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Influence of the punch speed on the die-wall/ powder kinematic friction during tableting

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Abstract

Influence of the compaction speed on the final tablet properties is an important challenge during the scale-up of a solid dosage form. This strain rate sensitivity is generally attributed to the time dependent deformation behavior of the powder. In this work, we studied the influence of the speed on another important factor during compaction: friction between the tablet/powder and the die.

An original experimental methodology was developed to study the evolution of the kinematic friction coefficient between the tablet and the die as a function of the sliding speed of the tablet on the die wall. This methodology made it possible to separate the speed used to make the tablet from the speed used to measure the friction coefficient. Results indicate that the kinematic coefficient of friction increases with the sliding speed following a logarithmic trend. This trend was observed for 4 different pharmaceutical excipients. Moreover, it was proved that the speed dependency is an intrinsic property of the friction between a tablet and a die lubricated using magnesium stearate.

keywords: friction, compaction, speed, strain rate sensitivity.

1. Introduction

Tableting is one the most important process for the manufacturing of drugs. Despite an apparent simplicity, the phenomena occurring during tableting are complex and neither fully described nor understood. Some classical manufacturing problems, like capping and sticking, even if known since more than a century, can still be industrial issues today. Among the challenges during the development of new tablets, scale-up is one of the most critical, because some problems may remain undetected during development. The main reason is often the difference between the tablet presses used in development and the presses used in production. The conditions of compression may be different between different kinds of presses, leading to different results in terms of tablet final properties. As a consequence, compaction simulators have been developed to fill the gap between development eccentric presses and large production rotary presses.

The main parameter that may change during the scale up from a development press to an industrial press is the compaction kinematic. In fact, on high capacity rotary presses with large turrets, the compaction speed can be very high with punch tip maximal speed much higher than on eccentric presses. It is well known that in a lot of cases, the speed of the punches, i.e. the speed at which the pressure is applied, can have consequences of the final tablet quality. This phenomenon is generally called Strain Rate Sensitivity (SRS), and has been widely studied in the literature ¹. Several aspects can explain the SRS. First, the deformation behavior of the powder can depend on the strain rate. Viscoelasticity corresponds to the changes of the elastic behavior and viscoplasticity corresponds to the changes of the plastic behavior with the strain rate. Both aspects have been largely studied ²⁻⁸. Another part that can explain the SRS is the air entrapment during compaction. When compaction speed

increases, air can have difficulties to escape and may thus change the apparent deformation behavior of the powder ^{9,10}.

The previous aspects refer to the deformation behavior of the powder. Nevertheless more recently ¹¹ it was shown that the interaction between the die and the powder, i.e. friction, can also be modified when the speed increases. In fact, the ejection force was found dependent on the compaction speed. Sun proposed a mechanism to explain this phenomenon. His hypothesis was that magnesium stearate was migrating toward the die during compaction. At slow speed, it would have more time to migrate toward the die. At high speed, lubrication would thus be less efficient. Nevertheless, there was no definite proof in the cited study that the migration of magnesium stearate was indeed responsible for the friction variations. Studies performed in other domains have also shown that the friction coefficient between two surfaces can change when the sliding speed between the two surfaces changes ¹².

Concerning the friction coefficient, it is important to separate the static friction coefficient from the kinematic coefficient of friction ¹³. The static coefficient of friction acts before the powder or the tablet starts moving. The kinematic coefficient of friction acts when the powder is moving. These two coefficients can be different, especially if there is adhesion between the two surfaces. In all this work, we will study the kinematic coefficient of friction.

The present work aims to study the influence of sliding speed on the kinematic friction coefficient between the powder and the die-wall. A protocol of measurement was defined in order to separate the compaction speed during the making of the tablet and the punch speed during the measurement of the friction coefficient. Moreover, external lubrication was also used, in order to have a level of magnesium

stearate on the die wall independent of the punch speed. This made it possible to isolate the dependence of the friction coefficient on the speed from the problem of magnesium stearate migration during compaction. At the end of the study, practical consequences of the variation of the friction coefficient are proposed.

2. Material and method

2.1. Materials

2.1.1. Powders

Experiments were performed on four classical pharmaceutical excipients: Lactose Monohydrate (GLac) (Excipress, ArmorPharma, Maen Roch, France), Spray-dried Mannitol (SDMan) (Pearlitol 200SD, Roquette, Lestreme, France), Anhydrous Calcium Phosphate (Dicafos A60, Budenheim, Budenheim, Germany) and Calcium sulfate (CaSul) (Compactrol, JRS Pharma, Rosenberg, Germany). External lubrication was performed using Magnesium Stearate (Partek Mg Lub, Merck, Darmstadt, Germany).

2.1.2. Powder compaction

All compaction experiments were performed on a compaction simulator Styl'One Evolution (Medelpharm, Beynost, France). This device is a single station instrumented tableting machine. It is equipped with force sensors (strain gauges) on the upper and lower punches and the displacements of the punches are monitored using incremental sensors. An instrumented die using strain gauge technology was used to monitor the die-wall pressure. All experiments were performed using round flat punches with a diameter of 11.28 mm. To normalize the experiments and decrease variability, the filling height was monitored to obtain tablets with a final thickness of about 3 mm. Exact compaction conditions are detailed below in this

article. External lubrication was used for all experiments using an external lubrication system. The system is composed of a pulsed air blow cabinet that makes it possible to spray magnesium stearate powder on the punches and the die before die filling.

2.2. Methodology for the friction coefficient measurement

As explained in the introduction, the aim of the work was to study the evolution of the kinematic friction coefficient as a function of the sliding speed of the tablet on the die wall. For this purpose, the following methodology was developed.

The first requirement was to eliminate any influence, on the friction coefficient, of the compression used to make the tablet. For this purpose, the first step of the method is to make the tablet always in the same conditions, i.e. with a constant compression kinematic. Two compression levels were arbitrarily chosen to make the tablets: 150 MPa and 250 MPa. Moreover, external lubrication was used to keep constant lubrication conditions. No internal lubricant was added, in order to discard any lubricant migration during compaction.

In a second step, the friction coefficient was measured by pushing the tablet with only one punch in order to make it move inside the die. During this kind of experiment, the coefficient of friction can be determined using the following equation ¹¹:

$$\mu = \frac{F_p}{P_{rad}\pi Dh} \quad (1)$$

where μ is the friction coefficient, F_p is the force needed to move the tablet, P_{rad} is the radial stress applied to the tablet, D is tablet diameter and h is the tablet thickness. If the peak force needed to start the movement is taken as F_p , μ will be the static coefficient of friction. If the plateau force to move the tablet at a constant speed is taken as F_p , μ will be the kinematic coefficient of friction.

In his work, Sun used the peak ejection force for F_p . This value is related to the static friction coefficient. The residual die wall pressure at the end of the decompression was taken to estimate P_{rad} and the thickness at the end of the decompression to estimate h . This is an approximation, first because some relaxation may occur between the end of the decompression and the ejection and second because due to the ejection force, the tablet is slightly recompressed. This may affect the height and the die wall pressure. The reason for this approximation was mainly due to the technology used for die wall pressure measurement. When strain gauges are used, the measurement of the die wall pressure is linked with the thickness of the tablet, which can only be known when both punches are in contact with the tablet, which is not the case during ejection. Die wall pressure can thus not be measured during ejection.

To overcome this problem, we changed the way of measuring the friction. After the first compression that made the tablet, the lower punch was moved down inside the die. Then, the tablet was pushed down in the die by the upper punch moving at constant speed. During this phase, a constant force can be achieved which corresponds to the force needed to move the tablet at a constant speed. This force is related with the kinematic coefficient of friction. The speed of the upper punch was changed during this phase to study the influence of the speed on F_p . At some point the tablet enters again in contact with the lower punch. At this moment, it is thus possible to measure both the die wall pressure and the tablet thickness for a given F_p . These values were then taken to calculate the kinematic friction coefficient corresponding to the speed used to push the tablet using equation 1.

The protocol can be summarized as (Figure 1):

(1) The tablet is made using a symmetrical compression under constant kinematic conditions and maximum applied pressure.

(2) The lower punch moves down and the upper punch is used to push the tablet down in the die. During this phase, F_p is measured. The speed of the upper punch can be varied.

(3) When the tablet enters in contact with the lower punch, it is possible to measure the exact height of the tablet and the value of the die wall pressure.

During the phase where the tablet is pushed down in the die, it was possible to obtain constant speeds from 0.25 mm/s to 40 mm/s. Six different speeds were used in this interval: 0.25, 1, 4, 10, 25 and 40 mm/s. The exact value of the speed was measured for each experiment. For each condition, three measurements were performed and all the individual measurement points will be displayed on the following graphs.

3. Results and discussion

3.1. Results

3.1.1. Evolution of the force needed to push the tablet (F_p)

In the first part of the experiment, the force needed to push the tablet down in the die is measured. An example of signals obtained for the same product (SDMan) at three different speeds can be seen in Figure 2. It can be seen that the force is fairly constant. A small peak force at the beginning is only obtained for the lowest speed. This means that in the condition used, especially thanks to the use of external lubrication, adhesion between the powder and the die is very low. There is thus in our case no significant difference between the static and the kinematic coefficient of friction. The force signal during the tablet movement is fairly constant, i.e. we can obtain a stationary state. A mean value of the plateau was calculated and taken as

F_p . Comparison for the three speeds gives a first idea of the evolution of the friction with the speed. F_p clearly increases when the sliding speed increases. It can be noted that for the highest speed, the force seems to increase at the beginning and decrease at the end. This is due to changes in the speed. In fact, at high speed, it takes more time to reach the desire speed and the device need to decelerate earlier to be able to stop at the right time. Constant speed is thus not obtained for the whole movement of the tablet. In this case, F_p is taken when the speed of the punch is stabilized at the right speed and not on the whole plateau.

This experiment was performed for the four products, using two different compaction pressures for the first compression (150 MPa and 250 MPa). The results of the evolution of F_p as a function of the different speeds can be seen in Figure 3.

For each product and for each compression point, the same trend is obtained. F_p increases when the punch speed increases. These results are coherent with those presented by Sun ¹¹ who found an increase of the ejection force when the speed increased. Nevertheless, for each speed, the tablet was obtained under the same conditions and external lubrication guaranties a reproducible lubrication state. Migration of Magnesium Stearate during compression is thus, in our case, not responsible of the evolution obtained. We can also note that the evolution of F_p with the speed follows globally a logarithmic trend.

In more quantitative terms it is interesting to note that F_p can be multiplied by factor until 4, which means the effect of the sliding speed is far from being marginal. Of course, the evolution of the force alone does not give a complete picture of the phenomena. For example, it is not possible to compare the results between the products and the different compaction pressures because the die wall pressure may

change between the different situations. To go further, it is thus important to study the evolution of the die wall pressure in order to be able to finally calculate the friction coefficient.

3.1.2. Estimation of the die wall pressure

As explained above the die wall pressure is measured when the tablet reaches the lower punch. At the same moment the force on the upper punch is also measured. For low speeds, this force is nearly equal to F_p , the force on the plateau. For high speeds, when the tablet touches the lower punch, the speed already began to decrease, and the force is lower than F_p . To be as coherent as possible, for the evolution of the wall pressure, the force considered for the upper punch was the one measured when the tablet touches the lower punch.

The evolution of the die wall pressure as a function of the force on the upper punch for the four products and two compression levels are presented in Figure 4. As expected, different die wall pressures were obtained for the different products, because this parameter is linked with the mechanical behavior of the powder. The values obtained for a compaction pressure of 250 MPa were higher than the values obtained for 150 MPa which was also expected.

For both compression pressure, the die wall pressure slightly increased with force applied by the upper punch. This increase was generally between 1 and 2 MPa on the force range explored. This increase can be explained by the slight recompression experienced by the tablet due to the force applied by the upper punch. It can be approximately represented by a linear relationship. These linear relations were thus

taken to calculate the value of the die wall pressure for each experiment in order to be able to estimate the friction coefficient.

3.1.3. Evolution of the friction coefficient

Finally, it was possible for each experiment performed to estimate the friction coefficient. All the results are presented in Figure 5.

The results indicate that for all the cases the friction coefficient increased when the punch speed increased. These results confirm the findings of Sun ¹¹, but thanks to our methodology, it is possible to affirm that, in our case, this evolution is not related with the migration of magnesium stearate during the compression phase, as the measurements for different speed were made on tablets obtained under the same conditions. This evolution is an intrinsic property of the friction between a tablet and a die lubricated with magnesium stearate. As for the F_p , the evolution of the friction coefficient follows globally a logarithmic trend.

If we compare the products, it seems that SDMan, GLac and CaSul gave a similar trend in terms of evolution. At low speed, they gave similar friction coefficients. At the highest speeds the results are slightly different, especially SD Man seems to give higher coefficients of friction. On the contrary, ACP has a different behavior with a much smaller dependence to the speed. If we consider the tablets obtained under 150 MPa, the increase of the friction coefficient for ACP was around 50% on the speed range whereas the increase was around 200% for the other products.

Another interesting point is to compare the friction coefficients obtained for tablets compacted at 150 MPa and 250 MPa. Changing the pressure used to make the tablet has two main consequences. First it changes the structure of the tablet and, as a consequence, it can change the structure of the interface between the tablet and

the die. Second it changes also the value of the die pressure applied on the tablet. Comparison of the coefficient of friction between the two situations for the four products is presented in Figure 6. From this results it is difficult to obtain a general trend. For SDMan and ACP, no influence of the first pressure is obtained. For CaSul and GLac, the results seemed to indicate a slight decrease of the friction coefficient when the pressure increases, especially at high speed. Nevertheless, for each product, the influence of the first compaction is much lower than the influence of the speed of the punch.

3.2. Discussion and consequences

3.2.1. Discussion

The protocol defined above made it possible to confirm that the coefficient of friction between the tablet and the die in a tableting machine is dependent on the sliding speed between the tablet and the die, as observed by Sun. As the conditions to make the tablet were kept constant for each sliding speed and external lubrication was used, the increase in the friction coefficient can be considered as an intrinsic property of the friction between a tablet and a die lubricated with magnesium stearate.

Lubrication using magnesium stearate, is generally considered as a boundary lubrication, i.e. magnesium stearate forms a thin film at the surface of the die that interpose between the two sliding surfaces ¹⁴. In this kind of situation, as the lubricating film will reduce the surface interaction between the two sliding surface (here the die and the product in the tablet), the friction is due almost entirely to the shear strength of the lubricating film¹⁵. Several studies have shown that the shear strength can be dependent on the shear rate, and as a consequence, the friction

coefficient can be dependent on the sliding speed¹⁶. More precisely, Campen et al. reviewed recently a number of published studies where the friction coefficient increases with of the sliding speed according to a logarithmic trend¹². Results presented in Figure 5, are totally coherent with the cited literature as it shows that, in the velocity range studied, the friction coefficient increases with the sliding speed in a logarithmic way. This means that the shear strength of magnesium stearate is dependent on the shear rate, as it was already demonstrated for thin layers of stearic acid¹⁷.

As noted before, CaSul, GLac and SDMan gave nearly the same result which is understandable, as the friction is mainly govern by the properties of the magnesium stearate film. Nevertheless, some contact between the two layers (excipient/metal) may remain, which could explain that the values are not totally the same. The case of ACP is different because it gave a different trend for the evolution of the friction coefficient, with a higher value at low speed and a smaller sensitivity to the sliding speed. To try to understand this different trend, the sliding surface of the tablets (i.e. the tablet band) was observed using scanning electron microscope. Photographs of the tablet band for the four products obtained under a pressure of 150MPa can be observed in Figure 7. These images make it possible to compare, between the four products, the effective contact surface between the tablet and the die.

GLac and SDMan gave relatively smooth tablet band and a more porous surface is observed for CaSul. Nevertheless, for these three products, a large part of the band is indeed in contact with the die. The situation is completely different for ACP. To better visualize the surface, another photograph obtained with a higher magnification is presented in Figure 8. The surface is far from being smooth and the structure of the grains is still clearly visible on the photograph. It is easy to see the points that

were in contact with the die, because they present a very smooth surface. Using image analysis, it is possible to estimate that only around 10-15% of the apparent surface was indeed in contact with the die. This means that, for example, the actual local die-wall pressure is much higher than the value considered in this study (which take into account the whole side surface). Even if it is difficult to propose a complete mechanical explanation of the observed variation, this difference in contact surface and pressure constitute a plausible explanation of the typical behavior of this product. Finally, it must be noted that the grade of ACP used in this study (Dicafos A60) has very typical properties compared to other commercial available grades ¹⁸.

3.2.2. Practical consequences

Increasing the sliding speed between the tablet and the die wall increases friction. This has of course practical consequences in terms of tableting. First, it might explain part of the strain rate sensitivity observed during compaction. Moreover, during compaction, the sliding speed between the powder and die is not constant, both during the compression and when considering the different parts of the tablets.

During compression, as the punches have to move backward, punch speed is decreasing when approaching the compression top. This means that the friction coefficient is of course not a constant during the compression event. More precisely, when approaching the compaction top, as the speed decreases, friction also decreases. This result might explain why most of the time, the friction coefficient measured in the literature are decreasing during the compaction event^{19,20}.

Moreover, for example in a symmetrical compaction, the center of the tablet is fixed, whereas the parts in contact with the punches are moving at the punch velocity. Between these two parts of the tablet, there is a speed gradient. This means that

312 considering a constant coefficient of friction for the interaction between the die and
313 the tablet is clearly an oversimplification of the reality.

314 **Conclusion**

315 In this work, we demonstrated that the kinematic friction coefficient between the
316 tablet and the die during compaction is dependent on the sliding speed of the powder
317 on the surface. Thanks to the methodology developed, we were able to prove that
318 this effect is obtained independently from the compaction phase used to make the
319 tablet, i.e. it represents an intrinsic property of the friction between a tablet and a die
320 lubricated using magnesium stearate.

321 As a consequence, friction between the tablet and the die is dependent on the
322 compaction kinematics used and also changes during a compaction experiment
323 because the sliding speed between the tablet/powder and the die is not constant
324 during the compaction. It is also not the same at all the points of the tablets. This
325 effect may have consequences in the stress evolution during compaction, especially
326 in terms of strain rate sensitivity.

327 **Acknowledgement**

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329 Recherche (ANR), under grant ANR-17-CE08-0015 (project CliCoPha).

330 **Legend to figures**

331 **Figure 1:** Schematic representation of the protocol for friction measurement. UP:
332 upper punch, LP: lower punch

Figure 2: Force on the upper punch during the pushing of the tablet in the die for three different punch speeds.

Figure 3: Evolution of the force needed to move the tablet in the die (F_p) as a function of the upper punch speed for the four products. First compression (a) 150MPa and (b) 250MPa.

Figure 4: evolution of the die wall pressure as a function of the force on the upper punch. First compression (a) 150MPa and (b) 250MPa.

Figure 5: Evolution of the friction coefficient as a function of the punch speed. First compression (a) 150MPa and (b) 250MPa.

Figure 6: Influence of the pressure used to make the tablet on the evolution of the friction coefficient with the speed. (a) SDMan, (b)CaSul, (c) GLac, (d) ACP.

Figure 7: SEM photographs of the band of tablet obtained under 150MPa: (a) GLac, (b) SDMan (c) CaSul and(d) ACP.

Figure 8: Photograph of the band of an ACP tablet obtained under 150MPa.

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