**Particules Surface Formulation Effects on Powder Rheology.**

Shirin Enferad1,2, Claire Gaiani2, Jeremy Petit2, Jennifer Burgain2, Véronique Falk3, Philippe Marchal3, Sergiy Antonyuk4, Fabian Krull4, Sébastien Kiesgen de Richter1, Mathieu Jenny1

*1 Université de Lorraine, LEMTA UMR 7563, Nancy, France; 2 Université de Lorraine, LIBio, F-54000 Nancy, France; 3 Université de Lorraine, CNRS, LRGP, F-54000 Nancy, France; 4 Institute of Particle Process Engineering, Technische Universität Kaiserslautern, Kaiserslautern, Germany*

*\*Corresponding author: shirin.enferad@univ-lorraine.fr*

**Highlights**

* Hydrophilic and hydrophobic formulation did not alter particle shape and size.
* Lactose-coated glass beads showed a surface roughness incensement.
* The hydrophilic glass bead showed highest, lactose coated bead lowest flowability.

**1. Introduction**

The study and comparison of formulated powders are a great interest as it can suggest solutions to improve the transport of real powders. The flowability of glass beads was largely investigated in previous studies [1-2]. The dependency of powders flowability to the moisture [3] and formulation [4] is known phenomenon. In fact, particle–particle interactions determine the macroscopic behavior of the powder. Powder flowability is expected to decrease due to liquid bridge formation between two particles dependently to the humidity [5]. Moisture sensitivity depends on hydrophilic and hydrophobic treatments. Furthermore, the friction between particles in granular materials is known to influence particles flowability [6]. By modifying the roughness of the surface, lactose coating may modify friction between particles. The surface roughness is comparable to the one of the particles obtained by lactose agglomeration (having a lactose core) but the Young modulus of particle core influence the collision effect during the flow. Therefore, aforementioned treatments may influence the interactions between particles, consequently powder flowability.

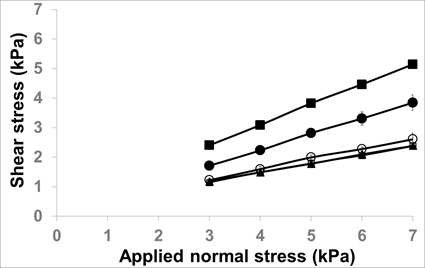
In this study, links between powder surface modification and rheology are investigated. For this purpose, glass beads with 100 µm mean size are employed. In order to study the influence of surface composition, various surface treatments leading to hydrophilic, hydrophobic and lactose-coating are performed on glass beads. Moreover, in order to investigate the influence of powder core composition, agglomerated lactose powders presenting similar mean particle size are also produced by high-shear wet granulation and characterized. Finally, the flowability of powders are studied and compared by performing tests with a FT4 powder rheometer (Freeman Technology) [7]. Furthermore, the TA Instruments AR 2000 Advanced Rheometer is in use to obtain the flow curves of powders. At the final, obtained results from both apparatuses will be compared to make links between the apparent viscosity of the powder depending on its formulation and the flowability measurements provided by FT4 rheometer.

**2. Methods**

The particle size distributions are determined by laser granulometry with a Mastersizer 3000 supplied with an Aero S dry dispersion unit. FT4 powder rheometer is used in order to study powders flowability. In this paper, stability and rotational shear cell tests are performed on the powders. Stability test intends to evaluate powder flowability in a low-stress environment, whereas the rotational shear cell test is devoted to analysis of powder flow properties in high-stress conditions. Moreover, TA Instruments AR 2000 Advanced Rheometer is implemented with the objective of studying the rheology of powders. A cylindrical Couette geometry with a vane is used to insure that the powder does not slip on the inner cylinder walls. The angular velocity of the vane is imposed and the corresponding torque is measured by the rheometer. The torque time evolution monitoring ensures that a steady-state flow is established for each value of the rotational speed. Thus, the relation between the shear stress and the shear rate in permanent regime can be determined.

**3. Results and discussion**

Fig 1 shows the evolution of shear stress recorded at variable decreasing normal stress from 7 to 3 kPa for each sample. At the beginning of each test 9 kPa of pre-shear stress applied by FT4 in order to reach the critically consolidate state. In general, the higher the curve on the diagram, the poorer the flow properties [8]; therefore, according to this criterion, investigated powders can be ranked according to their flowability in shear stress conditions: hydrophilic glass beads ≈ control glass beads > hydrophobic glass beads> lactose-coated glass beads> agglomerated lactose powders.



**Control glass beads**

**Hydrophilic glass beads**

**Hydrophobic glass beads**

**Lactose coated glass beads**

**Agglomerated lactose**



**Figure 1.** Evolution of shear stress with applied

normal stress after pre-shear at 9 kPa applied on

100 µm powder samples. Error bars represent st

-andard errors; some were not visible as their size

was inferior to the marker size.

**4. Conclusions**

The size distribution measurement of particles after formulation evidenced that agglomerated lactose powders and lactose coated glass beads presented similar mean particle size but a wider size distribution than the rest of powders. Based on shear cell test of FT4 rheometer, hydrophilic and lactose coated glass beads were more and less flowable powders, respectively. The result of TA Instruments AR 2000 Advanced Rheometer (which is in progress) will be compared with the FT4 results. Besides, the microscale properties of powders will be studied by single particle impact and nanoindentation tests to determine the influence of the surface modification on the deformation and adhesion properties of particles. Also, links between flowability, rheometry and microscale properties of particle-particle interactions will be analyzed. Concerning nanoindentation tests low inter-particle friction and low elasticity expected to lead to good flowability.

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