



The build-up of detergent agglomerates in an auger filler

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ABSTRACT

Powder build-up formation is a fouling process in which a small fraction of a powder becomes a coherent, non-porous, film adhered to a surface known as build-up. Build-up tends to form in auger fillers where particles undergo plastic deformation, leading to the formation of build-up on the internal surface of the tube within which the auger is housed. To develop an understanding regarding the process by which build-up is formed, a combination of macro scale auger filling experiments and laboratory scale characterisation of the virgin powder has been undertaken. This involved a comparison of results obtained via uniaxial compaction followed by tablet diametric compressions, with the results of macro scale auger filler experiments. From this work, it has been determined that the build-up forming powders have Kawakita b^{-1} parameters of 0.5 MPa or less, and form disc-shaped tablets with strengths <0.5 MPa following compaction to 58 MPa.

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1. Introduction

The build-up of powders within powder handling processes is a significant problem for industry both in auger fillers and in items of equipment such as rotary valves, screw conveyors, tablet presses and roller compactors. The formation of build-up has the potential to lead to equipment downtime for cleaning, equipment breakdowns and cosmetic defects e.g. the presence of unwanted agglomerates. In some cases, these issues become so severe that it is not economically feasible to operate an item of equipment with a specific powder formulation [1].

Literature regarding has been published with respect to the adhesion of powder to the punches of tablet presses [2–9], and the rollers of roller compactors [10,11]. However, it has not been possible to find literature regarding the adhesion of powders to the internal surfaces of auger fillers, screw feeders and other similar items of equipment. This paper forms the first publication with respect to the formation of build-up in auger fillers and gives guidance regarding the powder characteristics which indicate build-up will be formed as a powder passes through an auger filler.

1.1. Build-up as form of particle agglomeration

The formation of build-up in auger fillers is a fouling process involving the transformation of solid discrete particles to a coherent non-porous film adhered to the tube's internal surface, see Fig. 1. As can be

seen from Figs. 2 and 3, the formation of build-up can be thought of as a pressure driven agglomeration process, involving plastic deformation of solid particles into a coherent solid mass. To gain understanding of the mechanisms by which build-up is generated literature related to pressure drive agglomeration processes will be discussed in conjunction with empirical observations.

From Figs. 2 and 3 the conversion of detergent agglomerate particles to build-up involves plastic deformation. In processes, such as tableting, plastic deformation leads to the creation of increased contact area which leads to increasing adhesive forces [12]. Rumpf et al. [13] stated that this process can be represented by Eq. (1) where the adhesive force acting between two plastic deforming spheres is proportional to the applied force and the ratio of the van der Waals pressure to the plastic yield pressure of the sphere.

$$F \approx \frac{p_{vdW}}{p_{pl}} F_a \quad (1)$$

where F is adhesive force, F_a is an applied force, p_{pl} is plastic yield pressure of the sphere and p_{vdW} is van der Waals pressure which can be calculated

$$p_{vdW} = \frac{h\omega}{8\pi^2 h_s^3} \quad (2)$$

where $h\omega$ is the Lifshitz–van der Waals constant with units of Joules and h_s is the separation distance and the adhesive force acting between particles prior to onset of plastic deformation is negligible.

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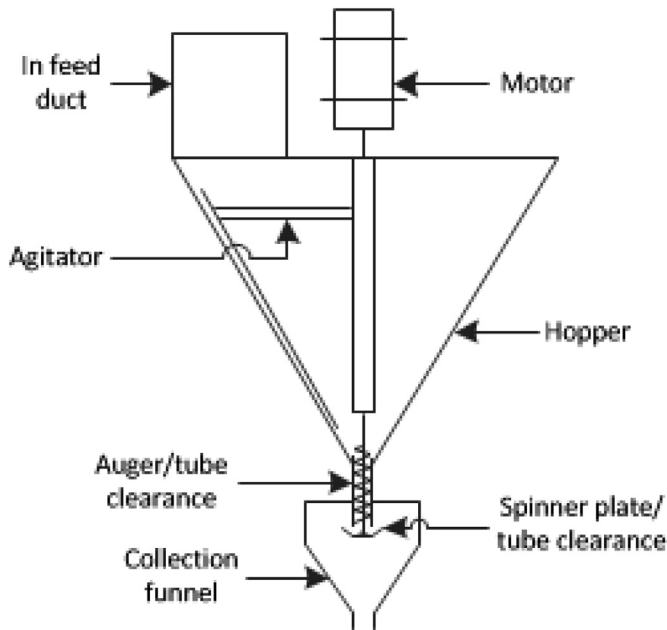


Fig. 1. Schematic of a typical auger filler.

From Eq. (1) it can be concluded that as van der Waals pressure increases as the separation distance h_s reduces, materials with a low plastic yield pressure will tend to give rise to large values of adhesive force F per unit of applied force F_a .

This then leads to the formation of a hypothesis that build-up is formed via a pressure driven agglomeration process similar to tableting and roller compaction, where particles of low plastic yield stress will be more likely to form build-up.

1.2. Measurement of particle plastic yield stress

The measurement of particle plastic yield stress can be achieved via the compression of single particles. However, as was stated by Adams and McKeown [14], powders are often comprised of particles with a wide distribution of mechanical properties which leads to the need to compress many particles to gain a statistically significant result, which makes this approach impractical for many industrially manufactured powders. To enable the generation of results within a reasonable time frame, an alternative technique is required. Generally, the method by which this is achieved is uniaxial confined compaction of a bulk powder. This involves confining a powder bed within a cylindrical die and measuring a force applied to the top surface of the bed, as a function of top punch displacement. A lumped parameter model is then applied to

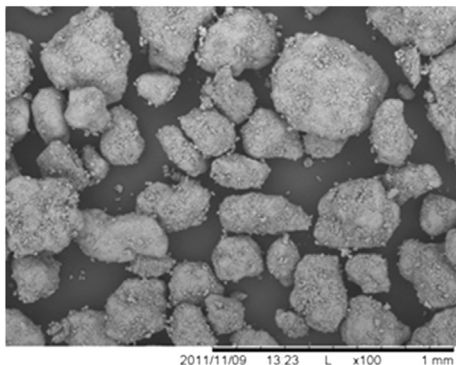


Fig. 2. An SEM micrograph of virgin detergent agglomerates.

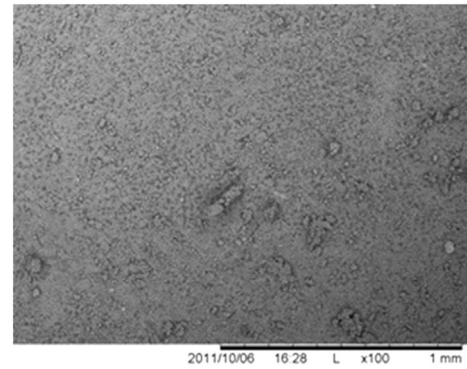


Fig. 3. An SEM micrograph of auger filler build-up formed from detergent agglomerates.

the experimental data to enable the calculation of parameters informative of the particles failure stress.

1.2.1. The Kawakita model

The Kawakita model, Eq. (3), is a widely used lumped parameter model, derived from the assumption that during compression, the product of the column height and an applied pressure are constant. The equation contains two constants, a and b . The a parameter is related to the voidage in the initial powder bed, while b is related to the strength of the individual particles within the compaction die and has units of reciprocal stress [14].

$$\frac{\sigma}{\varepsilon} = \frac{1}{ab} + \frac{\sigma}{a} \quad (3)$$

where σ is the applied stress, ε is strain defined via Eq. (4).

$$\varepsilon = \frac{h_i - h_\sigma}{h_i} \quad (4)$$

where h_i is the initial bed height and h_σ is the bed height for a given value of σ .

1.2.2. Tablet strength measurements

To aid in the characterisation of detergent agglomerates with differing mechanical properties and tendencies to form build-up during auger filling, measurements of tablet strength have been made via diametric compression. This method of characterisation induces a tensile stress acting in the transverse direction versus the compressive applied stress. The diametrical compressive test has become widespread in its use partly because it enables the use of a simple disc shaped specimen, in this case a tablet, to measure the force required to initiate tensile failure of a material [15]. To avoid effects associated with tablet size and shape Eq. (5) has been used to remove the influence of tablet size and shape [16].

$$\text{Tablet strength} = \frac{2F_t}{\pi D_t t} \quad (5)$$

where F_t is tablet breaking force, D_t is the diameter of the tablet and t is the thickness.

2. Materials and method

2.1. Materials

A series of surfactant containing granulated materials have been used in this study, these materials were supplied by Procter and Gamble and were manufactured at either commercial or pilot plant scale. The materials are listed in Table 1 with their surfactant binder contents, it should be noted that the type of surfactant binder and primary

Table 1
Surfactant contents for materials used in auger filler build-up study.

Powder	Surfactant content % w/w
SD1	18
AG1 Batches 1 & 2	24
AG2 Batches 1 & 2	26
AG2 Batches 3 & 4	24
AG3 Batches 1 & 2	24
AG4 Batch 1	25
AG4 Batch 2	21
AG5	45
AG6	25
AG7	23

particles varies between the materials, however, for commercial reasons it is not possible to state the full formulation of each material.

SD1 was manufactured via spray drying, AG6 which was manufactured by granulation in a batch paddle mixer all other materials were manufactured by continuous high shear granulation.

2.2. Methods

The following study utilises both macro and laboratory scale methods. Macro scale experiments were conducted to determine if a material forms build-up as it passes through an auger filler. Laboratory scale methods were used to determine if the powders tendency to form build can be predicted.

2.2.1. Macro scale auger filler experiments

Auger filler build-up experiments were performed using the methodology described by Hewitt [1]. Where powder is passed through an auger filler containing an auger with a diameter of 24.5 mm housed within tube with an internal diameter of 25.1 mm, resulting in a clearance of 0.3 mm, for a minimum of 1000 fills (3000 revolutions) or until the formation of build-up led to the auger filler tripping due to a high electrical current drawn by its drive. The Auger Filler used in these experiments was a semi-automatic filler manufactured by ALLFILL International Ltd. and was of a clutch break design (model number; S10, Serial number: 10503).

Auger filler build-up experiments the filler setup was as follows:

- 3 revolutions per fill
- Auger speed: 840 rpm in the clockwise direction.
- One second pause between fills
- Spinner plate/tube clearance: 8 mm
- #16 auger tooling:
 - Auger diameter: 24.5 mm
 - Straight funnel internal diameter: 25.1 mm
 - Auger tube wall clearance: 0.3 mm
 - Pitch: 31.7 mm
- Agitator type: flat blade running counter clock-wise during fills only at 20 rpm.

The purpose of the agitator is to feed powder into the auger and prevent rat holing in the hopper. As well as the auger, the agitator tends to build-up and so to minimize this issue the running of the agitator was limited to during fills only.

The test method employed was as follows:

- Start the filling of powder from the auger filler.
- At Intervals of 2 to 4 min filling was paused and the torque required to turn the auger manually was recorded.
- To reduce the mass of powder required per experiment, filled powder was returned to the auger filler's hopper at intervals of approximately 4 min.

Manual measurements of the torque were performed by turning the auger using a deflecting needle torque wrench (Manufacturer;

Torqueleader, Model number; ADS 25, Full measurement range: 0 to 27 Nm).

2.2.2. Moisture measurements

To assess the level of moisture, each powder was tested with an Infra-Red heat balance (Mettler Toledo HB43-S Halogen). The balance heated 2.0 ± 0.1 g of powder to 160 °C for 5 min. The method used for this measurement is the standard Procter and Gamble procedure used globally throughout Procter and Gamble laundry detergent business and described by Hassall [17]. This method has been developed and used over many years, a temperature of 160 °C is used because it is above that required to evaporate both free and chemical bound moisture within the powder. 5 min is long enough to ensure the entire sample has been heated to 160 °C and that the moisture is eliminated from the sample [17].

2.2.3. Uniaxial compaction

Uniaxial compactions were performed within a cylindrical close-fitting compression tableting die with an internal diameter of 31 mm. The die was placed between the platens of an Instron 4469 Universal Testing machine fitted with a 50 kN load cell with a resolution of ± 1.35 N. For each compaction a mass of powder was weighed accurately using an analytical balance (Manufacturer: AND, Model HF-3000G). For each compaction 4.00 ± 0.03 g of powder was transferred to the compaction die, the die was then gently shaken to create an even powder bed and the punch was lowered slowly to avoid damaging the agglomerate particles prior to compaction. The powder was then compacted to a force of 45 kN at a speed of 5 mm/min, the initial bed height was determined from the punch position at the start of loading. Additional details regarding this methodology can be found in Hewitt [1].

2.2.4. Diametric tablet compression

Following each uniaxial compression, the tablet formed was removed from the die and its thickness was measured using callipers to an accuracy of ± 0.01 mm. Values of tablet strength were then determined using a Vankell VK200 tablet hardness tester which measures the strength of tablets via diametric compression of horizontally positioned tablets. This strength is then converted to units of pressure using Eq. (5).

3. Results and discussion

3.1. Moisture and particle size data

Moisture content and particle size data for materials used in this study are presented in Table 2, moisture contents are the mean of five repeats quoted in conjunctions with standard errors. A standard error of 0.0 indicates that the standard error was <0.1 . Geometric mean particle sizes were measured via gravimetric sieving with spans calculated using Eq. (6).

$$\text{Span} = \frac{D_{90} - D_{10}}{D_{50}} \quad (6)$$

3.2. Repeats of auger filler build-up experiments

Figs. 4 and 5 show repeats of auger filler experiments demonstrating the repeatability of this auger filler experimental method, demonstrating that:

- The initial and final torque values are repeatable when measured to the nearest 0.5 Nm [1].
- The increase in torque associated with the onset of build-up formation is at the same point in time. This indicates that the uncertainty in the measurement of time associated with the increase in torque is primarily a result of the measurement frequency [1].

Table 2
Moisture content and agglomerate particle size data.

Virgin Powder	Batch	% w/w Moisture content	Geometric mean particle size (µm)	Span
SD1	NA	2.9±0.0	770	2.2
AG1	Batch1	6.1±0.1	368	1.6
AG1	Batch2	5.3±0.0	439	1.8
AG2	Batch1	4.7±0.0	486	1.6
AG2	Batch2	4.8±0.0	772	1.5
AG2	Batch3	5.0±0.0	596	1.9
AG2	Batch4	4.1±0.1	384	2.0
AG3	Batch1	10.5±0.2	347	3.6
AG3	Batch2	5.8±0.1	495	1.6
AG4	Batch1	6.8±0.2	538	1.5
AG4	Batch2	7.0±0.2	490	1.8
AG5	NA	2.5±0.0	443	1.3
AG6	NA	1.9±0.1	552	1.3
AG7	NA	8.5±0.0	487	1.4

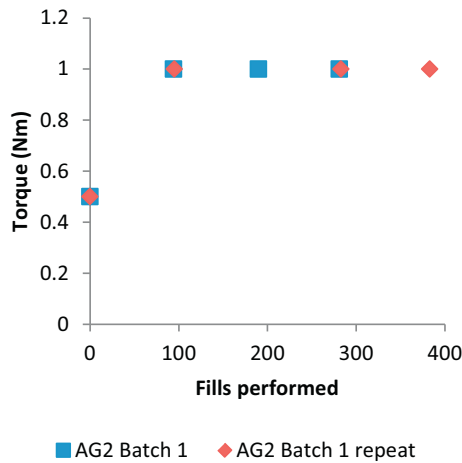


Fig. 4. Auger filler repeat experiments for AG2 Batch 1.

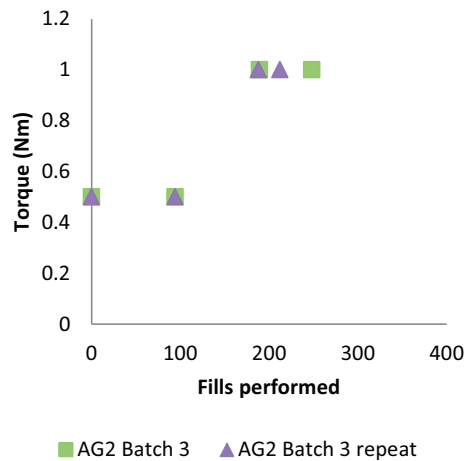


Fig. 5. Auger filler repeat experiments for AG2 Batch 3.

3.3. Analysis of results derived from uniaxial compaction experiments

Each of the powders listed in Table 2 were characterised via uniaxial compression, in each case stress strain curves were plotted (see Figs. 6, 8, 10 and 12) and subsequently converted to Kawakita plots (see Figs. 7, 9, 11 and 13). The Kawakita plots were then inspected to identify

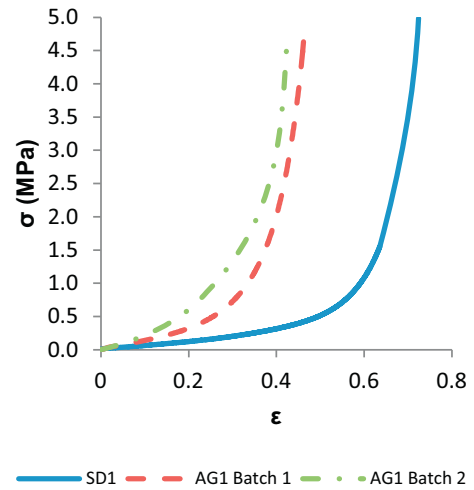


Fig. 6. Stress strain plots for SD1, AG2 Batch 1, AG2 Batch 2.

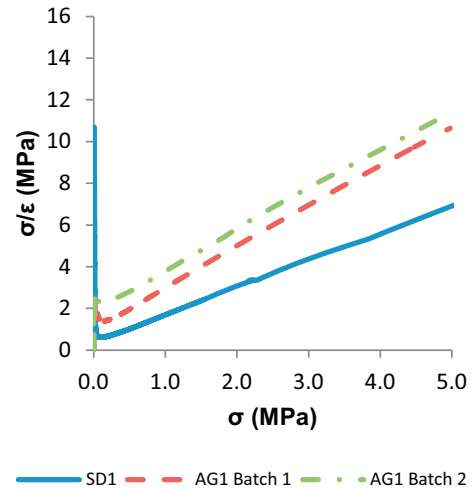


Fig. 7. Kawakita plots for SD1, AG2 Batch 1, AG2 Batch 2.

regions within which the Kawakita model gave a good fit to the experimental data which was confirmed by R^2 values being >0.99 in all cases. Table 3 gives the identified regions; curvature in the early part of the compaction process was avoided by excluding data below 0.5 MPa from the analysis. The exception to this was AG7, where it was necessary to exclude data below 5.0 MPa due to the large degree of curvature observed in this region. This may have arisen from a greater degree of fracture or elastic deformation in the early portion of the compaction of AG7 relative to the other detergent agglomerate powders tested.

Stress ranges are associated with regions of the stress strain plots where substantial changes in strain per unit stress occur and much of the plastic deformation may therefore be expected to occur. Therefore, it is reasonable to assume that application of the Kawakita model to data with these ranges will yield b^{-1} parameters informative of the detergent agglomerates plastic yielding behaviour.

3.4. An operating space model for auger filler build-up

Table 4 and Fig. 14 give Kawakita parameters, tablet strengths and agitator build-up observations. Kawakita parameters and tablet strengths are the mean of at least three repeats and are quoted in conjunction with standard errors. During auger filler build-up experiments manual auger torque measurements were made at intervals of 90 to 180

Table 3
Force ranges used to in order to select data which is a good fit to the Kawakita model.

Powder	Batch	Stress Range Studied (MPa)
SD1	NA	0.5 to 5.0
AG1	Batch1	0.5 to 5.0
	Batch2	0.5 to 5.0
AG2	Batch1	0.5 to 2.5
	Batch2	0.5 to 2.5
	Batch3	0.5 to 2.5
	Batch4	0.5 to 5.0
AG3	Batch1	0.5 to 5.0
	Batch2	0.5 to 5.0
AG4	Batch1	0.5 to 5.0
	Batch2	0.5 to 5.0
AG5	NA	0.5 to 5.0
AG6	NA	0.5 to 2.5
AG7	NA	5.0 to 10.0

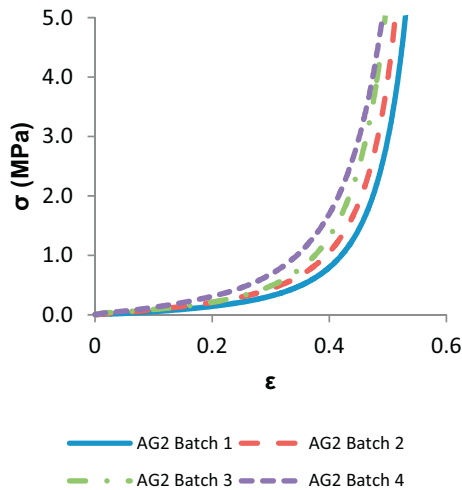


Fig. 8. Stress strain plots for AG2 Batches 1 to 4.

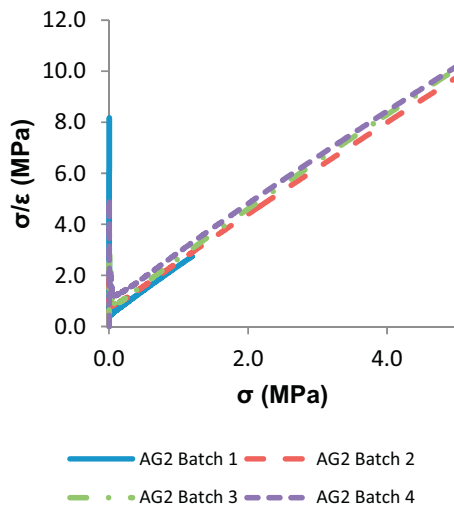


Fig. 9. Kawakita plots for AG2 Batches 1 to 4.

fills. Tube build-up onset is defined as the mean of, the number of fills associated with:

1. The last torque reading of 0.5 Nm
2. The first reading >0.5 Nm, typically 1.0 Nm.

The error quoted for tube build-up onset measurements is the uncertainty arising from the measurement frequency and not the accuracy of the individual measurements.

In many of the auger filler experiments build-up was observed on the low speed agitator in the auger filler's hopper on the forward-facing surface where no small clearance/gap exists (see Fig. 14). This suggests that this type of build-up was formed by a different mechanism to that observed on the tubes internal surface (see Fig. 15). This type of build-up is not a key focus of the investigation but has been noted for completeness.

In the case that no rise in torque was detected the experiment was continued for a minimum of 1000 fills and the tube was inspected at the end of the experiment to ensure build-up had not formed. The relevant powder was then classified as non-build-up forming, with respect to the tubes internal surface. Tablet strength and Kawakita b^{-1} parameters presented in Table 4 are plotted in Fig. 16, it can be seen that powders which form build-up on the tube's internal surface have both low b^{-1} parameters and low tablet strengths.

For agglomerated/granulated powders which compact via plastic deformation, the b^{-1} Kawakita parameter is related to the plastic yield stress of the agglomerate particles within the die [4]. It is proposed that the adhesive forces between agglomerate particles and the tube's internal surface are a function of the agglomerate particles plastic yield stress and for these materials the b^{-1} Kawakita parameter.

Build-up forming powders had low tablet strengths, potentially because their agglomerate particles were formed from soft plastic materials which agglomerated into a solid coherent mass during compaction. As these powders formed build-up and had low b^{-1} parameters this suggests that they are soft and plastic. Which suggests that that the forces holding the agglomerate particles together within their tablets are a function of their ability to undergo plastic deformation and ductile separation in preference to a brittle separation involving only elastic deformation. Eq. (7) proposed by Maugis and Pollock [18] describes ductile separation and predicts that in the case of a material of low yield stress and low plastic hardness reduced force per unit contact area will be required to achieve separation. This may explain the strong correlation between b^{-1} and tablet strength for build-up forming materials ($R^2 = 0.93$) shown in Fig. 16.

$$-F_d = P + 2\pi\omega_A R = \pi r^2 H \quad (7)$$

where F_d is the force require for ductile separation, P is the applied load, ω_A is the thermodynamic work of adhesion with units of Joules per meter squared, R particle radius, r is the contact radius, H is the plastic hardness which related to yield strength Y via Eq. (8) [18].

$$H = 3Y \quad (8)$$

As build-up was observed to be both coherent and adhered to the tube's surface, it is reasonable to assume that the formation of build-up will involve:

1. Adhesive contacts between particles and the tubes internal surface
2. Cohesive contacts between particles

Tablet strength measurements will reflect only the failure of cohesive contacts. However, in this study the tube surface was constant and the ductile failure of adhesive contacts between the stainless-steel tube and soft detergent agglomerates are likely to be dominated by the yielding of the softer material. Therefore, while it is possible that build-up formation could be prevented via modification of tubes internal surface, in this case it was reasonable to characterise only the soft detergent agglomerates.

Tube build-up onset is characterised as the point at which the presence of build-up on the tube surface leads to an initial rise in torque, indicating that build-up is able to provide a measurable resistance to the augers movement. Tube build-up onset and b^{-1} values presented

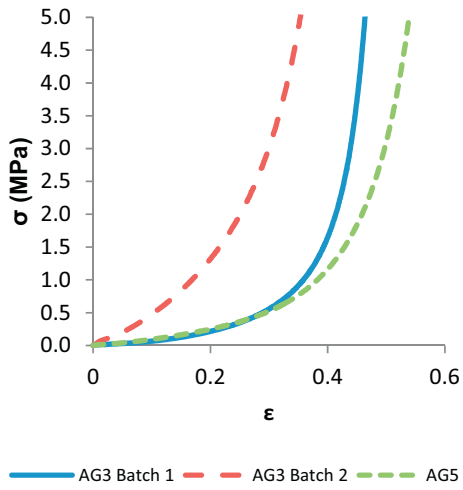


Fig. 10. Stress strain plots for AG3 Batches 1, 2 and AG5.

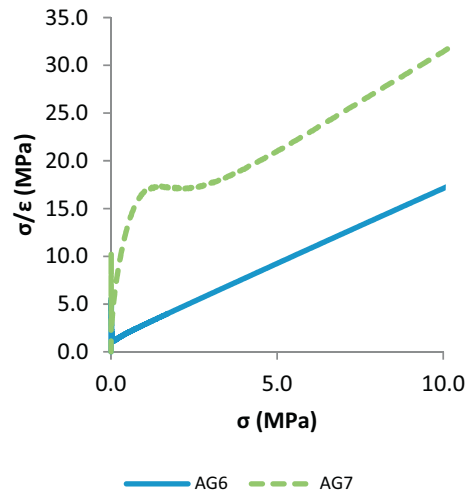


Fig. 13. Kawakita plots for AG6 and AG7.

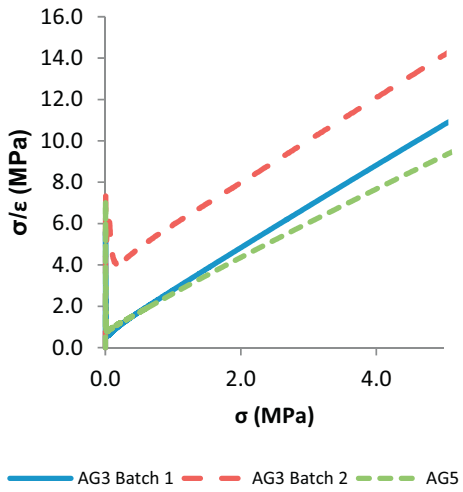


Fig. 11. Kawakita plots for AG3 Batches 1, 2 and AG5.

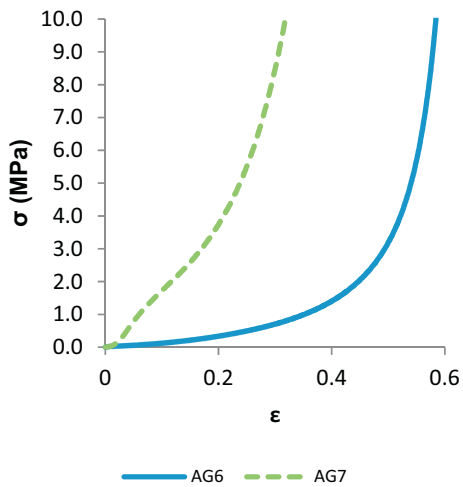


Fig. 12. Stress versus strain for AG6 and AG7.

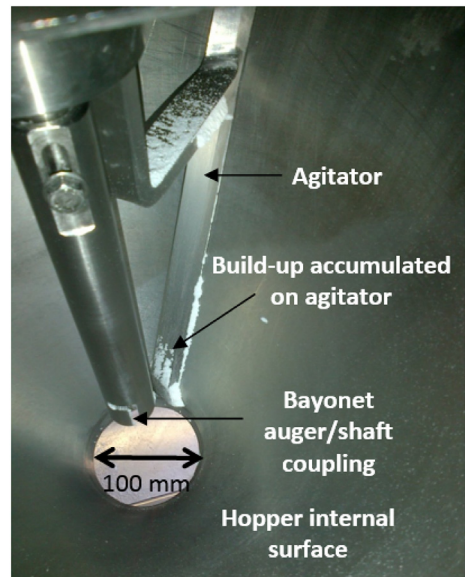


Fig. 14. Interior of auger filler hopper showing build-up on the agitator. The auger and tube were removed from the bayonet coupling post operation of the auger filler and powder was removed from the hopper.

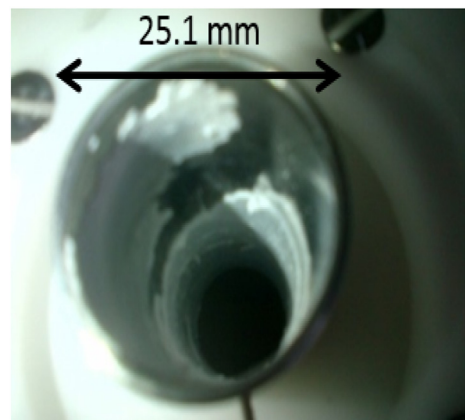


Fig. 15. Build-up on the tubes internal surface.

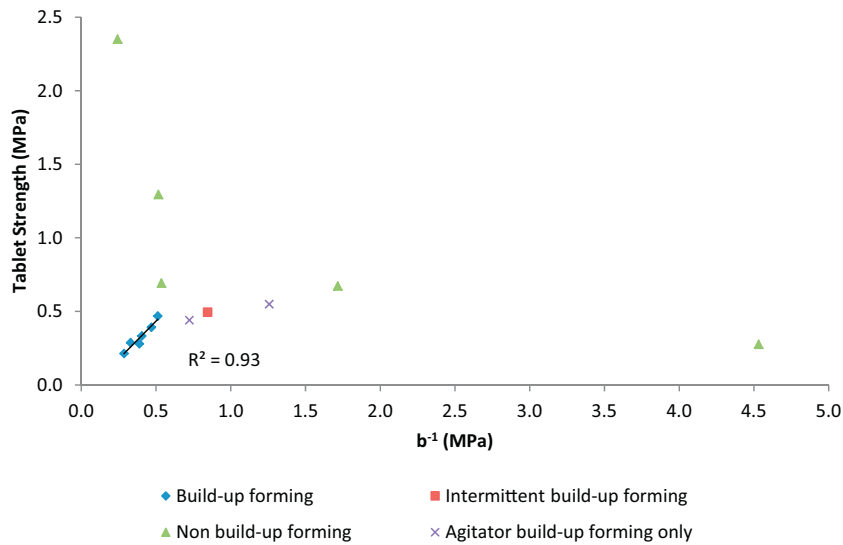


Fig. 16. An operating space plot for build-up and non build-up forming detergent powders.

in Table 4 are plotted in Fig. 15 showing that powders with low b^{-1} parameters and plastic yield stress (assuming that build-up forming agglomerates fail by plastic yielding) tend to give rise to an increase in torque, earlier in auger filler build-up experiments. It seems reasonable to assume that a number of agglomerate particles will pass through the auger fillers auger/tube clearance during each rotation of the auger and a fraction of these agglomerate particles will be transformed into build-up. The data presented in Fig. 17 suggests that this fraction is inversely proportional to the Kawakita b^{-1} parameter, which would indicate that agglomerate particles of low yield stress have a higher probability of forming build-up than particles of high yield stress.

Eq. (7) shows that while agglomerate particles of low yield stress will tend to undergo a greater level of deformation in response to a given force, their associated low values of plastic hardness will tend to mean that the work per unit contact area required to remove them from the tube surface will be lower than would otherwise be the case. This suggests that at some low value of plastic yield stress, adhesive forces may be insufficient to enable build-up to remain adhered to the tube's surface under the influence of the forces acting upon it. However, as can be seen in Fig. 17 this was not observed to be the case, which

suggests that the stresses acting to remove build-up from the tube surface are not sufficiently great to achieve this. This may be explained in a number of ways:

- The adhesive force acting between build-up and the tube surface must be sufficiently great to resist forces acting upon it.
- Build-up may undergo plastic flow under the influence of forces exerted upon it by the auger, which would act to dissipate forces prior to them reaching the build-up tube surface interface.

AG2 Batch 2 was excluded from the correlation presented in Fig. 17 as it is believed that data collected during its auger filling was significantly influenced by its large agglomerate particle size (geometric mean particle size 772 μm) relative to the other build-up and intermediate build-up forming powders included in this correlation (see Table 2). This is supported by the fact that during the auger filling of AG2 Batch 2, build-up was observed to form in a manner different to that observed in other experiments such as the auger filling of AG2 Batch 1. Specifically following build-up being observed to begin to be formed at 141 ± 47 fills, the auger was observed to rub against one

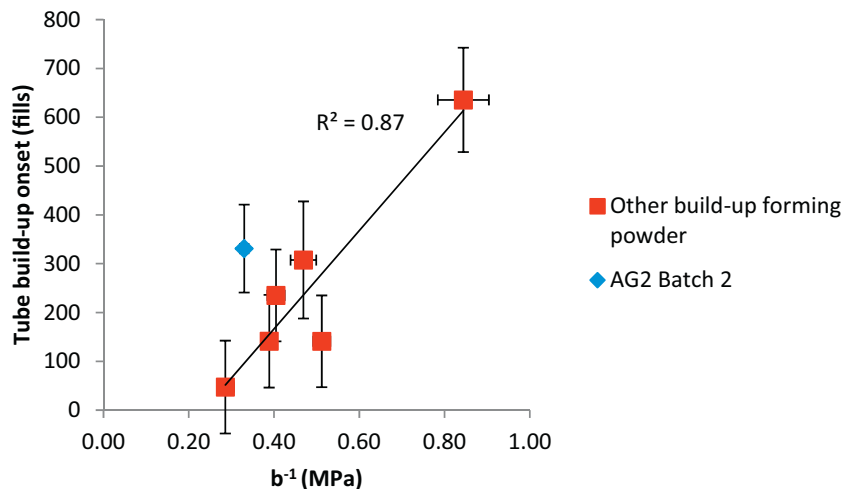


Fig. 17. Tube build-up onset as a function of b^{-1} .

Table 4

Kawakita parameters, tablet strengths and build-up observations.

Powder	Batch	σ_a	b^{-1} (MPa)	Tablet strength at 58 MPa (MPa)	Tube build-up onset (fills)	Agitator build-up observed
SD1	NA	0.76±0.01	0.24±0.02	2.35 ± 0.09	None	No
AG1	Batch1	0.52±0.01	0.47±0.03	0.39 ^a	308 ± 120	Yes
	Batch2	0.51±0.01	0.84±0.06	0.50 ± 0.01	636 ± 107	No
AG2	Batch1	0.53±0.01	0.29±0.02	0.21 ± 0.01	47 ± 95	Yes
	Batch2	0.54 ^a	0.33±0.01	0.29 ± 0.02	331 ± 90	Yes
	Batch3	0.52±0.01	0.39±0.01	0.28 ^a	141 ± 95	Yes
	Batch4	0.54±0.01	0.51±0.02	0.47 ± 0.01	141 ± 94	Yes
AG3	Batch1	0.49 ^a	0.40±0.02	0.33 ± 0.01	235 ± 94	Yes
	Batch2	0.49 ^a	1.72±0.08	0.67 ± 0.06	None	No
AG4	Batch1	0.50 ^a	0.72±0.02	0.44 ^a	None	Yes
	Batch2	0.53±0.01	1.26±0.09	0.55 ± 0.02	None	Yes
AG5	NA	0.59 ^a	0.54 ^a	0.69 ± 0.01	None	No
AG6	NA	0.61±0.01	0.52±0.10	1.30 ± 0.15	None	No
AG7	NA	0.47 ^a	4.53±0.19	0.28 ^a	None	No

^a Standard error < 0.1.

side of the tube with build-up forming on the opposite side of the tube only. This behaviour was not observed during any of the other auger filler experiments.

4. Conclusions

It has been shown that powders of low Kawakita b^{-1} parameter (< 0.5 MPa) and tablet strength (<0.5 MPa) form build-up while powders with higher values did not. The Kawakita b^{-1} parameter is known to be related to the failure stress of the particles within the die [4]. In the case of plastic particles, the failure stress will be the plastic yield stress. Rumpf et al. [13] stated that the adhesive force acting between two plastic deforming spheres is proportional to the applied force and the ratio of the van der Waals pressure to the plastic yield pressure of a sphere.

Powders of low plastic yield stress (and it follows low b^{-1} parameter) will tend to form build-up because:

- I. Low values of plastic yield pressure will lead to high values of adhesive force.
- II. As the separation distance between particles reduces due to plastic deformation the van der Waals pressure will increase and adhesive forces increase.

It is proposed that low tablet strengths are required for build-up to form because build-up forming agglomerate particles are formed from soft plastic materials which compact to form soft plastic tablets. These tablets are of low strength, due to the soft tablets failing via ductile separation of contacts, with the tablet providing limited resistance to contact separation due to their low plastic hardness.

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