AVALANCHING AND AERATION REGIONS FOR GLIDANTS

JEZERSKÁ Lucie[1], ZÁDRAPA František[1,2], ŽUROVEC David[1,2], ZEGZULKA Jiří[1,2]

[1]*VSB - Technical University of Ostrava, ENET Centre, Bulk Solid Center, Ostrava, Czech Republic, EU, jiri.zegzulka@vsb.cz*

[2]*VSB - Technical University of Ostrava, Faculty of Mechanical Engineering, Institute of Transportation, Ostrava, Czech Republic, EU*

Abstract

The characterisation of glidants is important for process control and the design of its processing conditions or handling devices in pharmaceutical industry. Frequently used, fine glidants as Aerosil®, Cab-O-Sil® and magnesium stearate are specific for characterisation of dynamic behaviour. These are complex powder systems, in particular because they contain varying amounts of air that can radically change (influence) their rheology or flow properties. The aim of this work is to describe the dynamic behaviour of the above mentioned sliding substances. Avalanching regions were determined using a dynamic angle of repose measurement method in a rotating transparent drum. Aeration regions and compressibility factors were measured by the FT4 Freeman Technology. The first results showed the change of avalanching regions of all the glidants with the rotation speed of the measuring drum. Tested glidants were included into the aeration regions and their dynamic behaviour was described.

Keywords: Glidants, rotating drum, dynamic angle of repose, dynamic behaviour

1. INTRODUCTION

Quality control not only of glidants that reduce friction between particles is a necessary part of manufacturing pharmaceutical processes. Characterisation of the basic properties is critical to ensure the efficiency of the process operations and consequently for customer satisfaction. A typical example for the quality control of glidants can be the particle size distribution or infrared spectrometry. Control of ready-made powder mixes is possible (in-process-control), for example, by measuring specific energy (FT4 Freeman), whose values determine the range for appropriate flow process behaviour or, conversely, reporting of problematic process [1]. To ensure trouble-free production, progressive innovation of the control of input raw materials is a necessity. The aim of this article is therefore to show simple ways of flow characterizing traditional glidants used in the pharmaceutical industry. The first method is the use of a simple rotating drum. Rotating drums have been extensively investigated in the past as they are an integral part of many industrial processes. The great majority of the published work on drums has focused on the various avalanching regions and phenomena that exist within the drum. In this paper we will focus on using the rotating drum as an instrument to determine the dynamic angle of repose as quality control of glidants and attempt to understand how this property may affect granular flow behaviour during the processes (mixing, transport etc.) [2]. The second considered method is an aeration test [3]. The presence or absence of air greatly influences the flow properties of the powders. With increasing air, the bulk density of the powder is reduced, the inter-particle spacing increases and the flow is easier. However, the course of this change varies for different pharmaceutical excipients and active substances and therefore it is necessary to make an aeration characterisation of each raw material in relation to the need for air [4]. Another possible characteristic in connection with the aeration behaviour of powders is the determination of the compression factor. Removal of air also significantly influences the properties of powders and always disturbs the flow. This phenomenon can be seen as a consequence of storage and transport of raw materials or as a side effect in the production process where vibration is used. Particles regroup and interlock. This will increase bond strength, shear resistance and, of course, powder flow. It is therefore clear that the energy required to dispense the powders varies according to the process point in which the raw material is. The difference in this energy for the fluidized and consolidated powder can be very profound. Avalanching and aeration characterisation methods for glidants show a relatively simple way to predict the flow properties of powders across the entire production process.

2. MATERIALS AND METHODS

2.1. Materials

For the experiments, three glidants commonly used in the pharmaceutical industry were tested. These are Aerosil® 380, Cab-O-Sil® M5 and magnesium stearate.

Aerosil®

Aerosil® 380 is hydrophilic fumed silica (SiO_2) with a specific surface area of 380 $m^2.g^{-1}$. Aerosil® 380 are small silica spheres covered with -OH group. This groups form hydrogen bonds with other aerosil particles, forming random network [5].

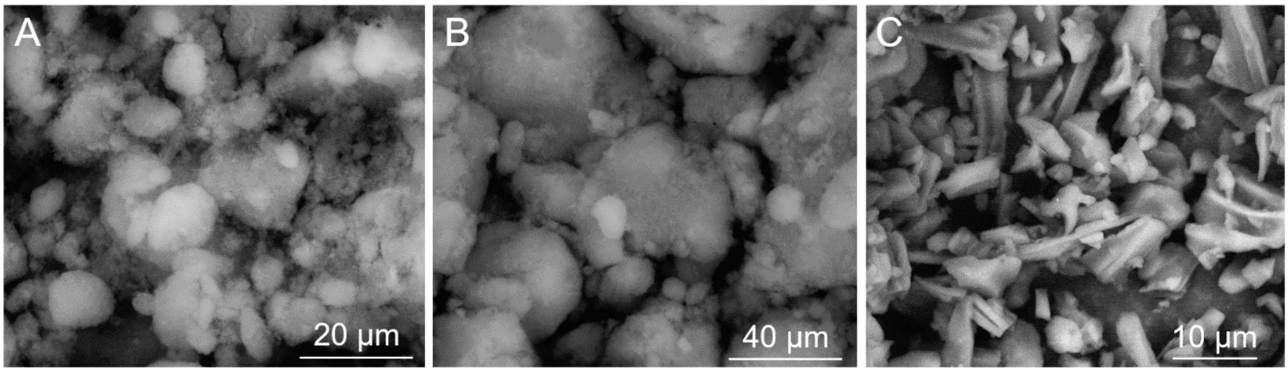

Figure 1 SEM images of Aerosil® 380 (A), Cab-O-Sil® (B) and magnesium stearate (C)

Cab-O-Sil®

Cab-O-Sil® is an untreated hydrophilic fumed silica (SiO_2) with medium surface with versatility, suggested for use in adhesives, coatings, cosmetics, foods, inks and pharmaceuticals applications. Cab-O-Sil® is white powder with density 2.2 $g.cm^{-3}$.

Magnesium stearate

Magnesium stearate is also known as octadecanoic acid and magnesium salt with chemical formula $C_{36}H_{70}Mg_4$. Magnesium stearate is the most common ingredient used in forming tablets because it's a lubricant. Known as a "flow agent". Just a minimal amount is required to coat a powder blend of virtually any active substance or supplement mixture.

2.2. Dynamic angle of repose

The dynamic angle of repose and dynamic behaviour of glidants was measured using a rotating drum of 140 mm diameter and 30 mm thick (**Figure 2**). Filling was 50 %. The rotation frequency ranged from 20 to 80 rpm. Thy dynamic angle of repose was evaluated five times for each material. The average value for each material was used.

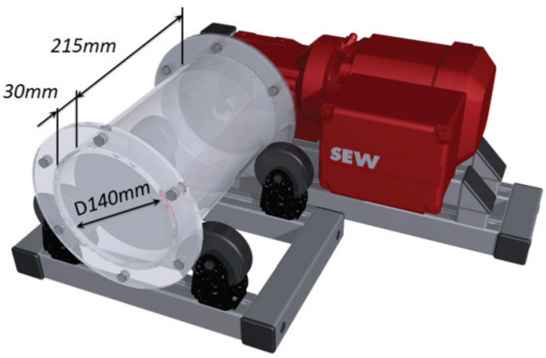

Figure 2 The rotating drum

2.3. Aeration and compressibility factor

During the aeration test performed on the powder rheometer FT4, the air is brought to the bottom part of the measuring cell while the whole column of the bulk material is aerated. It was observed how much air changes the flow properties of the test glidant by measuring the decreasing amount of flow energy (AE) that was recorded. The range in which flow energy is reduced is, of course, dependent on many physical properties of the powder (e.g. cohesion, particle shape, density). The aeration test was evaluated based on the Aeration Ratio (AR, equation 1) and Aeration Energy (AE) - flow energy during aeration.

$$Aeration\ Ratio, AR = \frac{Energy\ (Air\ Velocity\ 0)}{Energy\ (Air\ Velocity\ 10)} \tag{1}$$

Compressibility (or compressibility factor) was measured as the change in volume or density, respectively, depending on a normal load. The data obtained are quantified by expressing the percentage of compressibility for a normal load of 6, 8, 10 and 12 kPa. The FT4 Powder Rheometer was used.

3. RESULTS AND DISCUSSION

3.1. Avalanching regions and dynamic angle of repose

Avalanching regions were determined using the rotating drum method. This is not a standardized methodology, so the rotational speed of the drum in the selected range (20-80 rpm) was varied during the measurement, and the motion kinetics and the angle of repose of the test materials were observed on a video recording.

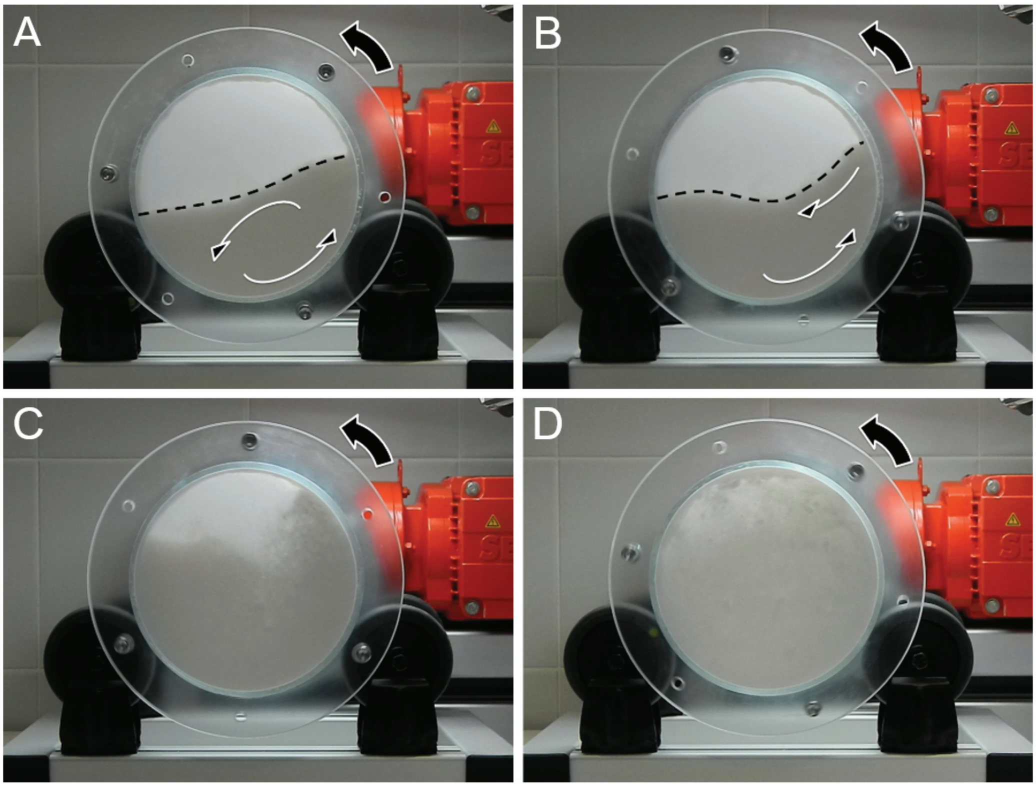

Figure 3 Dynamic behaviors of Cab-O-Sil® with increasing rotating frequency. A - rolling region, B - cascading region, C - swirled, D - dynamic swirled

During the experiment, it was observed that the increasing rotational speed of the drum changed the way that the mixture moved. The movement at lower frequencies for Aerosil® and Cab-O-Sil® was characterized by periodic changing of fine avalanches composed of fine powder particles. It was a rolling region, which, with the increasing frequency of rotation, switched to a cascade. A further increase in speed was unnecessary for Aerosil® and Cab-O-Sil®. Due to the significant amount of air in the two raw materials, they swirled and the dynamic angle of repose was no longer created. For the glidant magnesium stearate a totally reverse effect was observed. During measurement of the dynamic angle of repose, the size of the active zone changed.

The powder was theoretically divided into two zones. On the surface of the powder there was the so-called active zone across which the powder was spread, increased its volume and its rapid unidirectional flow was controlled by gravity. The other, static zone was beneath the surface (**Figure 4**).

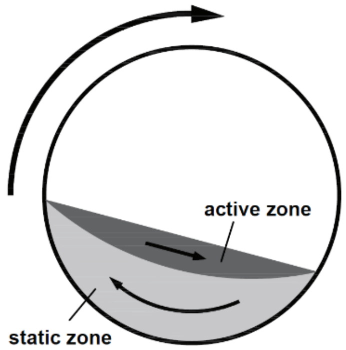

Figure 4 Active and static zone for magnesium stearate (scheme)

The powder was compacted in the static zone, the individual particles blocked each other, and the entire area moved as if it were in one piece. As the frequency increased, the active zone was enlarged and the static zone with only slight particle movement diminished, while in the active region there was particle movement. For the magnesium stearate, the movement was defined as a rolling for all tested drum rotational frequencies. **Table 1** shows the evaluated data. **Figure 5** shows the methodology of determination of the dynamic angle of repose for Aerosil® 380 (A), Cab-O-Sil® (B) and magnesium stearate (C).

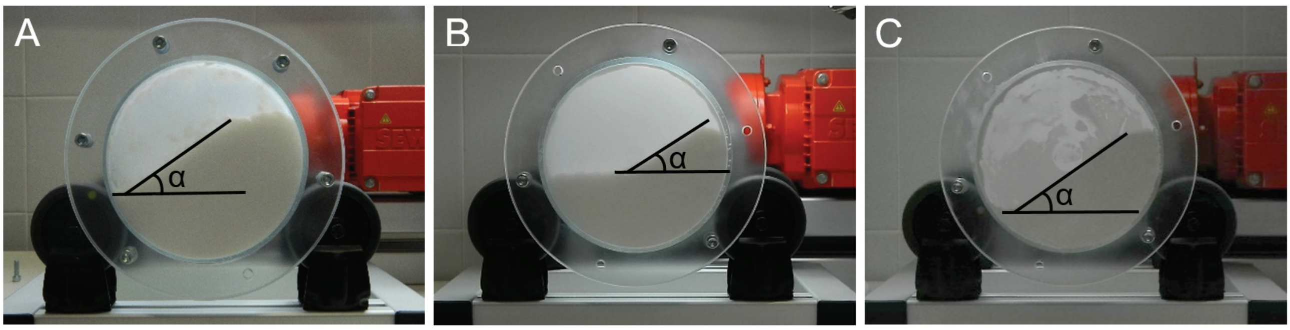

Figure 5 Angle of repose for Aerosil® 380 (A), Cab-O-Sil® (B) and magnesium stearate (C), 20 rpm

Table 1 Dynamic angle of repose of glidants

Rotating frequency, rpm	Dynamic angle of repose, °		
	Aerosil®	Cab-O-Sil®	Magnesium stearate
20	34 ± 1.5	30 ± 1.6	40 ± 1
40	35 ± 2	34 ± 2	34 ± 1.5
60	Undetectable	Undetectable	30 ± 1.6
80	Undetectable	Undetectable	16 ± 1.9

The dynamic angle of repose increases with increasing drum rotational frequency for Aerosil® 380 and Cab-O-Sil® (non-cohesive, well-flowing substances). For magnesium stearate it was determined that the dynamic angle of repose decreases with increasing rotational frequency. In the case of magnesium stearate the creation of the active zone was more pronounced and its size increased with the increasing frequency. It can be assumed that for powders with higher cohesion a higher rotational frequency is more suitable in the manufacturing process (mixing, granulation). Aerosil® 380 and Cab-O-Sil® have already been raised at the higher rotation speeds of the drum and it is therefore advisable to recommend lower speeds in the production process. For determination of the dynamic angle of repose of glidants the speed of 20 rpm is suitable. Dynamic angle of repose values are primarily influenced by the coefficient of friction and particle position in the rotating drum.

3.2. Aeration and compressibility factors

The resulting aeration test data for material sensitivity for aeration and compression factors are shown in **Table 2**.

Table 2 Aeration and compressibility factors

Parameters	Aerosil®	Cab-O-Sil®	Magnesium stearate
AR (Aeration Ratio), -	3.96	2.41	9.29
AE (Aeration Energy), mJ	4.38 ± 1.5	20.4 ± 2.2	5.38 ± 1.8
Compressibility factor (6 kPa), %	28.8 ± 0.7	27.0 ± 0.7	15.9 ± 0.1
Compressibility factor (8 kPa), %	31.1 ± 0.6	29.5 ± 0.7	16.9 ± 0.4
Compressibility factor (10 kPa), %	33.1 ± 0.6	31.6 ± 0.8	17.5 ± 0.6
Compressibility factor (12 kPa), %	34.9 ± 0.5	33.5 ± 0.8	18.1 ± 0.5

The evaluation of aeration regions is based on the AR values. It is true that powders of AR in the range of 2-20 can be aerated in a conventional manner. These are so-called “typical powders”. All the tested glidants fall into this category. This range is robust. The total flow energy for magnesium stearate was 50 mJ, for Aerosil® 17.4 mJ and for Cab-O-Sil® 49.2 mJ. The values indicate that the lower the flow energy, the easier the powder fluidization. The compression factor in the range of 6-12 kPa is always the lowest for the cohesive magnesium stearate. The measured value at 12 kPa was still lower than the compression factor of Aerosil® and Cab-O-Sil® already at 6 kPa. Magnesium stearate falls in the category of moderate compressibility powder. On the other hand, Cab-O-Sil® and Aerosil® are in the high compressibility powder group.

4. CONCLUSION

The aim of this article was to determine the avalanching and aeration regions for glidants as the basic characteristics for the necessary quality control of the input raw materials in the pharmaceutical industry. A simple methodology of determination of flow behaviour was shown for Aerosil®, Cab-O-Sil® and magnesium stearate. Aerosil® and Cab-O-Sil® were classified in rolling and cascading modes according to the dynamic angle of repose. At the frequency of 60 rpm both materials swirled in the rotating drum space. In the case of magnesium stearate it was determined that the dynamic angle of repose decreases with the increasing rotational frequency, and it was classified in the rolling mode. The glidants could not be defined in more detail depending on the aeration ratio and all three were assigned to the same aeration group. These powders are characterised by reduced total energy at increasing airflow rates. These glidants powders are moderately sensitive to aeration.

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