Journal of
Chemical Engineering & Process Technology

Hart, J Chem Eng Process Technol 2015, 6:1
DOI: 10.4172/2157-7048.1000215

Research article Open Access

Effect of Particle Size on Detergent Powders Flowability and Tabletability

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Abstract

Detergent powders are sometimes processed into compact tablets for cleaning purposes based on the advantage of dosage, minimised dusting and packaging its offers over their powder counterpart. The flow-ability greatly influences handling. The particle size within the powder plays a key role on handling, flow-ability and the strength of the produced compact detergent tablet. The Ariel powder used in this study was separated into different size ranges via sieving; thereafter flowability, compression and compaction were carried out. The compressibility of the powder under 2 KN compaction force was analysed using Heckel equation. It was found that at the same compaction pressure, smaller size particle powders formed compact of greater tensile strength than larger particle. However, the powder with size range of 125-180 μm showed higher flowability compared to other size range.

Keywords: Detergent powder; Particle size; Flowability; Tabletability

Introduction

It is well know that particle size and size distribution is one of the many factors that greatly impacts on the flowability of powder during handling, processing and tableting. Flowability is a vital parameter for efficient and effective transport, storage and handling of detergent powder in the detergent industries. It is the ability of the powder to flow in desired manner in a given processing or handling piece of equipment [1]. Flowability remains a crucial factor that affects the design and processing of detergent powder in handling equipment such as hoppers, silos, filling and packaging operations, conveying, etc. [2]. There are many techniques to assess the flowability of powders which includes; measuring the time required to discharge a given amount of powder from a flowmeter, carr's compressibility index, Hausner ratio, angle of repose and flow function [3,4]. However, the Jenike flow function is mostly used to classify the flowability of powders. The higher the flow function of the powder, the better the flowability. As poor powder flow can lead to sticking or caking during storage, prone to cohesion, rat-holing, arching, poor content uniformity and poor solubility [4].

On the other hand, detergent powders are currently been processed into compact powder and detergent tablets for cleaning purposes based on the advantages it present such as easy dosage, minimised dusting, easy packaging and safer for consumers [2,5]. A tablet is a defined assembly of discrete particles with each particles bonded to its closest neighbours. The strength of the tablet formed depends on particle size and particle rearrangement as well as densification and interaction between the particles by bonding during compaction [6]. Therefore, among the mechanical properties of interest in detergent tablet, the tensile strength is of importance to the detergent industries as it determines the tablet potential to sustain loading encountered during handing, filling and processing. The tabletability of the detergent tablet is described in *XY*-plot of tensile strength of the tablet versus compaction pressure. The tensile strength of the compacted tablets is determined by diametric compression. In diametrical compression test, the tablets are generally placed vertically between two platens and compressed until the tablet breaks; the value of the maximum force is used to calculate the tensile strength [7].

To form compact tablet however uniaxial compaction is used to consolidate the powder bed by reducing the voids and creating particle-to-particle bonding. During the tabletting, the applied stress causes the particles to rearrange themselves leading to increased bulk density, fragmentation, plastic and elastic deformations. The strength of the resulting tablet depends on the particle-to-particle bond formed during the compaction. The strength of the particle-to-particle bond in the tablet depends on the solid pressure (i.e., particle-to-particle pressure), which is the difference between the total applied pressure and interstitial air pressure in between the punches in the die. The tensile strength of the formed tablet depends on the strength of the particle-to-particle bonding, the porosity, and the resultant pressure. However, powder flow properties can be utilised to predict uniaxial compaction behaviour [1]. The coordination number within the tablet depends on the particle size and size distribution and on the solid fraction (or porosity) of the tablet [4]. Therefore, particle size and solid fraction (or porosity) greatly impact on the tensile strength of the tablet compared to the compaction pressure used in producing the compact. In addition, the solid fraction (or porosity) of the tablet is the direct result of the applied compression pressure [8].

Besides particle size distribution (*PSD*), shape, morphology and surface chemistry can affect the flow properties of powder and tabletability significantly. The flowability of powder is a multifunctional parameter which depends on particle size and size distribution, shape, particle interactions and moisture content [9,10]. Mullarney and leyva [11] investigated the possibility of predicting powder flowability based on the particle size distribution data. In this study, however, the effect of powder particle size on flowability and tensile strength of formed detergent powder tablet was investigated. The compressibility of the powder was evaluated using Heckel equation. The effect of particle sizes on deformation and rearrangement under compaction pressure was explored. However, the influence of particle size on the detergent powder activity and dissolution of compacted tablet are outside the scope of this study.

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Received January 22, 2015; **Accepted** February 18, 2015; **Published** February 26, 2015

Citation: Hart A (2015) Effect of Particle Size on Detergent Powders Flowability and Tabletability. J Chem Eng Process Technol 6: 215. doi: 10.4172/2157-7048.1000215

Material and Methods

The detergent powder used in this study is a Professional Ariel® Regular Powder supplied by Procter & Gamble, United Kingdom. The composition of the detergent power are thus oxygen-based bleaching agent, anionic surfactants, non-ionic surfactants, phosphate, polycarboxylate, zeolites, enzymes, perfumes, optical brighteners, butylphenyl methlpropional, hexyl cinnamal and linalool. The properties of the detergent powder are presented in Table 1. The true density of the detergent powder was measured with a helium gas pycnometer (AccuPyc™ II 1340, Micrometritics®, Germany).

The particle size distribution (PSD) of the as-received detergent powder was obtained using sieve analysis with a stack of sieves arranged in decreasing mesh size (2000, 1400, 1000, 710, 500, 355, 250, 180 and 125 µm, respectively) and shaken for 15 min at an amplitude of 1 mm on the sieve tower (Retsch AS200, Germany) to separate the detergent powder into various size range and to obtain a distribution by mass.

An important processability parameter is the flowability of the powder. The flow properties of the detergent powder and separated size ranges were measured using the Schulze ring shear cell tester RST-4 (RST-XS, Dietmar Schulze, Wolfenbuttel, Germany). The powder was loaded into the annular space between the concentric rings of the shear cell tester. The shear stress is applied through the pins at the corners of the shear lid. The powder was first pre-sheared under a consolidation stress of 10 kPa until a steady consolidated state is reached. To construct a yield-locus, the samples were pre-sheared with four other normal stresses 8.0, 6.0, 4.0, and 2.0 kPa. The consolidation stress (σ_1) of the detergent powder and the corresponding unconfined yield strength (σ_c) were determined by constructing Mohr's circles [12], from which the effective angle of internal friction (φ_e) and powder flow function (ff_c) (i.e., the ratio of σ_1/σ_c) was determined.

A confined uniaxial compression test (Figure 1a) was performed with compression forces of 2 kN using Zwick® universal material testing machine (Zwick-Roell Z030, Germany). The powder was compressed with flat-faced punches 13 mm in diameter with an approximate sample mass of 1.15 g at a constant compaction rate of 60 mm/min to form compact tablets. The compression data were registered during the experiments with a Zwick-testXpert II software. The compaction pressure was calculated from the applied force and the cross-sectional area of the punch.

The thickness of the compacted detergent tablets were measured with a digital calliper, before subjected to diametrical compression test (Figure 1b) at a compression speed of 0.5mm/min. The maximum force required to break the tablet under diametrical compression was recorded and converted into a tensile strength value, σ_t, using equation (1). Each test was carried in triplicate.

$$\sigma_t = \frac{2F}{\pi D t} \tag{1}$$

where F is the force required to break the detergent tablet, D is the diameter of the detergent tablet and t is the tablet thickness.

Parameter	Value
True density (g.cm^{-3})	2.0147 ± 0.01
PSD d_{50} (µm)	469.5 ± 19.5
Moisture content (%)	7.37 ± 1.35
Angle of repose (°)	32 ± 0.24
Adhesive strength (Pa)	238 ± 18.83

Table 1: Properties of the detergent powder.

Results and Discussion

Flowability

Detergent powders have wide particle size distribution ranges giving to problems in the course of their handling. Operations such as fillings, blending, transportation and processing need reliable flowability to avoid stoppage, downtime and wastage. To evaluate the influence of particle size on flowability, the flow function, ff_c, of each particle size ranges of the powder were measured using Schulze ring shear cell tester. The plot of the flow function as a function particle size range of the detergent powder is shown in Figure 2. It is clear that the particle size distribution have a significant effect on the flowability of the powder. The detergent powder with particle size ranges 125-180 µm and 180-250 µm show superior bulk flow properties compared to larger particles because their sphericity make them roll or slide over one another when shear stress is applied. However, the particles (<125µm) have high surface area to volume ratio, causing particle-particle interaction forces to inhibit flow when stress is applied [11].

The flowability of the powder with size ranges 125-355 µm and 710-1000 µm are easy flowing, while the other size ranges showed poor flowability. It has been reported that rough surfaces and irregular shape causes particles to interlock and resist bulk flow when shear stress is applied [11]. However, the poor flow behaviour of the smaller size range particles <125 µm can be attributed to the cohesive nature of the particles as they tend to agglomerate upon shear application. Smallness of the particle size of the powder could tend to reduce powder flow due to the increase surface area per unit mass has provided a greater surface area for surface cohesive forces to interact resulting in more cohesive flow. Powder of size range 1400 to greater than 2000 µm was not analyzed because the separated amounts were less than necessary for the test. The processability and handling of the detergent powder therefore will be improved if the particle size range can be narrowed to the range of 125 to 250 µm compared to a wider size range distribution.

Compressibility

Compressibility is the densification and reduction in volume of a powder bed in response to applied pressure. Heckel [13] proposed that the reduction of the powder bed porosity follows a first-order response with applied pressure. The force-displacement data obtained from the uniaxial compaction experiments was mathematically transformed according to equation (2) to describe the compressibility of the detergent powder.

$$\ln\left(\frac{1}{1-D_{rel}}\right) = kP + A \tag{2}$$

$$D_{rel} = \frac{\rho_b}{\rho_t} \tag{3}$$

$$\rho_b = \frac{m}{\frac{\pi D^2}{4}\left(h - d_i\right)} \tag{4}$$

$$\varepsilon = 1 - D_{rel} \tag{5}$$

$$k = \frac{1}{3}Y \tag{6}$$

where; D_{rel} is the relative density (i.e. solid fraction) of the powder bed at applied compaction pressure, ρ_b and ρ_t are bulk and true density of the powder, m is the mass of the powder, h is the initial height of the powder bed, d_i displacement, D is the diameter of punch, Y is the

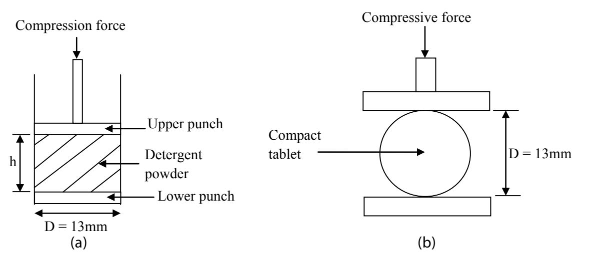

Figure 1: Schematic of (a) Confined uniaxial and (b) diametrical compression tests.

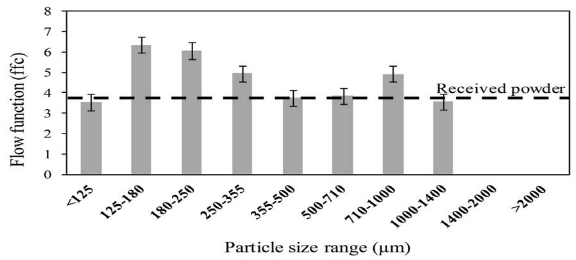

Figure 2: Effect detergent powder particle size on flowability.

yield strength and ε is the porosity. The constant *k* is a measure of the plasticity of the material and the constant *A* is related to the die filling and particle rearrangement before deformation and bonding of the discrete particles.

In Figure 3, the Heckel plots at the compression force of 2 kN for the received detergent powder and the separated powder of different particle size ranges using sieve are presented. The initial curvature describes particle fragmentation and re-arrangement without inter-particle bonding. The linear section of the plots represents plastic deformation.

Notably, the powder deforms more for the as-received detergent powder than the separated powder with size ranges 125-180, 355-500, and 500-710 µm, respectively. However, when compression pressure is greater than 15 MPa the densification is almost 100% (Figure 3) as the compacts are close to a solid continuum. This is as a result of the changes in material properties due to broken capsules containing perfumes and surfactant within particles as pressure increases during strain hardening. This results in negative porosities and the undefined parts of the Heckel plot beyond 15 MPa [14].

The linear section of the Heckel plots obtained for the different particle size range powders are linearly fitted to determine the constants *k* and *A*. It has been observed that the particle size is related to the deformation and fragmentation behaviour of powder under compression during compaction to form tablet [15]. The Heckel constants k and A describe the structure of the powder bed changes and consolidation mainly due to particle rearrangement, plastic deformation, and fragmentation. The effect of particle size on *k* and *A* are presented in Figure 4a and 4b.

The *k* values increases up to particle size 250-355µm and decreases sharply with increasing particle size. This implies that the plastic deformation for the material decreases with increasing particle size. However, the Heckel constant *A* increases with increasing particle size, expect for the powder with particle size range of 710-100 µm. This shows that for larger particle size powder there is greater particle rearrangement and fragmentation before bonding. In this regard, a greater compression pressure is required to create effective particle bonding. Hence, particle size of the powder will influence the final compact porosity, tensile strength and subsequently the compact dissolution.

Compatibility

The variation of tensile strength of the detergent with particle size range at 2kN compression force is shown in Figure 5. It is clear that the tensile strength of the tablet decreases with increasing particle size. Notably, at the same applied compaction pressure, smaller particles formed tablets of higher tensile strength than larger size counterparts. Additionally, the density of the compressed power increases with decreasing particle size, thereby reducing the porosity of the formed table which correlates with the increasing tensile strength with particle size decrease. This is as a result of particle size increment and the smoothness of the larger particles which reduces particles interlocking and bonding.

The tensile strengths of the size ranges 125-180, 180-250, and 250-355 µm are approximately the same, before significant decrease in tensile strength was noticed as the particle size increase from 355 to 1400 µm. Therefore, smaller particles provide a larger surface area for interparticle bonding than larger particles, hence the resulting tablets after compaction is stronger. However, Abdel-Hamid et al. [6] has reported the size of particles plays a vital role in the interaction as well as available surface area and bond propensity during compaction of powder material. A similar observation in the increase of tensile strength of tablet with decreasing particle size for L-lysine monohydrochloride dehydrate powder was reported by Sun and Grant [16]. The porosity of the compacted tablet decreases with decreasing powder particle size. Additionally, reducing powder particle size increase its surface area per unit mass and the feed powder quantities and this result correspond to increase tensile strength [17]. Smaller particle size of powders provided a greater surface area for surface cohesive forces to interact thereby making the tablet to increase in tensile strength

Statistical tools such as correlation and linear regression were used to test the relation between flowability and tabletability for the separated detergent power particle size ranges. The correlation coefficient (R^2) is a vital estimate of the strength of the linear regression analysis. The R^2 value of 0.273 was obtained and the association is significant at the P value of 0.554. The P value is therefore not significant at $P < 0.05$. Hence, the tensile strength of the detergent tablet and the flowability of the separated powder size ranges are poorly correlated. Flowability and tabletability are functionally dependent on the interparticle interaction. The interparticle interactions are strongly affected by surface properties such as texture, surface chemistry, and contact area. In compaction, the dominating interparticle interactions are identified as solid bridges, intermolecular forces, and mechanical interlocking. However, flowability is influenced by particle size, size distribution, shape, surface texture, surface energy, chemical composition, moisture content, particle geometry, and other factors [18]. Therefore, the flowability and tabletability are multifunctional parameters strongly influenced by geometrical, material, physical, chemical and mechanical properties of the particle.

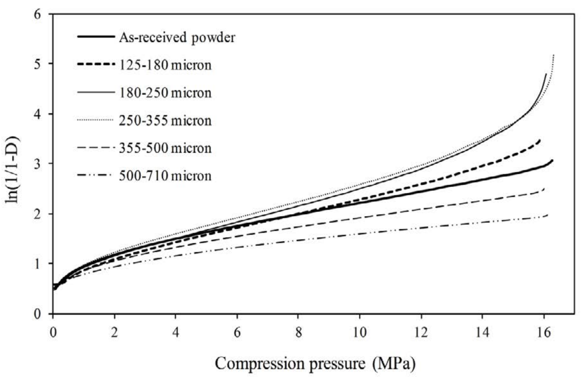

Figure 3: Heckel plots for the received detergent powder and separated particle size ranges (2 kN compression force).

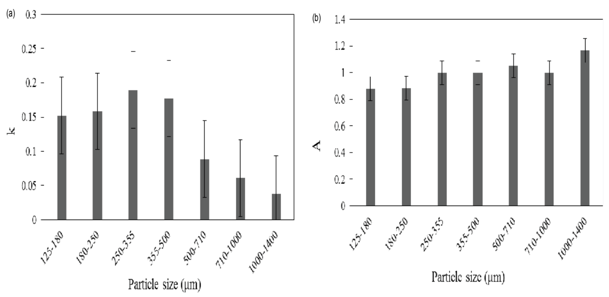

Figure 4: Effect of particle size on Heckel contacts (a) k and (b) A at 2 kN compression force.

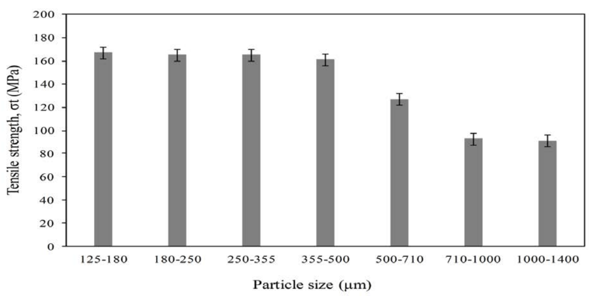

Figure 5: Variation of tensile strength with particles size for detergent powder (2 kN compression force).

Conclusions

The effect of particle size on detergent powder flowability and tensile strength of compacted tablet was investigated using Ariel detergent powder. Also investigated was the compressibility and compactibility of the as-received powder and the different separated particle size range powder using Zwick® universal material testing machine. It was found that the strength of the produced tablet was dependent on the particle size of the powder. Powder of smaller sizes showed higher tensile strength than larger particles because of their large surface area available for bonding. However, powder with size range 125-180 μm showed the highest flowability compared to other size ranges after sieving. This study is relevant with regard to the compressibility and compactibility useful to improve the production of cleaning and laundry detergent tablet with optimum strength to resist fracture on handling and transportation with the level of compaction that will enhance dissolution. However, the influence of particle size on activity of the detergent powder and the dissolution of the compacted detergent tablets require further investigation.

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