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# Evaluation of Powder Flow Prediction by Ring Shear Testing at Low Consolidation Stresses

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SUMMARY

The development of XS-Lr0 low-stress shear cell by Dietmar Schulze (model RST-XS.s) has confirmed the necessity of assessing powder flow at low consolidation stresses for improved powder flow prediction (Søgaard et al., 2014). This project assessed different powder flow properties at low consolidation stresses via stress walk and discrete shear tests. Previous methodology by Sogaard et al, 2014 was investigated and further developed using different pharmaceutical materials at a wide range of consolidation stresses (0.25 – 4.00 kPa). A polynomial function best described the relationship between consolidation stress and unconfined yield strength, with results being material dependent. The lowest pre-consolidation stress observed for reliable measurements was 0.25 kPa. The findings emphasise the need to refine current methodologies to measure shear stresses at low consolidation stresses to improve powder flow prediction.



## INTRODUCTION

Several powder-based unit operations within the pharmaceutical industry require adequate flow at a broad range of stresses. The multiplex nature of powder, depending on both intrinsic and systemic properties, obscures the development of a universally applicable powder characterisation technique. The universal shear-cell based techniques, with current linear regression prediction methods, inaccurately assess powder flow (Swize et al., 2019). Current lab-scale tests assess powder flow at high consolidation stresses (> 1 kPa) compared to manufacturing process conditions (<1 kPa) (Søgaard et al., 2014). XS-Lr0 low-stress shear cell was devised by Schulze Ring Shear Tester (RST-XS.S, Dr.-Ing. Dietmar Schulz, Germany) to bridge this gap. Sogaard et al, 2014 investigated the use of XS-Lr0 cell at consolidation stresses ranging from 0.25-8.00 kPa, demonstrating successful low stress powder flow measurement with a further need of improved methods. This project aimed to investigate the use of XS-Lr0 shear cell using different methodologies and pharmaceutical powders.

## MATERIALS AND METHODS

Avicel PH102 (DuPont Nutrition USA), Paracetamol (Pfizer IM, Sandwich UK), and Paracetamol blends (Blend preparation using Turbula T2F mixer (Bachofen AG, Germany) for 10 minutes at 46 cycles/min: 10, 25 and 50% Paracetamol, 2:1 Avicel PH102 and Lactose Fast Flo 316 (Kerry, USA) with 1% Magnesium Stearate (SpecGX LLC, USA)) were tested using a Dietmar Schultze Ring shear tester for their powder flow properties, such as flow function, effective angle of internal friction, wall friction, and bulk density. Two test modes were used: 1) discrete shear test and 2) stress walk. Fresh bulk specimen was used for each yield locus for the discrete shear test while stress walk used the same bulk specimen for all yield loci. Samples were tested in duplicate under ambient conditions, i.e., 50% RH and 20 °C at normal stresses ($\sigma_{pre}$) ranging from 0.25-4.00 kPa, with increments at 25, 38, 51, 65, 25% of $\sigma_{pre}$. Linear and non-linear models were applied using Microsoft Excel to identify correlations.

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## RESULTS AND DISCUSSION

Both methods generated non-linear data within major consolidation stresses of 0.25-4.00 kPa (Figure 1). Flow functions at higher stresses expressed a linear relationship while lower stresses expressed an evident polynomial relationship (Figure 1). Coefficient determination ($R^2$) of other regression models is shown in Table 1.

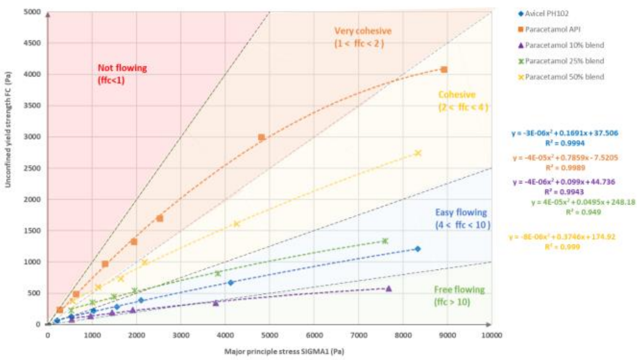


*Fig. 1. Flow functions of powders yielded from standard shear stress test.*

| | R2 value (FC) | | | |
|---|---|---|---|---|
| | **Linear** | **Logarithmic** | **Polynomial** | **Best trend** |
| **Avicel PH102** | 0.9998 | 0.9706 | 0.9998 | Polynomial |
| **Paracetamol API** | 0.9542 | 0.9984 | 1.0000 | Polynomial |
| **Paracetamol 10%** | 1.0000 | 0.9965 | 1.0000 | Polynomial/Linear |
| **Paracetamol 25%** | 0.8371 | 0.7719 | 1.0000 | Polynomial |
| **Paracetamol 50%** | 0.9999 | 0.9703 | 1.0000 | Polynomial |

***Table 1.*** *Description of flow functions via different coefficient determination ($R^2$).*

However, stress walk generated lower stresses and flow functions due to shear deformation effect (Figure 2), which is continuous shear displacement of powder with increasing applied stresses (Swize et al., 2019). Overall, results demonstrated stress walk as a successful time and material sparing method if the shear deformation effect is accounted for.

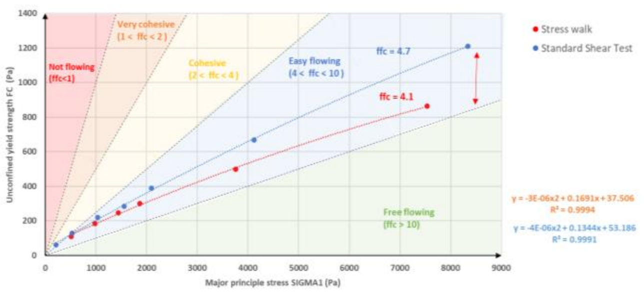


***Fig. 2.*** *Flow functions of Avicel PH102 yielded from discrete shear stress test (at 0.25 kPa) vs stress walk methodology.*

Consequently, the use of both polynomial and linear relationship predictive models at low consolidation stresses are unrepresentative (Table 2).

| | SIGMA 1 | Unconfined yield strength (FC) at 0.25 kPa | | |
|---|---|---|---|---|
| | | Experimental data | Predicted data | |
| | | | Linear | Polynomial |
| Avicel PH102 | 532 | 129 | 189 | 166 |
| Paracetamol API | 637 | 488 | 1202 | 240 |
| Paracetamol 10% | 539 | 82 | 154 | 152 |
| Paracetamol 25% | 526 | 224 | 106 | 332 |
| Paracetamol 50% | 559 | 375 | 551 | 516 |

***Table 2.*** *Powder flow comparison between experimental data and predicted data.*

Bulk density increased with increasing consolidation stress with changes in bulk density most prominent at lower consolidation stresses (<2 kPa). Cohesive powders (e.g.: Paracetamol) demonstrated largest increase in bulk density with increasing consolidation stress, indicating their poor flow.

## CONCLUSIONS

In conclusion, powder flow is non-linearly affected by applied consolidation stresses. Evaluation of the methods using multicomponent system (i.e., blends) was shown to be successful for industrial application. XS-Lr0 low-stress shear cell methodology is successful in assessing powder flow at a stress range of 0.25-4.00 kPa, with comparable results using stress walk methods. Further work is required to understand the nature of the polynomial relationship, shear deformation effect, effects of further pharmaceutical materials and to determine the edge of failure limits for <0.25 kPa consolidation stresses.

## ACKNOWLEDGEMENTS

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