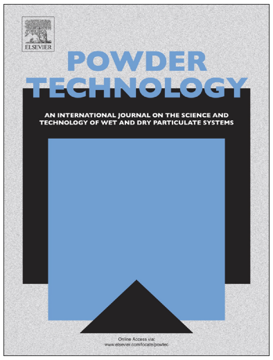

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Development and application of a novel cake strength tester

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Abstract

Caking can cause many problems in industries during processing or storage of particulate materials. Caking magnitude depends on several factors, for instance temperature, consolidation stress and storage time. In this research paper, a novel force displacement and easy-to-use caking tester for measuring quantitatively cake strength as a result of elevated temperature, consolidation stress and storage time is introduced. The developed tester outweighed the conventional uniaxial unconfined failure caking tester due to the defined location of the failure plane to maximise repeatability, the necessity for a lower quantity of powder, maximised exposed surface and lower wall friction as well as production costs. The experimental design has been conducted by changing the temperature, consolidation stress and storage duration. The results showed that the tester could distinguish cake strength between different experimental conditions. A statistical model has been successfully developed to study the effect of each variable on the cake strength.

Keywords:
plastic caking, caking strength, new caking tester, statistical analysis

Introduction

Sometimes, powder producer companies will face new challenges when storing powders at elevated temperature, humidity and/or consolidation stress. Particulate materials such as detergents, pharmaceuticals and food powders have the potential to gain strength to the extent that caked lumps can form during handling and storage. The magnitude of caking may range from fairly weak lumps to the permanent fusion of particles, whereby these materials may no longer be acceptable to the process or end user. Caking could also decrease or even stop production rates at industrial facilities, hence, often, extra equipment is needed to de-agglomerate the caked powder. Particulate materials should ideally retain their desired flow properties during processing, handling and storage as well as through the distribution chain to the final consumer [1]. These types of reduction in the handling behaviour of particulate materials can lead to huge costs being imposed on industries[2]. Such costs justify the need for developing a tester for measuring as well as means for predicting caking at industrial scales.

There are several mechanisms responsible for powder caking. In general, these mechanisms are divided into four groups, 1. chemical caking, 2. electrical caking, 3. mechanical caking and 4. plastic flow caking (temperature dependent caking). Many different inter-particle forces are responsible for the each of the caking mechanism. For instance, caking could happen due to different inter-particle forces such as liquid and solid bridges, van der Waals forces, magnetic force and electrostatic force [3]. The major adhesive force in a dry, consolidated and fine-graded powder bed is the van der Waals forces [4]. These interactions mainly depend on electric dipoles of atoms and molecules and the intensity of these interactions are affected by particle surface roughness, particle sizes and inter-particles distance (packing structure); these forces cause cohesiveness in the bulk solids [5] but no caking. Where moisture is present, liquid bridges between particles can be formed at the presence of moisture, and, hence, give rise to interparticle capillary forces. The capillary forces are formed as a result of surface tension, subject to the bridge surface curvature; hence this force contributes to the cohesive strength of the particulate solids. The formed capillary forces between particles do not themselves constitute caking forces, but are probably the initial mechanism which can cause subsequent cake formation [6] as described below. In contrast to liquid and van der Waals interactions, solid bridges make a continuous strong solid interaction between particles leading to a strong caked powder bulk [7]. Several mechanisms, i.e. dissolution-crystallisation [8], sintering [9,10] and supersaturations (Ostwald caking [11]) are responsible for solid bridges. In sintering [9], particles coalesce, by bridging between two or more particles. The coalescence of particles is driven by atomic diffusion, the system minimising the surface free energy by reducing the surface area. Other smaller particles are transported between the adjoining bigger particles in order to minimize the free surface energy of the new particle. The driving force for this process, sintering, is the difference between the capillary pressure and Laplace pressure in primary particles. Sintering is often associated with plastic deformation of the particles under pressure, increasing the interparticle surface contact; a high level of plasticity is associated with ease of relative movement of the atoms in the structure, facilitating at the same time the atomic diffusion that is thought to lead to bonding across these interparticle surfaces. This is therefore often referred to as plastic-flow caking. As particles coalesce it leads to irreversible densification and hardening of the bulk powder. In solvent evaporation [8], when the relative humidity of the surrounding environment is higher than a critical relative humidity, particles start to dissolve into adsorbed water forming liquid bridges between particles. If adsorbed water is then evaporated from these capillary bridges, dissolved solids then recrystallizes, thereby forming solid bridges between the particles. Furthermore, the liquid bridges can be supersaturated by decreasing the surrounding temperature until crystallization occurs. This caking mechanism can be seen in powders stored in closed containers where water evaporation is rare [12]. Ostwald caking [11] can also be responsible for the formation of solid bridges. In the Ostwald Caking mechanism, particles are dissolved in liquid bridges until saturated. However, particles with a small radius of curvature can dissolve in a saturated solution and make supersaturated solution. This supersaturated solution enables small particles to join, making large and harder particles in the powder, and thereby increasing the particle size and tensile strength of the powder bed. A further type of caking is due to chemical reaction either between particles, or between particles and the interstitial gas. For example, when humid air is present, cement will react with the moisture and the particles bond together due to the formation of products of reaction across the contact points [1].

Apart from the inter-particle forces, intrinsic properties of material, such as particle size and shape, cohesion, elasticity and level of hygroscopy [13], as well as extrinsic factors, i.e. level of consolidation and temperature have great influence on cake formation and needs to be addressed properly in order to avoid caking during storage and handling of the materials [13,14]. The plastic flow caking is the main focus of this research project.

The prerequisite for powder plastic caking is an increase in adhesion forces which could be related to an increase in Van der Waals forces between particles [15] augmented by likely sintering. These adhesive forces between particles determine the tensile strength of the caked powder which could be calculated through the Rumpf approach [16] modified by Molerus [17]. This model was initially developed to relate material strength, in terms of isostatic tensile stress, to the van der Waals forces, however it was also successfully applied to uniaxial state of stresses [18,19]. This approach is based on several assumptions, i.e. particles are spherical and monodispersed which is not valid for most of the powders used in industries. The other obstacles to use Rumpf approach for predicting caking strength are firstly unknown composition of the tested powder, hence it was not possible to establish the Hamaker constant, secondly measurements of the mean curvature radius at contact points as well as the plastic compressive yield strength of the material at the contact point need special technique and apparatus. In light of Rumpf approach limitation, statistical modelling for predicting and interpreting the caking strength results could be a useful approach.

A strict definition of caking is difficult to formulate, because its progression involves different stages. At any given stage, lumps may be few or numerous, of different sizes and of varying degrees of hardness. In practice, a quantitative caking measurement in each of the above stages is highly desirable [20]. There are number of different instruments and techniques for evaluating powder caking at these stages [21]. The capability of a caking tester to correctly determine the level of cake strength during storage would be the most desirable feature of such a tester. These methods are divided into several categories, for instance

a) tester based on stirred impeller (rheometer): The tester measures the vertical stress necessary to turn an impeller moving downwards through the caked sample and report it as the cake strength [22]. In another study, cake strength was reported as a torque necessary to rotate an impeller through the bed of caked powder, however this method is not suitable for powder that has undergone strong cake formation [23].

b) fluidized bed testers: This method investigates the effect of relative humidity and temperature on powder stickiness. The relative humidity of the fluidized air is gradually increased until the powder becomes stickier at a given fixed air flow rate. Extra fluidization above the sticky point of the powder is not possible due to formation of channels in the fluidized bed column. This method is useful for determining 'sticky' curve of the powder as a function of temperature and/or humidity [21]. This test determined at what humidity the powder transitions from free flowing to cohesive due to moisture effects, but does not give an indication of cake strength, or even whether caking will occur when the moisture dries out.

c) blow tester: an imposing stream of air is introduced to the bed of caked powder. The velocity of compressed air gradually increases till the point where the flow rate is sufficient to dislodge particles from the powder bed. The air velocity at which particle dislodging occurs is correlated to the cake strength of the sample [24].

d) penetration method: this method is based on probe penetration through the caked samples. Billings et al. [24] and Ozkan et al. [25] used a penetrometer technique to measure the stress needed for the penetrometer to pass through the caked sample. In another study, ball indentation technique was developed to measure the hardness of the compacted powder. This method is very useful as an indication for the onset of caking at the surface of the powder bed [26].

e) force induced uniaxial compression method: some research works have been conducted to develop a method based on the principle of a uniaxial compression test in order to break a caked sample. A modified (porous cylinder base in order to let the moisture migrate to the sample) uniaxial compression tester used to evaluate cake propensity of different powders at elevated temperature, humidity and powder consolidation. However, authors did not confirm if the powder bed reached environmental equilibrium [27,28]. Fitzpatrick et al. [29] developed force-displacement caking tester to measure the caking strength of the amorphous maltodextrin, skim milk powder and crystalline common salt at elevated humidity and storage duration. They measured caking strength by using a texture analyser as the maximum force for the rod to pass through the caked sample.

f) shear tester: this technique has been used to measure caking of particulate materials at room conditions or elevated temperature, humidity and consolidation stress [30–32]. The details of shear tester principles have been illustrated elsewhere [33]. This method is time consuming, since powder will be consolidated for a desired period of time before a single point in yield locus line can be measured.

f) tensile tester: Leaper et al [34] reported the stress necessary to pull apart the two halves of a split cylinder containing caked material as the tensile strength of the caked specimen. Wang et al. [35] used a centrifuge technique to create a tensile stress inside the bed of caked powder.

The tensile stress was determined when the powder bed start to fracture at a constant rotational speed.

The aims of this study were to develop a simple and easy to use caking tester in order to quantitatively measure the temperature dependent caking strength of a detergent powder at different temperatures, storage times and levels of consolidation stress. The main focus of this work is to determine if the developed tester could successfully measure caking strength and distinguish it between different experimental conditions. The second aim is to determine the most influential experimental variables on cake strength of the material through developing a statistical model.

Possible plastic flow caking mechanism

In this work, it is assumed that the cake strength increase occurs because of an increase in the cross-sectional area at each inter-particle contact throughout the bulk solid, through plastic deformation of the particles (creep) as a function of temperature and or pressure over a long period of time. This contact area increase will result in an increase in the bulk density (compaction), reduction in void fraction, and an increase in the tensile strength of material.

Materials

The powder used was a commercial household detergent which is sensitive to plastic flow caking. The instantaneous flow properties of the powder were measured with the Brookfield Powder Flow Tester (PFT). Flow function, i.e. unconfined yield strength values, f_c, reported as a function of the major principal stress, σ_1, during consolidation, is reported in Figure 1. The unconfined yield strength in powder shear tester is a property derived from the static yield locus through the determination of the unconfined yield Mohr circle. This parameter, therefore, is a combined function of the material cohesion, c, and also of the static angle of internal friction, ϕ [33]. These parameters are reported in Table 1. The flow functions are important because their representation is the mean by which powder flowability is usually reported and classified, according to the Jenike classification [36] or in other words according to the flow factor value, $ff = \sigma_1/f_c$. The classes generally considered are the free-flowing (ff>10), easy flowing (4<ff≤10), cohesive (2<ff≤4), very cohesive (1<ff≤2) and hardened (ff≤1). Figure 1 reports the relevant flow regions and the flow factor lines at the boundaries. For most materials, ff reduces with time under consolidation, but never exceeds a value of 1 even for the most cohesive powder unless some mechanism of force growth (such as sintering or Ostwald ripening) occurs at the particle contact points. Often for materials where such caking occurs, ff can be very much lower than 1, indicating very hard lumps.

The instantaneous Flow Function of the tested powder falls on the limit between the easy flowing and cohesive ranges, with a slight tendency towards lower flow factors at high consolidation stresses.

Greenwich Caking Tester (GCT)

A novel force displacement and easy-to-use caking tester, Greenwich Caking Tester, (GCT) for quantitatively measuring cake strength of powder was developed with some similarity to the Johanson hang- up Indicizer [37] and to the tester developed by Fitzpatrick *et al*. [15]. The sketch and dimension of the developed tester are illustrated in the Figure 2. It excels the uniaxial unconfined failure caking (UUFC) strength tester since it has a low height to diameter ratio to minimise the wall friction effect during consolidation. Furthermore, it has a defined location of the failure plane to maximise repeatability, it needs minimised sample volume and it has higher exposed surface. The GCT is designed to measure caking strength of the powder as a result of elevated temperature and consolidation stress. The developed caking tester is not suitable to evaluate caked powders with few lumps and low magnitude of hardness. The caking strength of this kind of powder can be measured by other alternative methods, such as sieving.

The GCT consisted of a 95 mm internal diameter cell with a 30 mm diameter circular hole at its centre (Figure 2b). The circular hole is closed by using the designed base (Figure 2a), in order to prevent powder falling out of the cell during caking test. A mould with the external diameter of 115 mm was used for better filling the cell (Figure 2c). Powder were poured off into the caking tester and the excess amount were scraped off. Then, the lid (Figure 2d) is placed on the powder bed and loaded with different dead weights to get the desired consolidation stress (1.9 kPa, 7.5 kPa and 21.3 kPa). The consolidated samples were then stored at elevated temperatures of 27 °C, 37 °C and 47 °C for 2, 4 and 7 days. The experimental conditions were selected based on the storage conditions of detergent powder at different locations. After storage, the base was detached from beneath of the tester, the lid was taken and then the tester centred below a plug (0.0295 m diameter, Figure 2e) attached to texture analyser to perform a cake strength measurement. Cake strength measurements were performed under quasi-static conditions. Texture analyser was programmed in a way that the plug was moved downwards without any rotation at the constant speed of 0.4 mm/s. The force necessary for the plug to penetrate and pushed out the plug of the caked sample through the cell hole was registered and reported. The circumferential area of the disc sheared out was used to calculate the stress applied to the shear plane. The registered highest stress was reported as cake strength of the powder. An example of stress-displacement curve during cake strength measurement is depicted in Figure 3.

Experimental design

Full factorial experimental designs have been conducted. The variables were temperature, *Te*, consolidation stress, *Stre*, and consolidation duration, *Dur*. Each variable considered at 3 levels that were chosen to be representative of commonplace conditions of temperature, stress and duration experienced in the shipping of these powders. This gave us 3×3×3=27 experimental conditions, in order to improve the precision of the statistical model as well as examining the suitability of the tester at different experimental conditions.. All experimental conditions were reported in Table 1 in appendix. Each experimental condition were conducted in triplicate so in total 81 tests have been performed.

Results and discussion

The cake strengths of all experimental conditions are presented in Figure 4a for 2 days storage time, Figure 4b for 4 days storage time and Figure 4c for 7 days storage time. In general, the shift in caking strength is observed, as storage time, consolidation stress and temperature increase. Furthermore, the developed tester and the approach for caking strength measurement showed its potential for correctly evaluating the caking propensity of the powder at the different experimental conditions. Repeatability of the caking strength of all samples has also been evaluated. The error bars in Figure 4 represent the standard deviation (maximum10%) between test repetitions.

Statistical model development

Explanatory variables for statistical modelling of caking strength, *Ca*, are temperature, Te, consolidation stress, *Stre*, and storage time, *Dur*. In addition, their squared values and interactions are also considered in the proposed model. The influence of explanatory variables on the response variable were compiled, modelled and analysed by multiple linear regression (MLR) method using the MODDE 12.0 software package (Umetrics, Umeå, Sweden). All values were scaled and centred before evaluation. Log transformation of the response variable is used in the model, since the caking strength could not be negative. In fact, positive outcome from the model could be attained by the data log transformation. The MLR method is applied to develop the linear relationship between log of response variable and explanatory variables. The model is defined by Equation 1:

$$\log\left(Ca_i\right) = \sum_{j=1}^{10} x_{i,j}\beta_j + \varepsilon_i \tag{1}$$

Where $x_{i,j}$ is the j^{th} explanatory variable for observation i, β_j is the corresponding coefficient and ε_i is the random error term with normal distribution. The matrix of explanatory variable for observation i is defined as:

$$\mathbf{x}_i = \begin{bmatrix} 1 & Te_i & Stre_i & Dur_i & Te_i^2 & Stre_i^2 & Dur_i^2 & Te_i \times Stre_i & Te_i \times Dur_i & Stre_i \times Dur_i \end{bmatrix} \tag{2}$$

One method of estimating the regression parameters, $\boldsymbol{\beta} = [\beta_1, ..., \beta_{10}]^T$, is Ordinary Least Squares (OLS) method. OLS shows how well a model with a particular set of parameters fits the experimental data. In this method, the regression parameters are estimated by minimizing the sum of squared errors (SSE) with respect to $\boldsymbol{\beta}$:

$$\boldsymbol{\beta}^* = \arg\min_{\boldsymbol{\beta}} SEE = \arg\min_{\boldsymbol{\beta}} \sum_{i=1}^{n} \left(\log(Ca_i) - \log\left(\hat{C}a_i\right) \right)^2 \tag{3}$$

where $\boldsymbol{\beta}^* = \left[\beta_0^*, ..., \beta_{10}^*\right]^T$ is the vector of the estimated parameters that minimize the SSE, *n* is the number of experimental points, $\log(Ca_i)$ is the response variable of the i^{th} observation and $\log\left(\hat{C}a_i\right)$ is the corresponding fitted value which calculated from $\log\left(\hat{C}a_i\right) = \sum_{j=1}^{10} x_{i,j}\beta_j$.

The total variations in the response variable come from two terms:

1) SSE which is the variation due to random errors:

$$SSE = \sum_{i=1}^{n} \left(\log(Ca_i) - \log\left(\hat{C}a_i\right) \right)^2 \tag{4}$$

2) SSR is the variations due to explanatory variables that quantifies how far the fitted values are from the response variable mean, $\log\left(\bar{C}a\right)$:

$$SSR = \sum_{i=1}^{n} \left(\log\left(\hat{C}a_i\right) - \log\left(\bar{C}a\right) \right)^2 \qquad (5)$$

where $\log\left(\bar{C}a\right) = \frac{1}{n} \sum_{i=1}^{n} \log\left(Ca_i\right)$.

According to this, R^2 is defined as a goodness of the fitted model of a linear regression:

$$R^2 = \frac{SSR}{SSTO} = 1 - \frac{SSE}{SSTO} \qquad (6)$$

where $SSTO$ is total sum of squares, $SSTO = SSE + SSR$.

Since R^2 is a proportion value, it is always a number between 0 and 1. Higher R^2 means that the higher percent of the variation in response variable is explained by the variation in the explanatory variable. The R^2 was 0.95 for the developed model. The R^2 value will increase when more terms are added to the model which finally leads to an overfitting; instead an adjusted R^2 (R_a^2) that consider the number of terms in the model could be used from equation 7.

$$R_a^2 = 1 - \left(\frac{n-1}{n-J-1} \right)\left(1 - R^2\right) \qquad (7)$$

Where J is the number of explanatory variables in the model. The adjusted R^2 value is 0.93 suggesting that the model could explain 93% of variation.

The model was also evaluated by using the *coefficient of multiple determination* (Q^2). This coefficient estimates the future predictive capability of the model. In other words, it estimates the quantity of the variance in the response variable which could be predicted, where 0 indicates no predictive ability at all and value of 1 shows the best predictive capabilities of the model. The developed MLR model had an excellent predictive ability with a Q^2 of 0.94.

Figure 5 illustrate the observed response (experimental values) against predicted response. The dotted black reference line indicates the condition where the predicted value would be the same as the observed one. In other words, plot with the points close to a reference line indicates a model with good predictive ability.

The model regression coefficients and p-values are presented in Table 2. The significance levels of p-value are 0, 0.001, 0.01 and 0.05. The p-values below these levels indicate the significance level of the coefficient. Based on this, the main variables (temperature (*Te*), consolidation stress (*Stre*), and consolidation duration (*Dur*)) are all strongly significant in the model. The square of consolidation stress and temperature are also strongly significant in the model. However, the p-values of the interaction between the main variables as well as square of duration are above the significance level and hence are not significant in the model. That means the linear relationship between these interactions and the response variable cannot be investigated.

The behaviour of the model is best demonstrated through the application of surface response contour plots. These graphs were applied to understand the relations between two independent variables on the response model. The surface response plots for the caking strength as the function of temperature and consolidation stress at different storage durations were depicted in Figure 6 (Figure 6a at 2 days storage, Figure 6b at 4 days storage and Figure 6c at 7 days storage). Plots are divided into different area, from 10 kPa to 100 kPa, where 10 kPa is the lowest measured caking strength and 100 kPa is the largest one.

Exploring the surface response plots in Figures 6a, b and c shows the caking behaviour. Taking as a critical condition the consolidation stress that leads to a low level of caking as defined by *ff*>1 (i.e. cake strength less than applied consolidating stress, the pale blue region) the limiting consolidation stress decreased from 10 kPa to 4.5 kPa (6a, 2 days duration) and from 9.5 kPa to 3.7 (6b, 4 days duration) when temperature rose from 27 °C to 47 °C. A reduction in the area beneath the lowest caking strength region is observed by comparing Figure 6a, b and c. This is an indication that the critical consolidation stress to attain low level of caking magnitude reduced continuously as a results of increased storage duration.

The highest strengths of the caked powder were measured after 7 days storage and its contour plot is depicted in Figure 6c. The lowest caking strength were predicted to happen in the consolidation stress below 7.5 kPa in temperature range of 27 °C to 45 °C. The high strength caking was measured when temperature and consolidation stress went beyond 41 °C and 16 kPa respectively. It can be seen from the red and black region, where the magnitude of caking is very high, that temperature plays an important role in defining a critical stress that leads to high level of caking. For instance, the critical stress decreased from 15.5 kPa to 12.4 kPa when temperature rose from 40 °C to 47 °C. These contour plots could be used for predicting the temperature and consolidation stress lead to the formation of caking strength with different magnitudes.

A fault with the statistical model that was generated from the surface fitting by the commercial software MODDE was that it appears to show a peak cake strength at 19kPa for all temperatures, and a reduction at higher consolidation stresses. There was no evidence in the experimental data for this, nor would it appear to be a likely real behaviour at this temperature (although extreme temperatures could cause softening of the particles leading to cake strength reduction). This fault is an artefact of the analysis, and the authors plan to explore means to avoid this effect in the model by improving the modelling techniques. Over the range up to 19kPa the surface-fit model gave a good fit to the data so the important lesson to learn may be that the model should only be used within a space that is a little smaller than the extent of the experimental data used to produce it; certainly never use it to extrapolate, and be cautious near the edges of the experimental data range.

Conclusion

A simple and easy to use caking tester has been developed for measuring caking strength when powders are exposed to various mechanical and environmental (elevated temperature) conditions. The caking tester has potential as a commercial tester due to its high repeatability, ability to measure low level of caking strength, low level of production costs, the ability to differentiate between different experimental conditions, low powder usage and high level of selectivity. The tester showed the capability to differentiate caking strengths between different experimental conditions when temperature, consolidation stress and consolidation duration varied. A statistical model was developed to describe and predict the material caking strength. The developed model showed high predictive capability with Q^2 value of 0.94. The most influential variables on the caking strength were evaluated by using the model. Consolidation stress, temperature, consolidation duration and square of consolidation stress were the most significant variables on caking strength magnitude respectively. The contour plot graphs were applied to study the effect of two independent variables (temperature and consolidation stress) on the caking propensity of samples as well as predicting the caking strength at different experimental conditions. The developed approach is useful when determining powder shelf life as a function of temperature and consolidation stress in order to avoid powder caking.

The practical advantages of this tester compared with the popular previous approach of using a cylindrical uniaxial compression failure test for cake strength [38] have been investigated by the authors and will also be published shortly.

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Nomenclature

c	cohesion, Pa
Ca_i	response variable of the i^{th} observation
$\hat{C}a_i$	model fitted value
$\bar{C}a$	response variable mean
f_c	unconfined yield strength, Pa
ff	flow factor, -
J	number of explanatory variables in the model
n	number of experimental points, -
R^2	coefficient of determination, -
Q^2	coefficient of multiple determination, -

Greek symbols

$\boldsymbol{\beta}$	**vector** regression parameters
$\boldsymbol{\beta}^*$	vector of the estimated regression parameters
β_1	model intercept
β_2	coefficient of temperature
β_3	coefficient of consolidation stress
β_4	coefficient of consolidation duration
β_5	coefficient for the interaction terms of squared temperature
β_6	coefficient for the interaction terms of squared consolidation stress
β_7	coefficient for the interaction terms of squared consolidation duration
β_8	coefficient for the interaction terms of temperature and consolidation stress
β_9	coefficient for the interaction terms of temperature and consolidation duration
β_{10}	coefficient for the interaction terms of consolidation stress and consolidation duration
ε	random error
ρ_b	bulk density, kg m^{-3}

ϕ	static angle of internal friction, degree
ϕ_e	effective angle of internal friction, degree
σ_1	major principal stress, Pa
σ_{c}	normal consolidation stress during pre-shear phase, Pa
σ_{E}	normal stress during shear phase, Pa

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Table 1. Flow properties of the tested powder

σ_E	φ_e	φ	C	ρ_b
0.977	38.4	32.4	0.112	594
1.944	38	32.8	0.191	621
2.908	38.6	32.9	0.315	638
3.373	39.8	32.7	0.507	655
4.839	40.9	32.5	0.746	672

Table 2. Effects for the scaled and cantered factors in the response model coefficients.

Coefficients	Coefficients value	p-value
Temperature (Te)	0.263	$7.057*10^{-31}$
Consolidation stress (Dur)	0.534	$1.715*10^{-15}$
Consolidation duration (Stre)	0.118	$1.716*10^{-13}$
Te^2	-0.128	$1.774*10^{-7}$
$Stre^2$	-0.348	$2.309*10^{-19}$
Dur^2	0.025	0.284
*Te*Stre*	-0.025	0.096
*Te*Dur*	0.015	0.314
*Dur*Stre*	-0.026	0.080

Figure 1. Flow function of the powder

Figure 2. Developed caking tester dimension a) cell base, b) cell, c) mould, d) lid and e) plug

Figure 3. An example of stress displacement curve during cake strength measurement

Figure 4. Caking strength of the bulk powder after a) 2 days; b) 4 days and c) 7 days storage at different consolidation stresses and temperatures.

Figure 5. Observed vs. Predicted values for the regression model

Figure 6. Surface response contour plots for powder caking strength at different storage duration a) 2 days; b) 4 days and c) 7 days based on the developed MLR model showing the combined effect of varying the consolidation stress and temperature.

Highlights

- A new caking tester has been developed to measure powder plastic caking.
- Tester could distinguish caking strength between experimental conditions.
- The tester overcomes the limitation of traditional uniaxial caking tester.
- A statistical model has been developed.
- Proposed approach could be used to determine the suitable storage condition.