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Temperature and time consolidation effect on the bulk flow properties and arching tendency of a detergent powder

Abstract

Time consolidation flow properties of a commercial detergent powder were measured at elevated temperature of 27 °C, 37 °C and 47 °C with the Brookfield Powder Flow Tester (PFT). A substantial increase of powder cohesion was observed at elevated temperature and after time consolidation. The Jenike method for silo design was used to correlate the powder time consolidation flow properties at elevated temperature to critical outlet slot to attain arch-free silo. The dependence of the critical diameter on temperature and time consolidation is successfully described by the model.

Keywords

Arching, Brookfield Powder Flow Tester, Jenike Method, Silo Design, Time Consolidation Flow Function

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1. Introduction

It is reported that around 60% of products in the chemical industry are produced as particulate solids [1]. All these materials must be transported, conveyed or handled. Therefore, the characterisation of the flow behaviour of powders plays an important role in industrial applications [2]. Powder flow properties are mainly dependent on the magnitude of cohesive forces between particles, such as van der Waals' force and capillary forces, as well as forces between particles and the storage container surface [3]. When these forces increase because of changes in temperature or consolidation stress the powder flowability deteriorates and,

therefore, becomes more difficult to handle [4, 5]. A reduction in flowability of powder at an industrial facility could for example decrease the production rate as a result erratic or unreliable discharge from storage hoppers or even stop production for extreme cases such the formation of stable arch over the outlet. The latter arching problem commonly occurs because of cohesion increases between particles at elevated temperature and or after periods of time consolidation [6]. Due to the large outlet sizes that maybe required to break these arches, specific silo discharge equipment, such as; planetary screws, multiple screws "live bottom", vibrating slat , or air induced system, are often applied as a solution to facilitate silo discharge. However, unfortunately, implementation of these approaches cause an increase in particulate solids handling costs [7].

A theoretical method was established by Jenike to predict the minimum hopper opening slot during discharge in order to prevent arch formation in wedge and conical shape hoppers [8]. This method is a function of hopper geometry (conical or wedge shape), slope angle, wall friction and of the bulk material flow properties. It is essential to evaluate the bulk material flow properties at conditions similar to the realistic industrial conditions, for instance at high humidity [9–11], at very low consolidation stress [12–14] and at high temperature [15]. Smith et al. [16] preheated two powders to 750 °C and then measured their flow properties with the Jenike shear tester without controlling the temperature during experiments. In another study, the torque necessary to rotate an impeller in a bed of different cohesive powders at an elevated temperature of up to 700 °C were performed by Zimmerlin et al.[17]. Modified Schulze shear testers capable of working from 80 °C to 220 °C and up to 500 °C were developed by Ripp and Ripperger [18] and Tomasetta et al., [19] respectively. Tomasetta et al., [19] measured the flow properties of several powders at ambient and 500 °C. However, experimental results did not show a univocal effect of temperature on the flow properties of the tested powders.

Few studies have investigated the effect of temperature on flow properties of powders, however there is no study considering time consolidation flow properties of a commercial detergent powder at elevated temperature. Furthermore, there are no studies considering the

effect of elevated temperature and time consolidation flow function on silo design for a detergent powder. These materials are often subjected to elevated temperatures in handling, for instance in storage hoppers in plants in tropical areas, so the effect of these on lump formation in the powders is critical to product quality. In the light of these research gaps, the aim of this work is to assess the effect of temperature and time consolidation on the flow properties of a commercial detergent powder. Consequently, arching tendency of the powder at the tested experimental conditions were evaluated using the Jenike approach.

2.   Material and method

A commercial spray dried washing detergent powder (the most popular detergent powder sold worldwide) was provided by a manufacturing company. The composition of the detergent powder is confidential (commercially sensitive), however it is a salt-base powder with addition of zeolite, sulphate and colour. The particle size distribution of the detergent powder was evaluated by using vibrational mechanical sieves following the standard procedure of ASTM D6913. The D_{10}, which is the diameter at which 10% of the sample's mass is comprised of particles with a diameter less than this value, is 65 μm; the D_{50}, median particle size distribution, is 250 μm; and D_{90}, means that 90% of the sample has this size or smaller size, is around 480 μm. For measuring time consolidation flow properties of samples at high temperature, a Brookfield Powder Flow Tester (PFT, Brookfield Engineering Laboratories, Inc., Middleboro, MA, US) [20] was placed in an environmental chamber. PFT is a ring shear tester which operates by applying a vertical compression through the lid onto the powder sample contained in the annular trough (internal volume 230 cm^3, external annulus diameter 152.4 mm). An internal automated procedure controlled by the 'powder flow software'is used to operate the cell to reproduce the sequences of normalstresses and the shear movement necessary to define the yield loci, as is described in Figure 1 assuming a linear (Coulomb) yield locus. The temperatures in the climate chamber were set to; 27 °C, 37 °C and 47 °C and consolidation durations programmed for 24h and 72h, giving total 6 experimental conditions. The maximum possible environmental temperature for measuring flow properties of powder

with the PFT is 50 °C. The relative humidity was kept at 15% in all experiments. The number of pre-shear points (number of yield loci lines) and of shear points (number of yield locus points) were set to 2 and 5 respectively. The samples were renewed after the first yield loci line determination.

The typical shear stress chart for the pre-shearing and shearing phase, which used for defining material flow properties, is reported in Figure 1. The test method follows two main steps. In the first step, pre-shear phase, the powder sheared at the defined pre-shear normal stress, σ_c, until attaining the steady state shear force needed to keep the lid in the fixed position. In the second step, shear phase, the normal shear stress, σ_{Ni}, which is lower than the stress applied to the sample at pre-shear phase, is applied to the sample. The maximum measured shear stress value at the shearing phase used to calculate the failure strength of the pre-consolidated samples. Repeating the same procedure with constant applied normal stress during pre-shear phase and decreasing the normal load at shear phase enables to define the yield locus line [21, 22].

The flow properties of the sample defined by using a continuum mechanics approach. Particularly, the Mohr-Coulomb approach is used to describe the stress distribution inside the particulate solid. State of stresses is described by using the Mohr's circle on both normal stress, σ, and shear stress, τ, plane. Major principal stress, σ_1, is determined from the crossing point of the big Mohr's circle and the σ-axis in Figure 1. This Mohr's circle is tangent to the yield locus line which is passing through the yield locus points. The unconfined failure strength of powder, f_c, is derived from the intersection of smaller Mohr's circle and the σ-axis. Therefore, the unconfined failure strength, calculated from equation 1, is a function of Cohesion, C, and angle of internal friction, ϕ, which calculated from the slop of the yield locus [21, 22].

$$f_c = 2c\frac{\cos\phi}{1 - \sin\phi} \tag{1}$$

Flow function, the ratio between unconfined yield strength and major principal stresses, of the detergent powder at room temperature and without time consolidation stress is reported in

Figure 2. The flow function, *FF*, representation is the way to classified and report powder flowability. In fact, Jenike [23] used flow factor value, $ff = \sigma_1/f_c$, for flowability classification of powder. His considered classes are free-flowing ($ff>10$), easy flowing ($4<ff\leq10$), cohesive ($2<ff\leq4$), very cohesive ($1<ff\leq2$) and non flowing ($ff\leq1$). The flow function of the spray dry washing detergent powder was classified as easy flowing at ambient temperature for instantaneous conditions, but shows an unusal convex upward curvature, i.e. flow function gradient increases with stress. The wall friction properties of the detergent powder against a stainless steel wall were also measured with the PFT at the same experimental conditions of temperature, storage time and consolidation stress.

2.1. Jenike theory of critical hopper outlet size

For predicting the lowest wedge-shape hopper opening size to attain an arch free silo, the hopper design procedure developed by Jenike [24] was followed. In this method, the arch weight is balanced by the vertical component of the abutment stress, which is the stress within the material parallel to the arch surface close to the walls. Equation 2 was derived from calculating force balance assuming that the arch thickness is constant through the formed arch as well as the arch only hold its own weights.

$$f_c < \frac{\rho_b\, g\, D}{h(\alpha)} \tag{2}$$

where f_c is the unconfined yield strength of the detergent powder, D is the effective outlet size, ρ_b is the powder bulk density, g is the acceleration due to gravity, $h(\alpha)$ is (a shape factor) a function which takes into account the effects of variation of the thickness of the arch with the silo geometry and the hopper half-angle, α. For measuring $h(\alpha)$ a graphical plot for a wedge shape hopper at different hopper opening half-angle was reported by Schulze [23].

In mass flow silos, the consolidation stress at the outlet, σ_1, depends on the distance from the virtual hopper vertex. Making the hypothesis of radial stress field and stationary flow, Jenike derived the value of the major principal stress in the arch abutment, σ_1:

$$\sigma_1 = \rho_b \, g \, D \, \frac{\left(1 + \sin \varphi_e\right) s\left(m, \alpha, \varphi_e, \varphi_w\right)}{2 \sin \alpha} \tag{3}$$

where s is a complex function depending on the hopper geometry (wedge or conical), on its half angle, α, on the tensional state (m=1 for active state, m= -1 for passive state), on the powder effective angle of internal friction and wall friction, φ_e and φ_w respectively. By combining Equations 2 and 3, it is possible to obtain the free flow criterion:

$$f_c < \sigma_1 \, \frac{\left(1 + \sin \varphi_e\right) s\left(m, \alpha, \varphi_e, \varphi_w\right)}{h(\alpha) 2 \sin \alpha} = \frac{\sigma_1}{ff} \tag{4}$$

where ff, as defined in the equation above, is the flow factor, the ratio between consolidation stress in the hopper and the stress required to support a stable arch. The flow factor was calculated by Jenike and available in diagrams for wedge shape hoppers for different values of φ_e, where ff appears as a function of α, and φ_w. On the f_c - σ_1 plane, the flow factor line (σ_1/ff) cuts the flow function curve, $FF(\sigma_1)$, that is the experimental constitutive equation of the material in which the unconfined yield stress, f_c, is given as a function of the consolidation stress σ_1:

$$f_c = FF(\sigma_1) \tag{5}$$

The intersection between the flow function and the flow factor line provides the critical unconfined yield strength of the material, f_c^*. ρ_b^* is the critical bulk density of the material at the arch. The smallest outlet size, D_c, providing arch free flow, hence is given by:

$$D_c = \frac{f_c^* h(\alpha)}{\rho_b^* g} \tag{6}$$

3.   Results and discussion

Table 1 reports the time consolidation flow properties of the tested detergent (namely, normal consolidation stress during pre-shear phase σ_c, temperature T, cohesion c, bulk density ρ_b,

angle of internal friction φ, effective angle of internal friction φ_e and angle of wall friction φ_w) at 3 different temperatures and storage durations of 1 and 3 days.

Table 1. Results of the shear tests performed with the PFT at elevated temperature.

Time	σ_c	T	c	ρ_b	φ	φ_e	φ_w
[day]	[kPa]	[°C]	[kPa]	[kgm³]	[°]	[°]	[°]
Instantaneous	0	22	0.04	380	24.8	32	23.6
1	1.2	27	0.372	571	33.3	38.4	26.5
		37	0.586	588	33.1	41.7	29.6
		47	0.877	610	33	42.5	42.2
	2.4	27	1.015	619	33.7	40.5	18.3
		37	1.41	624	33.3	43.4	19.4
		47	1.951	660	34.8	44.9	23.8
3	1.2	27	0.292	622	32.1	37.5	30.3
		37	0.908	630	32.4	45.4	38.7
		47	0.965	645	35.3	48.6	44.5
	2.4	27	0.855	662	32.9	38.7	19.7
		37	2.534	679	33.7	47.2	25.2
		47	5.772	684	33.3	49.4	26

Considering the effect of both consolidation duration and temperature on the powder flow properties, inspecting Table 1 indicates that cohesion, c, increases with temperature and consolidation stress. Elevated temperature makes detergent particles more plastic and thus more cohesive. In contrast, the angle of internal friction, φ, is hardly dependent on the change in temperature and consolidation stress. Hence, the increase of the unconfined failure strength (calculated from Equation 1 and reported in Figure 1 and Figure 3) of the tested detergent is as a results of cohesion increase. Bulk density of the detergent is slightly shifted when both consolidation stress during pre-shear phase and temperature increased. When higher stresses are applied to the powder, particles rearrange and bridges and gaps between particles are eliminated and a more efficient packing of particles are formed, hence the volume slightly decreases which leads to a higher bulk density. When temperature increases, to some extent particle melting cause higher contact area between particles which leads to lower porosity in bulk powder, and, hence a bulk density increase.

The wall friction angle, φ_w, is an important parameter in determining the minimum hopper half angle required for designing mass flow silo without the possibility of arch formation. The wall friction angle reports the necessary stress to move powder along the surface of the desired wall material, i.e. 304 stainless steel (with a 2B cold rolled finish) in this research. The angle of wall friction increased by almost 60% and reached 44.5 ° (at 47 °C -3 days) from 26.5 ° (at 27 °C-1 day). The increased wall friction angle could be as a result of higher thermoplasticity at tested elevated temperatures which cause the surface of particles to become stickier. Furthermore, at the elevated temperature the powder moisture content decrease which may lead to an increase in friction of the particles and the wall surface mounted on the PFT lid. Apart from storage temperature, time consolidation was shown to have an influence on powder wall friction. This could be due to the fact that some adhesive bonding between individual particles, i.e. crystal bridge formation, as well as bonding between particles and wall may require a longer time to form. In addition, higher compaction stresses may cause stronger particle-wall interactions which all lead to an elevated wall friction angle.

Figure 3 reports time consolidation flow functions of the detergent powder at different temperatures. Particularly, Figure 3a reports the flow function after 1 day storage and Figure 3b after 3 days storage. Measuring temperature dependent time consolidation is an important parameter for silo design, because only then the calculation on the formation of arch is correctly possible to perform. Inspection of Figure 3a reveals that the flow function of the tested powder at higher temperature increased compared to the lower tested temperature after 1 day storage. Flow function of detergent powder tested at 27 °C and 37 °C were classified in the cohesive region while the flow function at 47 °C falls in the very cohesive part with a slight tendency towards lower cohesive flow index at higher consolidation stress. A slight shift of major principal stress when temperature increased were also observed. Comparing Figure 3a&b shows that at 27 °C the flow functions falls on the boundary of the cohesive and easy flowing ranges but the 1 day storage period shows a slightly greater strength than the 3 day

storage. This is due to the reduction in cohesion and angle of internal friction of the detergent when storage duration increased to 3 days (see Table 1)

Temperature increases to 37 °C and 47 °C caused a dramatic increase in the unconfined yield strength of the detergent, and hence, the flow function shifts from easy flowing region to very cohesive region. The shift in flow function curve could be illustrated by the formation of interparticle bridges due to increase in the particle contact areas due to plastic flow of the particles over time bulk powder sintering at the temperature slightly lower than melting temperature of the detergent powder. This is also reported during storage of plastic particles at ambient temperature [23]. The other reason could be an increase in adhesive forces at elevated temperature [23]. Particularly, Chirone et al [15] reported that the change in flow properties of ceramic powder at high temperature is due to the change in van der Waals forces.

There were no repetitions reported in Table 1. However, one repetition for the flow function measurements of sample which stored for 1-day at 37 °C was cried out. The results showed les than 2% deviation between the 2 flow function tests. Furthermore, the previous study which have done by Salehi et. Al [21] showed that the standard deviation between different repetition of flow function measurements using PFT are usually negligible.

3.1 Model Results

According to the Jenike approach for silo design, the powder flow properties reported in Table 1 were used to calculate predicted Jenike design opening slot values reported in Table 2.

Table 2. Main outlet design values

Time	T	ff	f_c^*	ρ_b^*	α_m	$h(\alpha_m)$	D_c
[day]	[°C]	[-]	[kPa]	[kg.m^{-3}]	[°]	[-]	[m]
Instantaneous	22	1.1	0.3	343	28.6	1.12	0.1
1	27	1.4	0.66	556	31.4	1.15	0.141
	37	1.4	1.20	577	27.9	1.14	0.242
	47	1.3	1.50	597	16.0	1.08	0.377
3	27	1.4	0.555	604	27.7	1.13	0.106
	37	1.2	1.79	614	18.1	1.09	0.324

| 47 | 1.2 | 2.04 | 627 | 16.4 | 1.08 | 0.358 |

The flow factor values, *ff*, were determined by using the diagram provided by Jenike [23]. The critical value of the unconfined yield strength, f_c^* as well as the critical value of bulk density, ρ_b^*, in equation 6, were determined by the intersections between the flow factor line with both flow function and density curves. α_m is the maximum possible hopper half angle to have the mass flow discharge and above this angle the core flow regime is dominant in the hopper. Values of $h(\alpha_m)$ were evaluated based on the method developed by Schulze [23]. The f_c^* and ρ_b^* were used in equation 6 to calculate the prediction values of the critical slot opening to avoid arching (D_c).

In general, the value of D_c increased with temperature and storage duration. In particular, the critical outlet opening to avoid arching was largest for the highest storage temperature and storage duration. The critical hopper half angle to attain mass flow discharge, or in other word to avoid core flow discharge, decreased by almost 50%, from 31.4 ° (27 °C-1day) to 16.4 ° (47 °C-3days). This indicates that for a fixed amount of headroom, the designed silo will have a lower storage capacity when the detergent powder is intended to be stored at high temperature for 3 days. In a core flow silo, only the material in the channel above the opening slot flows at the initial stages of discharge. Afterwards, materials placed in the stagnant area (located at the silo periphery starting at the hopper walls) could discharge. These zones are usually formed asymmetrical, hence causing unfavourable stress on the silo walls. The other unfavourable issue in the core flow silos are rathole development which formed from consolidated stagnant zones. Core flow silo have several disadvantages compared to mass flow silo where the potential problem of arching remains [23].

The critical opening slot of the tested detergent stored at 27 °C for 3days was slightly reduced compare to the detergent stored at the same temperature but under consolidation for one day. This behavior is in-line with the flow function classification of the detergent at the two tested storage durations (see Fig. 3). The predicted critical opening slot for all tested conditions seem

to be very large. It has to be recalled that the Jenike procedure is a silo design procedure and, as such, tends to provide silo design estimates on the safe side. In particular, over-estimated predicted critical opening diameter in comparison with the experimental values have been reported in several studies [7, 25, 26].

Conclusion

Flow properties of a commercial washing detergent powder at high temperature and at different time consolidation have been studied. The flow function showed considerable dependence on temperature and consolidation duration. The Jenike approach for silo design has been followed for predicting the minimum hopper opening slot to attain arch-free silo. The critical outlet slot width to avoid arching increased by a factor of 3.5, and reached to 0.358 m from 0.1 m (at Instantaneous condition), when the temperature and time consolidation increased to 3 days and 47 °C respectively. The results showed that in order to correctly design a silo, it is essential to evaluate the bulk material flow properties at situation similar to the realistic industrial conditions where the powder may store for several days at elevated temperature.
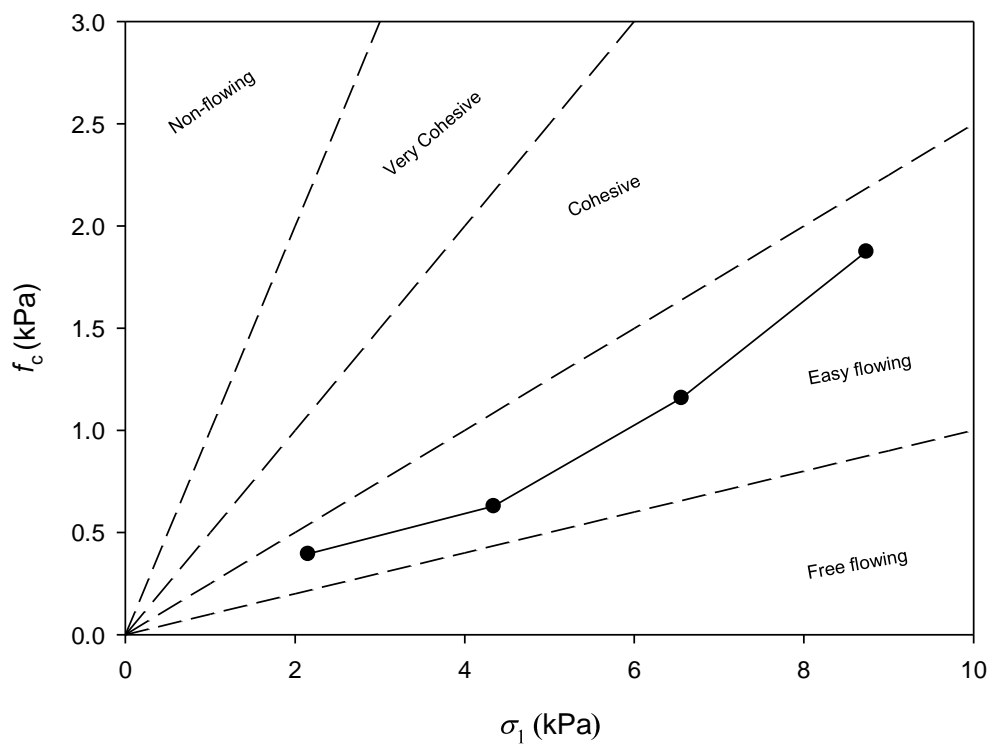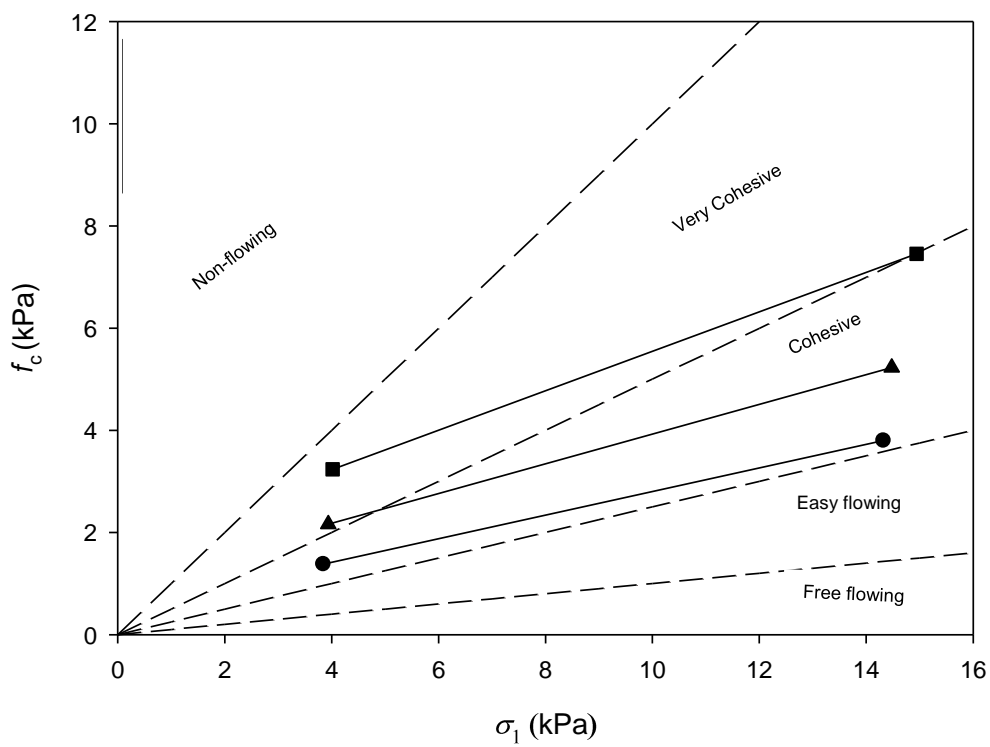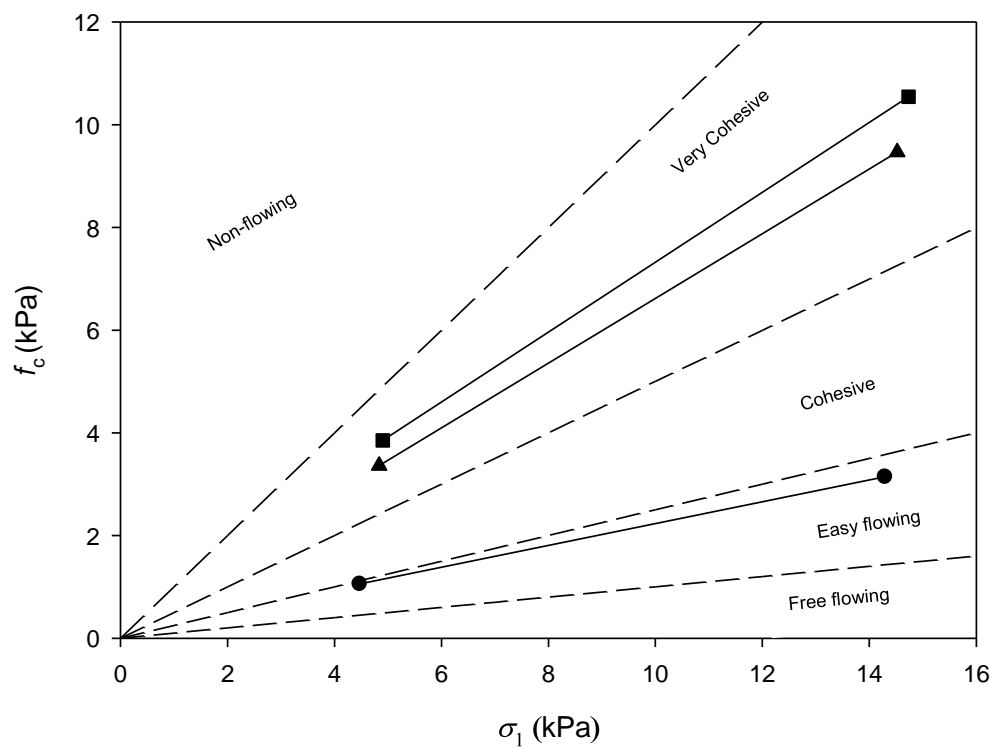
Figure 1. Procedure for evaluating yield locus line (assuming Coulomb) [21].

Figure 2. Flow function of the tested detergent at room temperature without applying time consolidation stress (instantaneous flow functions). (The vertical axis has been expanded by a factor of 4:1).

a)

b)

Figure 3. Time consolidation flow function of the detergent powder at different temperature. ●, 27 °C; ▲, 37 °C; ■, 47 °C. a) after 1 day storage and; b) after 3 days storage.

Acknowledgement

List of symbols

C	[Pa]	cohesion
D	[m]	effective outlet size
D_c	[m]	critical outlet size
f_c	[Pa]	unconfined yield strength
f_c^*	[Pa]	critical unconfined yield strength
ff	[-]	flow factor
FF	[-]	flow function
g	[m/s^2]	the acceleration due to gravity
h	[-]	function which takes into account effects of variation of thickness of the arch with the silo geometry and the hopper half-angle α
R	[Pa]	radius of Mohr circle at steady state condition
P	[Pa]	centre coordinate of the Mohr circle at steady state condition
S	[-]	a function depending on hopper geometry, on half angle, α, on the tensional state ($m = 1$ for active state, $m = -1$ for passive state), on effective angle of internal friction, ϕ_e, and on wall friction, ϕ_w,
α	[°]	hopper half angle
φ_i	[°]	kinematic angle of internal friction
φ_e	[°]	effective angle of internal friction
ϕ_w	[°]	angle wall friction
σ_1	[Pa]	major principal stress
$\bar{\sigma}_c$,	[Pa]	average normal consolidation stress
σ_N	[Pa]	normal stress during shear phase

τ	[Pa]	shear stress
τ_c	[Pa]	shear stress at steady state condition during pre-shear phase
$\bar{\tau}_c$	[Pa]	average pre-shear shear stress
ρ_b	[kg m^{-3}]	bulk density
ρ_{b*}	[kg m^{-3}]	critical bulk density
ρ_t	[kg m^{-3}]	tap density

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Table and Figure captions

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