tablet σ_p is the ratio of the destructive force to the cross-sectional area of the tablet:

$$\sigma_p = \frac{P}{d \cdot h}, \text{ Pa} \tag{1}$$

where *P* is the destructive force, H; *d* – tablet diameter, m; *h* – tablet height, m.

Destructive force P of a tablet is a force applied in the diametrical direction at which the tablet is destroyed.

In this study, the destructive force of the tablet was determined on the device for strength determining the PTB-M 300 N Pharma Test. The working element of the device is two sponges directed towards each other, which move in the horizontal plane and compress the tablet in the diametrical direction. The accuracy of the device is 1 N. Each test was repeated 10 times.

Determination of tablet friability

Tablets were tested for friability using the "ERWEKA TAR II" device, where working element is a rotating drum with one blade (European pharmacopoeia, 2024).

The drum was made from transparent polymer material. The inner diameter of the drum is 283–291 mm. The rotational speed was 25±1 rpm. A blade of a certain shape was installed in the drum, which creates the lifting and lowering of the tablet. During the rotation of the drum, the tablets fall from a height of 156±2 mm or 6 inches (European pharmacopoeia, 2024).

10 tablets are dedusted, weighed and placed in the drum, close the lid and turn on the device for 4 minutes. After that, the procedure of weighing them is repeated.

The friability index P, %, was determined by the formula:

$$F = 100 - \frac{P_b - P_f}{P_b} \cdot 100 \tag{2}$$

where P_b, P_f – the mass of tablets before and after experiment.

Assumptions

- API does not have a significant effect on strength and friability indicators;
- The change in strength and friability of tablets, like other quality indicators, depends on the number of compressing cycles – that is, how many tablets were produced by one press couple (die-punch). The indicator "Number of compressing cycles" is convenient to use when the results are analysing and comparing, for example, if the experiment was carried out on other compression machine with a different rotary table speed, or on static (crank) presses.
- The strength and friability indicators are depended not the rotary table rotational speed, but by the speed of movement of the punches caused by it, and, accordingly, by the speed of compressing and pushing out the tablet.

Results and discussion

Effect of number of compressing cycles on tablet strength

For two tablets of tablets, it was observed a decrease in their strength up to 10% during three hundred thousand cycles of compressing cycles, which is 80 hours of the tablet press operation.

With an increase in the number of compressing cycles from 28 to 280 thousand, which is approximately from 8 to 80 hours of press operation, the strength of tablets 1 decreases from 1.02 to 0.91 MPa, and of tablets 2 – from 1.52 to 1.40 MPa (Figure 2). The decrease in the strength of tablets occurs according to a parabolic dependence – the first 100–150 thousand cycles (30–35 hours of press operation) it is insignificant, and then it accelerates. Over time, the strength of the tablets decreases by more than 5%, therefore, in order to guarantee constant product quality, the process should be stopped and measures taken to restore the initial strength. In this study conditions, the process should be stopped at 200–250 thousand compressing cycles, that is, approximately 60–70 hours of press operation.

The manufacturer must guarantee the stable strength of the tablets, but it decreases during long-term operation of the tablet press. One of the methods known in practice to maintain stable strength is monitoring the condition of the surface of dies and punches, in particular, to carry out periodic polishing (Qiu et al., 2020).

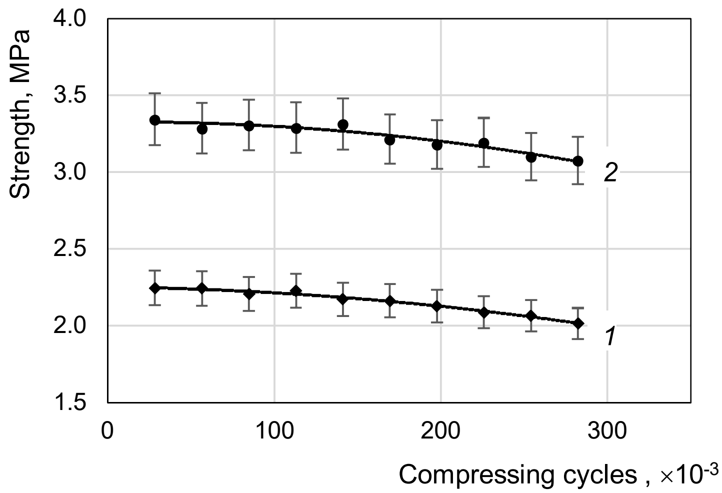

Figure 2. Effect of number of compressing cycles on the strength of tablet:
1 – Tablets 1; 2 – Tablets 2

Preliminary observations in this experiment showed that after polishing the working surfaces of the punches and dies, the strength of the tablet returns to the initial values within 7–10 times, but then other disturbing factors appear that lead to a decrease in the initial strength of the tablets. This issue requires additional research.

In the case of this study, it is suggested to polish the pressing tool after 48 hours of working operation.

The decrease in tablet strength during long-term press operation can be explained by the fact that a sticky layer is formed on the surface of the press tool, and, accordingly, the force (stress) of friction and pushing out the tablet in the die increases (Figure 3). As a result, the surface layers of the tablet are less strong, and the distribution of the tablet density along the height and width is inconsistent.

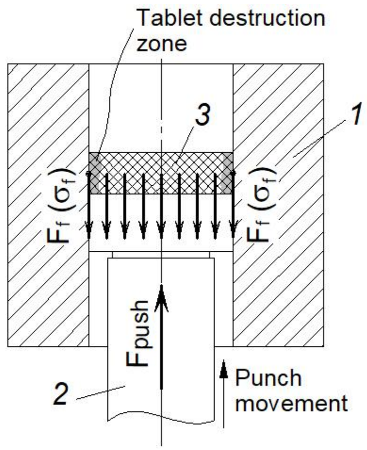

Figure 3. Forces acting on tablet lateral surface during pushing out from die:
1 – die; 2 – punch; 3 – tablet.
F_f – friction force, H;
σ – friction stress, Pa.

The gray region is zone of intensive destruction of the tablet lateral surface.

Effect of rotary table speed on tablet friability

Information obtained from specialists who maintain tableting equipment and personal observations have shown that tablets made at high rotary speeds have lower strength and higher friability rates. The rational speed of the rotary table is usually determined based on production experience and validation protocols of the drug production process.

When the rotation speed of the rotary table increases from 35 to 80 rpm, the percentage of friability of tablets 1 increases from 0.15 to 1.24% (Figure 4).

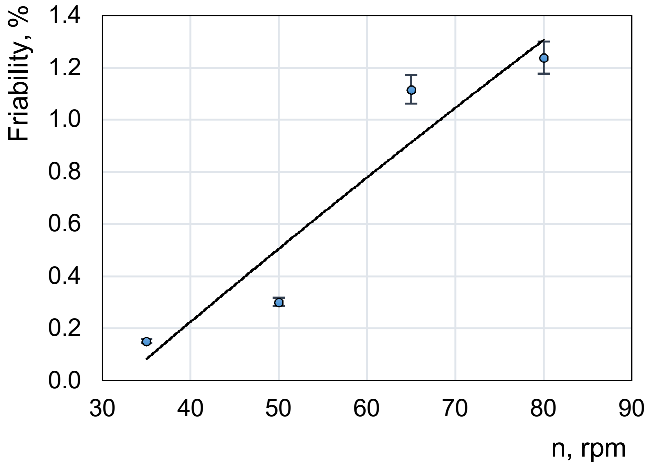

Figure 4. Effect of rotary table speed (n) on tablet friability

According to the above assumption, friability is affected not by the rotation speed of the rotary table, but by its derivative – the speed of movement of the punches in the die, and, accordingly, the speed of pushing of the tablet from the die. It is explained by the fact that at high speeds of pushing the tablet out of the die, the outer layer of the tablet is destroyed due to high frictional forces (Fig. 3). This layer is excessively worn away in the following technological and finishing operations. This type of defect is negatively manifested during coating process in drum and fluidized bed machines (Salawi, 2021), transportation between equipment and during the packaging of tablets in blister.

The effect of sliding speed on the friction coefficient or stress has been reported in other studies. For example, for semi-finished products and food products with high adhesion strength indicators (bread, meat products, cheeses), the friction (or adhesion) stress increases significantly at high sliding speeds, and this affects their machining by cutting. For products with low moisture content, the effect of sliding speed on friction (adhesion) stress is insignificant (Gubenia et al., 2010).

Additional research is needed for other factors affecting on a friability, in particular, vibration in the press tool, surface roughness of the die and punch, methods and depth of periodic polishing.

Effect of number of compressing cycles on tablet friability

An increase in the friability value was observed with increasing the compressing cycles: at the beginning of the experiment, the percentage of friability was 0.18–0.30%, at the end of the experiment it was 1.19–1.24% (Figure 9).

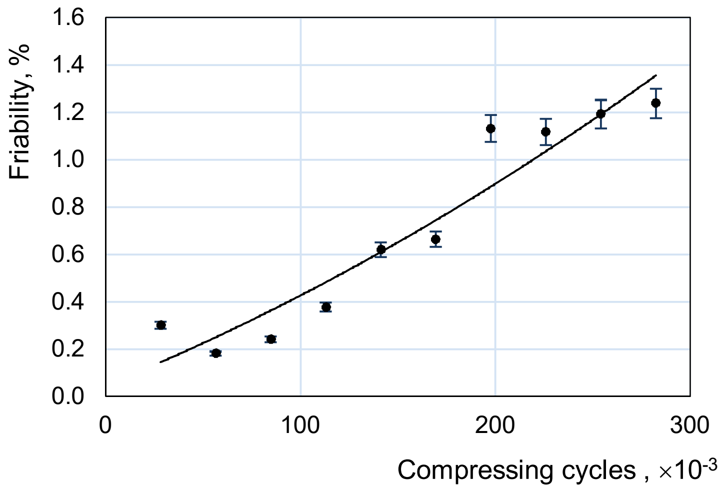

Figure 5. Effect of number of compressing cycles on friability of tablets

The consequence of tablet friability during packaging – dusty channels for feeding the tablet into the cell of the blister pack (Trudko et al., 2024) (Figure 6). In the event of dusting, the tablets get stuck in the streams and do not enter into the blister cells – empty cells appear. It takes time to clean equipment elements from dust, and it is undesirable in production.

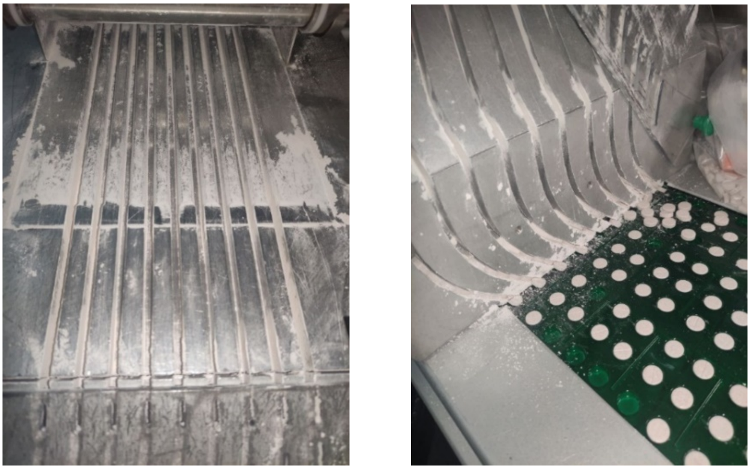

Figure 6. Dusty cells of feeder in the blister machine

Effect of rotary table speed on the strength of the tablets

In industry conditions, a rational compressing speed is usually set for each type of tablet, which depends on the rotary table speed (Tye et al., 2005). At high compression speeds, tablet strength decreases (Swarbrick, 2007).

During the experiment, an almost linear dependence of tablet strength was observed within the range of rotary table speed from 35 to 80 rpm (Figure 7).

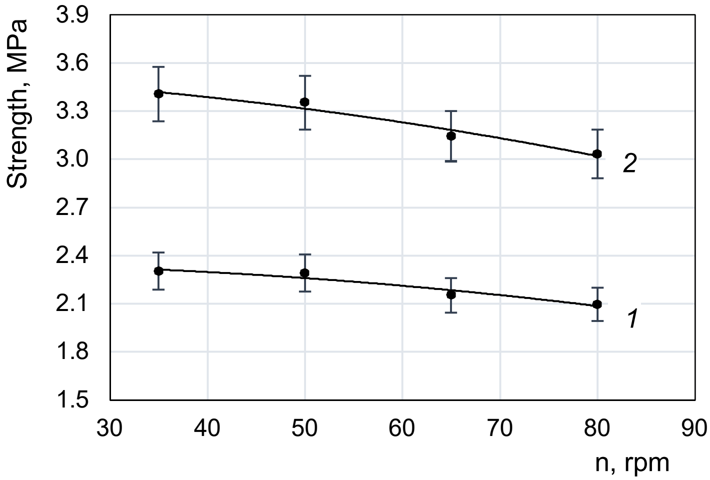

Figure 7. Effect of rotary table speed (n) on tablet strength:
1 – Tablets 1; 2 – Tablets 2

It is explained by the fact that at high compression speeds the tablet structure does not have time to form, and high pushing out speeds lead to increased frictional forces and destruction of the outer layers of the tablets.

Within the examined range of rotary table speed, the strength of tablets decreases to 20%. Taking into account the need to ensure stable indicators of the quality of tablets, it is not advisable to exceed the rotary table speed more than 50 rpm.

Tablet strength is probably also affected by the holding time when the upper and lower punches are stationary. The holding time is depended on the size of the flat surface of the punch head flat, which is in contact with the pressure roller (Swarbrick, 2007). It should be noted that the head radius and angle, which are responsible for the movement of the punch, decreases, and, accordingly, the speed and acceleration of the punch will increase.

Conclusions

1. The number of compressing cycles in the press couple “Die-punch” affects the strength of the tablet: after 300,000 cycles, the tablet loses up to 8% strength on average. This is explained by the sticking of the product on the surface of the die and the punch, an increase in the roughness of their surface and, accordingly, an

increase in the coefficient of friction and adhesion strength. As a result, the outer layer of the tablet has lower strength indicators and is intensively destroyed.

2. Within the working range of the rotary table speed from 30 to 80 rpm, the strength of the tablets can decrease by 10%. This is explained by a change in the compressing speed and a decrease in the holding time of the tablet in the die.
3. With an increase in the rotary table speed from 30 to 80 rpm, the tablet friability increases from 0.2 to 1.2%, it is more than the regulated pharmacopoeial (1%) and recommended (0.5) norms. It is due to a decrease of the holding time of tablet in the die and a higher pushing out speed, which reduces the strength of the outer layers of the tablet. High friability leads to a worse quality of applying a protective coating to the tablet, an increase in the number of scraps at the coating stage, and dustiness of the feeder of the blister machine.
4. Increasing strength and friability indicators imposes restrictions on the rotary table speed. For the tablets used in this study, it is 50 rpm. An increase of speed, and, accordingly, productivity, is possible due to more frequent polishing of the surface of dies and punches.

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