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Flowability of Pharmaceutical Powders: Impact of Humidity

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1. Introduction

Surrounding conditions impact the behavior of many powders. Depending on the geographical location, bulk solids exhibit differing flowability due to different environmental conditions, e.g. Caracas (30°C, 80 %rH) vs. Boulder/Colorado (25°C, 35 %rH). The powder adjusts dynamically to its environmental condition until reaching saturation at a certain point (1). Besides that, many powders show a "sticky point". At this point, a significant change in flow and adhesion behavior has been reported (2).

Granular media generally tend to increase their cohesive character when surrounded by moisture. This is attributed to several mechanisms whereas formation of adhesive films on the grain surface, liquid bridging between the grains, or if the cohesive behavior decreases, a "lubrication" of the interstitials between grains are considered as the main underlying mechanisms (3).

The investigated powder is a pharmaceutical cold and flu hot drink which comprises high amounts of vitamin C, citric acid, sugar and pharmaceutically active materials. The powder exhibits a tendency to cake and time consolidate. This is most likely due to a partial dissolution in condensed water as Groen et al. suggests for a pure citric acid (2). This partial dissolution is desired as the powder is supposed to dissolve in water. As the investigated powder contains a large amount of citric acid as well, a similar "sticky point" is expected. However, this property results in the forming of knots and clumps which is problematic for storage and processing.

Thus, the aim of the presented work is to investigate changes in the powder's flowability behavior after exposure to different relative humidity levels as well as to investigate the difference between consolidated and non-consolidated samples with the help of powder shear cell measurements.

2. Methods

In order to investigate the flowability of the pharmaceutical powder at different environmental conditions, its flowability coefficient ff_c is determined via shear cell measurements.

This powder characterization method is based on the work of Jenike (4). It was used for hopper bin and silo design (1). It helps to simply judge the flow/non-flow of a silo, hopper or bin.

Shear cell measurements generally consist of consecutive preshear and shear-to-failure intervals. During these, different normal stress levels are applied while the powder is sheared. This method yields certain powder characteristics such as a powder's unconfined yield strength σ_c (the "strength" of a powder) and the major principle stress σ_1 (the total applied stress on a powder, taking into account normal stress and shear stress) among others. These two magnitudes are used for the calculation of the flowability coefficient $ff_c = \sigma_1 / \sigma_c$. The coefficient is applied to categorize a powder's flowability from non-flowing ($ff_c < 1$) to free-flowing ($ff_c > 10$). It is typically depicted in a flowability diagram as demonstrated in ***Figure 2***.

Additionally, to the flowability coefficient ff_c, characteristics such as the cohesion, the bulk density, the tensile strength, the effective angle of internal friction etc. are determined via shear cell

Figure 1 *Powder shear cell which is used for investigating the flowability of pharmaceutical powders under different environmental conditions.*

measurements. (see (4) for more details regarding shear cell measurements).

The applied normal stresses and environmental conditions for the shear cell measurements at which the powder's flowability coefficient ff_c is generated are given in Table I.

Table I *Overview of sample preparations and measurements carried out for testing the influence of humidity on flowability*

Sample preparation – environmental conditions	Sample measurement – preshear normal stresses
7 %rH (4h, 30°C)	3 kPa / 6 kPa / 9 kPa / 12 kPa
35 %rH (4h, 30°C)	3 kPa / 6 kPa / 9 kPa / 12 kPa
65 %rH (4h, 30°C)	3 kPa / 6 kPa / 9 kPa / 12 kPa
95 %rH (4h, 30°C)	3 kPa / 6 kPa / 9 kPa / 12 kPa

An Anton Paar Modular Compact Rheometer (MCR) equipped with a convection temperature device, a humidity generator and a powder shear cell as shown in ***Figure 1*** are used to conduct the shear measurements.

3. Results

Figure 1 shows the flowability coefficients ff_c in regard to the environmental conditions to which the powder was exposed. While the pharmaceutical powder exhibits a free flowing/easy flowing character when exposed to relative humidities of 7 %rH and 35 rH%, the powder becomes cohesive when exposed to 65 %rH. When the relative humidity is further increased to 95%, the powder becomes very cohesive for low stresses and eventually turns to not-flowing for increasing stresses. The powder behavior is then more solid-like. These results are in good agreement with the sticky point measurements from Groen et. al. (2) where the sticky point of citric acid was estimated at 55% rH (30°C).

4. Conclusions

The investigated pharmaceutical powder shows a change in its flowability in dependence of the surrounding humidity. Its free-flowing character changes with increasing relative humidity level until its flowability is entirely lost. The processing of such a powder is then no longer possible. This effect is mainly attributed to the contained citric acid being one of the main ingredients. Thus, flowability investigations of pharmaceutical powders containing citric acid are crucial to ensure impeccable processing.

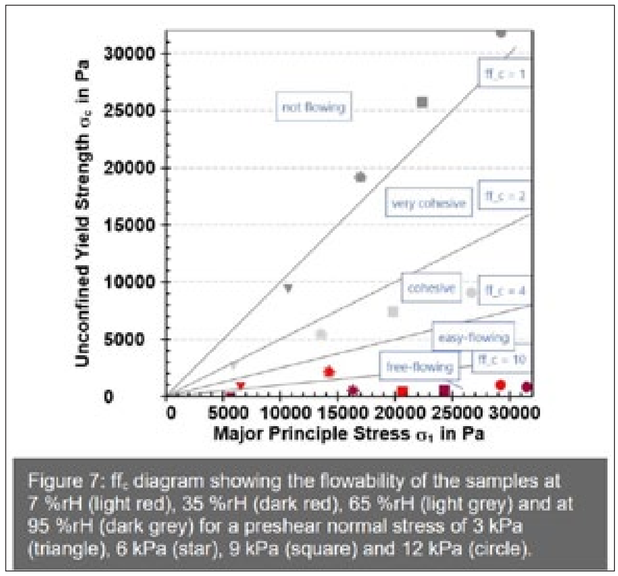

Figure 2 *Flowability coefficients ff_c of the samples at 7 %rH (light red), 35 %rH (dark red), 65 %rH (light grey) and at 95 %rH (dark grey) for a preshear normal stress of 3 kPa(triangle), 6 kPa (star), 9 kPa (square) and 12 kPa (circle) (1)*

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