Meeting Minutes – IFPRI Powder Flow Project

Location: CPE, FEPS, University of Surrey Date: 14th January 2019 Time: 10:00 AM– 12:00 PM

Present: Marty Murtagh, Navin Venugopal, Tim Freeman, Colin Hare, Azza Mahmoud **Apologies:** Charley Wu

Meeting Objectives

- Updates on progress
- Discussion on next steps

Matters raised

- Measured hardness by vertical consolidation of DT51 titania has been found to be higher for the FT4 method than the Instron method. This may be explained by the difference in bed diameter between the two techniques.
 - ACTION: Azza will investigate the effect of bed diameter on the powder bed hardness (DT51), of vertically consolidated beds using the Instron.
- Constraint factor has been shown to vary with major principal stress for DT51. This may be due to the unconfined yield stress being determined by a linear fit to the yield locus
 - ACTION: Azza will apply the Warren spring curve fitting on the generated yield locus of shear cell tests (DT51) and apply indentation measurements based on the determined principal stresses.
- Generation of a flat surface following critical consolidation has proven challenging. The vacuum suction and the splitting after shearing (in FT4 shear cell) methods will not be explored further.
 - ACTION: Azza will carry out indentation onto critically consolidated beds onto sheared bed using Peschl shear cell (shear zone occurs at split plane).
- Generation of a reproducible packing state may be possible with vertical consolidation by first conditioning the powder using the wire attachment of the 1 ml FT4 shear cell, followed by vertical consolidation.

- ACTION: Azza to assess the reproducibility of packing fraction and hardness measurement with this method.
- Agreement on using the new Schulze shear cell tester (RST-XS.s low-stress cell) with interest in showing the raw time data.
 - o ACTION: Colin to communicate with Dietmar Schulze to provide the software update
- Shearing at constant volume with fixed lid position throughout the test (by FT4 shear cell) will be explored.
 - ACTION: Azza to communicate with Katrina Brockbank to obtain test procedures, and carry out tests with this method.
- The following materials will be used to compare measurements in the RST.XS.s, RST-01.pc, FT4 shear cell and potentially the Peschl shear cell, under low (≤ 1 kPa) normal stresses. Furthermore, testing of optimal gain settings in the FT4 shear cell will be carried out by Freeman Technology
 - \circ $\;$ Limestone; the certified reference powder used for shear cell calibration
 - Microcrystalline cellulose (MCC) as an easy flowing pharmaceutical excipient, with the care of humidity controlling.
 - It was noted that this material is prone to moisture uptake, so care in conditioning is required
 - Titania powder (R104 or DT51 grade) as a very cohesive fine powder with nanoparticle size level.
 - \circ $\,$ Pea Protein, which is a food powder with strong slip-stick behaviour.
 - Glass frit (unmilled), as a high-density powder to allow the investigation of significance of the weight of the powder within the shear head vanes on the shear plane, at low normal stresses.
 - ACTION: Azza to find out if the Peschl shear cell is capable of operating at such low normal stresses
 - ACTION: Azza to order the remaining powders and send a 1 litre sample of each material to Tim.
- Material characterisation was agreed to include: particle size, particle shape, particle (true) density, Envelope (agglomerate) density, Surface energy
 - It was noted that envelope density will vary with stress level. ACTION: Azza and Colin should consider how to characterise this under the stress level of interest

Action Items

Azza will:

- Investigate the effect of bed diameter on the powder bed hardness (DT51), of vertically consolidated beds using the Instron.
- Apply the Warren spring curve fitting on the generated yield locus of shear cell tests (DT51) and apply indentation measurements based on the determined principal stresses.
- Carry out indentation onto critically consolidated beds onto sheared bed using Peschl shear cell (shear zone occurs at split plane).
- Assess the reproducibility of packing fraction and hardness measurement with the wireconditioning vertical consolidation method.
- Communicate with Katrina Brockbank to obtain test procedures for shearing at constant volume, and carry out tests with this method.
- Find out if the Peschl shear cell is capable of operating at such low normal stresses
- Order the remaining powders and send a 1 litre sample of each material to Tim.

Azza and Colin will:

• Consider how to characterise envelope density under the stress level of interest

Colin will:

• Communicate with Dietmar Schulze to provide the software update to provide normal stress vs time data