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Discrete element modelling of the quasi-static uniaxial compression of individual infant formula agglomerates

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Abstract Infant formula is usually produced in an agglomerated powder form. These agglomerates are subjected to many transient forces following their manufacture. These can be difficult to quantify experimentally because of their small magnitudes and short durations. Numerical models have the potential to address this gap in the experimental data. The objective of the research described here was to calibrate a discrete element model for these agglomerates using experimental data obtained for quasi-static loading, and to use this model to study the mechanics of the particle response in detail. The Taguchi method was previously proposed as a viable calibration approach for discrete element models. In this work, the method was assessed for calibration of the model parameters (e.g., bond stiffnesses and strengths) considering three responses: the force at failure, strain at failure and agglomerate stiffness. The Weibull moduli for the simulation results and the experimental data were almost identical following calibration and the 37\% characteristic stresses were similar. An analysis of the energy terms in the model provided useful insight into the model response. The bond energy and the normal force exerted on the platens were strongly correlated, and bond breakage events coincided with the highest energy dissipation rates.

Keywords Discrete element modeling; Uniaxial compression; Quasi-static crushing; Simulation; Granular materials; Taguchi methods

1. Introduction

Infant formula is a substitute for human milk which is commonly produced as an agglomerated powder using a spray drying process. Once formed, these agglomerates are subjected to many collisions, both with other agglomerates and with the inner walls of equipment, as they are conveyed within the manufacturing plant. This can cause high levels of agglomerate breakage: an undesirable process potentially causing bulk densities to fail to comply with specifications. The forces experienced by individual agglomerates at any instant are very difficult to quantify experimentally due to their small magnitudes and short durations. As a result, it is seldom possible to conduct a thorough experimental investigation of the response of agglomerates when subjected
to such dynamic loads. For example, Hanley, Cronin, et al. (2011) subjected individual agglomerates to mechanical loading using both quasi-static uniaxial compression tests and dynamic drop tests. Data such as forces and strains at failure, agglomerate stiffnesses and coefficients of restitution were obtained using this approach. However, no insight could be obtained into the failure mechanism of the agglomerates or the evolution of their internal geometric structure during loading.

Discrete element modelling (DEM) is a numerical simulation technique that can be used to study the mechanics of the agglomerate response in detail. The DEM algorithm was originally proposed by Cundall and Strack (1979), and has often been used to simulate the uniaxial compression of agglomerates, particularly of soil particles (Cheng, Nakata, & Bolton, 2003; Golchert, Moreno, Ghadiri, & Litster, 2004; Robertson & Bolton, 2001) although other materials have also been simulated (Antonyuk, Palis, & Heinrich, 2011; Khanal, Schubert, & Tomas, 2005; Samimi, Hassanpour, & Ghadiri, 2005). DEM is capable of providing detailed information about the evolution of the internal geometric structure of an agglomerate over time. The algorithm adopts a soft contact approach, so that the particles are modelled as rigid bodies which are permitted to overlap at the contact points to simulate compressive contact deformation. A suitable contact model is necessary to relate the inter-particle force with the overlap. As described in earlier studies (e.g., Thornton & Liu, 2004), virtual bonds can be included between spheres to create agglomerates. Agglomerate breakage can occur when these bonds fail. It must be noted that the DEM of an agglomerate is an idealisation of the real physical material, as is the case for any such numerical model. Many of the model input parameters cannot be measured easily in a physical test. Hanley, O'Sullivan, Oliveira, Cronin and Byrne (2011) proposed applying the Taguchi approach to DEM calibration and demonstrated the potential of this method using synthetic data. This established Design of Experiments (DOE) method seeks to find the relationship between the system parameters and the output by using a structured pre-planned methodology for obtaining experimental data. It is often applied to efficiently find the optimum combination of parameter settings from all of those tested based on some user-defined criteria. In this paper, the real utility of the Taguchi method is highlighted by calibrating the DEM using experimental uniaxial compression data obtained by Hanley, Cronin, et al. (2011).

This paper firstly describes the physical tests that were used to characterise the behaviour of infant formula agglomerates. The approach taken to calibrate the DEM is described and it is demonstrated that the Taguchi method is suitable for calibration using real experimental data. The ability of the calibrated model to simulate quasi-static compression of infant formula agglomerates was evaluated and key energy terms were tracked throughout the compression process to provide detailed information about the mechanics of the model response.
2. Model calibration

2.1. Uniaxial compression tests

The infant formula considered in the study was a commercial spray-dried powder which contained 56.1% carbohydrate, 22.1% fat, 15.4% protein, 4.4% ash and 2% moisture. The agglomerates tested for all experiments were taken from the sieve fraction between 710 µm and 850 µm. While full details of the uniaxial compression tests are given by Hanley, Cronin, et al. (2011), an overview is provided here so that the calibration process can be understood.

Uniaxial compression tests are widely used as a measure of particle strength. The experimental data presented in this paper were obtained using a Stable Micro Systems TA.HDplus texture analyser with a 5 kg (≈ 49 N) load cell (Stable Micro Systems Ltd., Godalming, Surrey, UK). A total of 457 agglomerates were compressed individually on a flat, glass plate at a constant loading rate of 10 µm/s until a force of 0.29 N was attained (equivalent to a mass of 30 g). An algorithm was implemented in MATLAB (v.7.0.1, The MathWorks, Natick, MA, USA) to obtain three responses from each set of raw data:

1. the normal force at the point of failure of the agglomerate (N);
2. the strain at the point of failure of the agglomerate (%); and
3. the agglomerate stiffness (N/m). Note that the stiffness of both the physical and simulated agglomerates was defined as the slope of the linear region prior to the point of failure on a plot of force versus deflection.

2.2. Agglomerate structure

Certain physical characteristics of the agglomerates were measured to create the discrete element model. The average length and width of the agglomerates were obtained using a Malvern PharmaVision 830 instrument (Malvern Instruments Limited, Malvern, Worcestershire, UK). This instrument contains a camera, controlled by means of an actuator, which scans slowly over particles at rest on a glass plate. Ninety agglomerates in total were analysed using this apparatus. The height of each agglomerate was found directly from the texture analyser as the distance between the bottom of the platen and the top surface of the glass plate at the instant when the instrument detected contact with the agglomerate. The length and width of each agglomerate were calculated using an algorithm which involved fitting a bounding ellipse around the projected area of the agglomerate (Malvern Instruments Ltd., 2004). The mean length, width and height of the agglomerates were 1212, 937 and 678 µm, respectively.

A Carl Zeiss Gemini Supra 40VP field emission SEM (Carl Zeiss SMT AG, Oberkochen, Germany) was used to obtain 37 scanning electron microscope (SEM) micrographs of
agglomerates of infant formula in the 710–850 µm size range. Samples of infant formula for analysis were affixed to aluminium stubs using double-sided carbon tape and sputter-coated with a thin layer of chromium using an Emitech K550X coater (Quorum Technologies Ltd., Ashford, Kent, UK). The image analysis software ImageJ (v.1.43u, U.S. National Institutes of Health, Bethesda, MD, USA) was used to measure the approximate diameters of the particles at the surface of the agglomerates, which were assumed to be representative of all particles comprising the agglomerates. A lognormal distribution was fitted to the measured particle diameter data. The mean particle diameter was 48 µm, with a standard deviation of 21 µm. The normalised bond radii (a required simulation parameter) were estimated by dividing the length of the overlap between two particles by the smaller of the particle diameters. The diameter of each particle was estimated manually for a circular equivalent projected area. This is shown in Fig. 1, for which the normalised bond radius was 0.648 (= 31.6/48.8). The mean of the values recorded was 0.711 (standard deviation of 0.154).

![Figure 1: SEM micrograph of an infant formula agglomerate (2000 x magnification) which illustrates the methodology used to measure particle diameters and normalised bond radii.](image)

2.3. Simulation methodology for uniaxial compression

Rigid spheres were selected as the base particles in the 3D DEM simulations for computational efficiency. The agglomerates required were produced from these spheres by sequential addition. One seed sphere was placed at the origin. Other spheres were then placed successively and randomly within an ellipsoidal volume surrounding this seed sphere. Each active sphere was moved randomly until it came into contact with another sphere, when it was fixed in position (ensuring no overlaps). The random increments of displacement were modified by point transformation to continuously reduce the distance between each active sphere and the origin until
contact occurred with a pre-existing sphere. Russ (1994) describes this method of depositing particles on a surface as “diffusion-limited aggregation”, which results in very open, dendritic structures. This algorithm was implemented in MATLAB and the particle positions and radii were written to a text file before being imported to the commercial DEM software package PFC3D (Particle Flow Code in 3 Dimensions, v.4.0, Itasca Consulting Group, Minneapolis, MN, USA). This package was used to conduct all of the simulations described in this paper.

A standard linear contact model was used and the spheres were bonded together using parallel bonds. These bonds transmit both forces and moments (Potyondy & Cundall, 2004), and were added after the particle positions had been defined. The specification of parallel bonds requires five parameters in the software: the bond radius multiplier ($\lambda$), normal and shear strengths, and normal and shear stiffnesses. The strengths and stiffnesses are input in units of stress rather than force, and the bond failure criterion, based on beam bending theory, takes into account both the force and moment transmitted at the bonded contact. The bond radius is calculated as the product of the smaller of the two particles interacting in the bond and $\lambda$. Other parameters required in the DEM included the surface friction and density of the spheres, their normal and shear stiffnesses, and the local damping coefficient. Local damping adds a damping-force term to the equations of motion: this term opposes motion, is not velocity-dependant (unlike viscous damping) and is proportional to the local damping coefficient (Itasca Consulting Group, 2008). As mentioned in the introduction, many of these parameters are difficult to measure accurately; for this work, appropriate values for all these parameters were found using a calibration process.

The diameters of the rigid spheres were randomly selected from a lognormal distribution using the parameters obtained experimentally. The sphere diameters were restricted to the range 20–125 µm to prevent particles with unrealistic diameters from being generated. Agglomerate dimensions were subject to some variability; however, these were generally within 10% of the mean values measured experimentally. The mean number of spheres in an agglomerate was 688, with a standard deviation of 107. Fig. 2 shows an example of an agglomerate generated by this approach.
Figure 2: Generated agglomerate containing 738 spheres, which has a length of 1.27 mm, width of 1.06 mm and height of 0.75 mm. The colour bar indicates the size of the spheres in mm.

Each agglomerate was individually compressed between stiff, horizontal, frictionless platens until a strain of 50% was attained. The bottom platen was kept stationary and the upper platen approached it at 0.04 mm/s. This velocity was sufficiently low to ensure that the loading was quasi-static. The initial distance between the platens was equal to the agglomerate height.

2.4. DEM calibration

The Taguchi method was used to determine those DEM parameters which could not be obtained by experiment. This approach was proposed by Hanley, O’Sullivan, et al. (2011) and demonstrated to be suitable for this application, although this study did not consider real, experimental data. In this paper, the applicability of the method is evaluated using physical test data. A general introduction to the Taguchi method is given by Ross (1988). The Taguchi method makes use of orthogonal arrays which provide the required combination of factor levels for each trial; these arrays are designated as $L_n$, where $n$ is the required number of trials. An initial parameter study was conducted using an $L_{18}$ array which identified the bond strengths and stiffnesses as being particularly influential. Several sets of simulations were then conducted using saturated $L_9$ arrays. The four available columns were allocated to the parallel bond strengths and stiffnesses, and levels were varied by orders of magnitude. The bond radius multiplier was set at 0.7, approximately equal to the mean recorded for the physical agglomerates. The values of all of the remaining parameters were fixed temporarily. One standard agglomerate was used for these simulations, which had the aim of minimising the differences between three DEM responses (the force at failure, strain at failure and agglomerate stiffness) and the equivalent mean experimental results.

Once the physical tests and numerical simulations were in reasonable agreement, appropriate settings needed to be identified for the remaining parameters: particle density, particle friction,
particle normal and shear stiffnesses, and the local damping coefficient. At this stage, multiple agglomerates were introduced to ensure that parameters were widely applicable and to mitigate against the chosen reference agglomeration being atypical in some regard. After these simulations were completed, the responses considered corresponded quite well to the experimental means. However, no consideration had yet been given to the shape of the distributions. The only way of checking this was to obtain sufficient data to yield representative distributions of the three responses which necessitated running a larger number of simulations. Initially 20 simulations were run using identical parameters, each using a different randomly-generated agglomeration. The mean strain at failure was too high; the results obtained from the initial $L_{18}$ array indicated that this could be reduced by adjusting the particle friction. A larger set of 130 simulations were run using these updated parameters. As the agglomerate stiffnesses remained excessive, the bond stiffnesses were reduced to give the calibrated set of parameters given in Table 1. These parameters were used to obtain all of the results presented in this paper. It should be emphasised that the geometric variability of the simulated agglomerates made calibration of the model both difficult and time-consuming.

### Table 1: Calibrated parameters used for the DEM simulations

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Calibrated value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle density ($\text{kg/m}^3$)</td>
<td>$1 \times 10^8$</td>
</tr>
<tr>
<td>Particle normal stiffness (N/m)</td>
<td>$4 \times 10^7$</td>
</tr>
<tr>
<td>Particle shear stiffness (N/m)</td>
<td>$4 \times 10^7$</td>
</tr>
<tr>
<td>Particle friction</td>
<td>0.65</td>
</tr>
<tr>
<td>Bond radius multiplier</td>
<td>0.7</td>
</tr>
<tr>
<td>Bond normal strength ($\text{N/m}^2$)</td>
<td>$1 \times 10^9$</td>
</tr>
<tr>
<td>Bond shear strength ($\text{N/m}^2$)</td>
<td>$1 \times 10^9$</td>
</tr>
<tr>
<td>Bond normal stiffness ($\text{N/m}^3$)</td>
<td>$2.4 \times 10^{15}$</td>
</tr>
<tr>
<td>Bond shear stiffness ($\text{N/m}^3$)</td>
<td>$2.4 \times 10^{15}$</td>
</tr>
<tr>
<td>Local damping coefficient</td>
<td>0.3</td>
</tr>
</tbody>
</table>

*a* Note that density scaling was used (Section 2.6)

### 2.5. Model sensitivity to small perturbations

Simulations demonstrated that the model was highly sensitive to small perturbations in the input parameters, i.e., changing one of the parameters in Table 1 by a slight amount, e.g., 1%, could cause the responses of the simulations to change completely, demonstrating a high level of complexity in the structure of these agglomerates. However, the initial low-strain behaviour was unaffected by perturbations, that is, the responses were found to deviate only after some strain value which differed for each agglomerate. This is demonstrated in Fig. 3 which compares the average force on the platens obtained for one agglomerate using (a) the bond stiffnesses or (b) the bond strengths given in Table 1 with the equivalent responses using perturbations of ± 1%. The responses on Fig. 3 are identical up to a strain of around 12%, after which differences become apparent between the output plots.
Figure 3: Plots comparing the average normal force exerted on the platens (N) against axial strain (%) for uniaxial compression of one agglomerate conducted using the settings given in Table 1 except for small perturbations in (a) the parallel bond normal and shear stiffnesses and (b) the bond normal and shear strengths.

2.6. Density scaling

The quasi-static simulations of particle compression used density scaling: by increasing the particle density to physically-unrealistic values, the time required to run each simulation was greatly reduced. This technique has been widely used by other researchers (e.g., Sheng, Lawrence, Briscoe, & Thornton, 2004; Sykut, Molenda, & Horabik, 2008; Thornton, 2000). It was considered acceptable to increase the density in this manner because no gravitational fields were active in the simulations. Fig. 4 illustrates this point for one representative agglomerate using very large perturbations in particle density which differed by orders of magnitude. By comparing Figs. 3 and 4, it can be seen that very large changes in particle density have no more of an effect than slight perturbations in other simulation parameters.

Figure 4: Plots of the average normal force exerted on the platens (N) against axial strain (%) for three simulations conducted using particle densities which varied by orders of magnitude (from $1 \times 10^6$ to $1 \times 10^8$ kg/m$^3$), where all other parameters were held constant.
3. Comparison of DEM and experimental results for quasi-static compression

3.1. Probability histograms and density functions

A total of 130 simulations were conducted using a different randomly generated agglomerate geometry in each case and the parameters given in Table 1. Probability histograms of the DEM force at failure, strain at failure and agglomerate stiffness responses are shown in Fig. 5. The corresponding probability density functions (PDFs) of lognormal distributions fitted to the simulation data and to the experimental results are superimposed on each histogram as solid red and dashed green lines, respectively. Considering the force at failure, the simulation results corresponded exactly to the experimental results. The simulation results for the strain at failure were somewhat skewed compared to the experimental results: for 30% of the simulations, the strains at failure exceeded 25% whereas the same was true for only 14% of the physical agglomerates tested. For the stiffness, the simulation results exhibited a marked skew towards smaller stiffnesses, yet the distribution was also long-tailed; 59% of the simulations had stiffnesses below 5 kN/m while 8% of the agglomerates had stiffnesses exceeding 20 kN/m. The means and standard deviations of the DEM results are compared with those of the physical agglomerates in Table 2.

Figure 5: Probability histograms of the responses obtained from the DEM simulations for (a) force at failure (N), (b) strain at failure (%) and (c) agglomerate stiffness (N/m), with the corresponding PDFs of lognormal distributions fitted to the simulation data and to the experimental results shown in red and green respectively for comparison.
Table 2: Comparison of the means and standard deviations of the results obtained for force at failure (N), strain at failure (%) and agglomerate stiffness (N/m) for the physical agglomerates of infant formula subjected to uniaxial compression and the equivalent DEM simulations

<table>
<thead>
<tr>
<th>Response</th>
<th>Physical</th>
<th>Simulated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force at failure (N)</td>
<td>Mean</td>
<td>0.0714</td>
</tr>
<tr>
<td></td>
<td>Std. dev.</td>
<td>0.0528</td>
</tr>
<tr>
<td>Strain at failure (%)</td>
<td>Mean</td>
<td>14.409</td>
</tr>
<tr>
<td></td>
<td>Std. dev.</td>
<td>10.433</td>
</tr>
<tr>
<td>Agglomerate stiffness (N/m)</td>
<td>Mean</td>
<td>4648.5</td>
</tr>
<tr>
<td></td>
<td>Std. dev.</td>
<td>2921.2</td>
</tr>
</tbody>
</table>

3.2. Weibull analysis

The experimental results and simulation outputs were also compared by applying Weibull statistics to the data. Eq. (1) is the Weibull equation for the case of agglomerate compression, in which $P_s$ is the survival probability for agglomerates of size $d$ when exposed to a uniform tensile stress $\sigma$, $\sigma_o$ is the characteristic stress at which 37% ($e^{-1}$) of such agglomerates survive and the exponent $m$ is the Weibull modulus (McDowell & Amon, 2000).

$$P_s(d) = e^{-\left(\frac{\sigma}{\sigma_o}\right)^m}.$$  (1)

For this work, $d$ was specified to be the distance between the compression platens at the point of failure of the agglomerate (McDowell & Amon, 2000). Eq. (1) may be rewritten in the form of Eq. (2) by applying logarithms:

$$\ln\left[\ln\left(\frac{1}{P_s(d)}\right)\right] = m\ln\left(\frac{\sigma}{\sigma_o}\right),$$  (2)

where $\sigma$, the tensile stress at failure, was approximated as a function of $F$, the force at failure, using Eq. (3) (McDowell & Bolton, 1998):

$$\sigma = \frac{F}{d^2}.$$  (3)

The survival probability, $P_s$, was calculated using the mean rank position approach (Cheng et al., 2003):

$$P_s(d) = 1 - \frac{i}{n+1},$$  (4)
where \( n \) is the total number of data points for \( \sigma \) and \( i \) is the index of a particular agglomerate when ordered by increasing \( \sigma \). Hence, the agglomerate for which \( i = 1 \) has a survival probability which is close to unity, assuming a reasonable population size, and the lowest \( \sigma \) of all \( n \) agglomerates.

Based on Eq. (2), \( \ln \left( \frac{1}{P_s(d)} \right) \) was plotted against \( \ln(\sigma) \) for both the experimental and simulation data and a linear regression trendline was fitted to each set of data (Fig. 6). The slope of each trendline is the Weibull modulus, \( m \), and \( \sigma_o \) was found by taking the exponential function of the absolute value of the y-intercept, following division by \( m \).

**Figure 6: Comparison of Weibull plots for the infant formula agglomerates tested experimentally and the simulated DEM agglomerates.**

Both plots in Fig. 6 have a similar appearance and the linear trendlines are almost parallel. The Weibull moduli were extremely close: 1.495 for the DEM results and 1.494 for the experimental compression data. The 37\% characteristic stresses were also similar: 281 kN/m\(^2\) for the simulations compared to 257 kN/m\(^2\) for the physical agglomerates. Both \( R^2 \) values were above 0.9 (0.97 for the simulations and 0.92 for the experimental agglomerates) which indicates that these data sets were well described by a linear model and hence, that Weibull statistics are appropriate for these agglomerates.

4. DEM energy and failure mode analysis

The good agreement between the Weibull data from the physical tests and the results of the numerical simulations provides strong evidence that the model calibration was successful. In order to have complete confidence that the DEM accurately captures the micromechanics of the agglomerate response, it would be necessary to develop a model which is far more sophisticated; however, considering the mechanical response of the agglomerates in terms of energy leads to an
improved understanding of the model itself, which in turn may provide some insights into the response of real agglomerates.

4.1. Characterisation of the mechanical response using energy

The data available within a DEM code allow calculation of the various components of mechanical energy within the system. PFC (Particle Flow Code) facilitates the tracking of six energy terms (Itasca Consulting Group, 2008):

1. Body work: the total accumulated work done by all body forces on the assembly;
2. Bond energy: the total strain energy of the assembly stored in the parallel bonds;
3. Boundary work: the total accumulated work done by all walls on the assembly;
4. Frictional work: the total energy dissipated by frictional sliding at all contacts;
5. Kinetic energy: the kinetic energy of all bodies in the assembly;
6. Strain energy: the total strain energy of the assembly stored at all contacts.

Only three of these terms (bond energy, frictional work and boundary work) were of relevance for the quasi-static simulations as gravity was inactive and the kinetic energy and strain energy were both negligible. Fig. 7(a) shows the evolution of the significant energy terms during compression of three representative agglomerates to strains of 50%. The cumulative energy dissipated and the energy dissipated from the bonds are also shown for comparison. The strain energy is the energy stored in the contact springs that exist at unbonded contacts as well as bonded contacts (where these contact springs act in parallel with the parallel bonds), and is calculated for both the normal and tangential springs. This term remained low (< 1 µJ) during the agglomerate compression. The only source of energy input to the system was boundary work, which was approximately equal to the energy dissipated. In all cases, more energy was dissipated by bond breakage and deformation than by friction, although both were significant.
Figure 7: Plots for uniaxial compression of three representative agglomerates showing (a) five energy terms (µJ) against axial strain (%) and (b) the normalised average normal force on the platens and three normalised tracked energy components (boundary work, frictional work and bond energy) for the first 10% of axial strain.

It was instructive to focus on a restricted region of strain in greater detail. Fig. 7(b) examines the same three agglomerates as Fig. 7(a), in which the average normal force on the platens and all energy terms were normalised by their maximum values. The trends in bond energy and normal force were very similar: whenever the force on the platens decreased sharply, the strain energy stored in the parallel bonds also decreased. The frictional work increased accordingly at these points, indicating that part of the agglomerate moved relative to another contacting part. A strong positive correlation existed between bond energy and normal force: the mean correlation coefficient was 0.860 (standard deviation of 0.104) when calculated for all simulated agglomerates. The rate of change of the boundary work was highest immediately before the local maxima of force or bond energy and was lowest following the points of rapid energy dissipation. As the compression was strain-controlled, this increase in the rate of boundary work corresponds to an increase in the increment of resistive force provided by the agglomerate just before failure.

Fig. 7(b) indicates that sharp decreases in the normal force coincide with high rates of change of the frictional work, and this is confirmed in Fig. 8(a). The reason for these high rates of energy dissipation is relative motion between contacting parts of the agglomerate. This microstructural rearrangement also causes the sharp reduction observed in the normal force. Fig. 8(b) plots the number of bonds failed and the derivative of dissipated energy against axial strain for the same three representative agglomerates. This figure demonstrates that the points at which energy dissipation rates were high coincided with bond breakage events, confirming the dominant effect of bond rupture, rather than bond deformation without failure, on the system response. For example, Fig. 8(b) shows that the sharp decrease in force and bond energy (and increase in
frictional work) at 3.3% strain for the first agglomerate in Fig. 7(b) was caused by the failure of only one parallel bond.

**Figure 8:** Plots for uniaxial compression of three representative agglomerates showing (a) the average normal force on the platens (N) and the derivative of frictional work (J/m) against axial strain (%) and (b) the number of bonds failed and the derivative of dissipated energy (J/m) against axial strain (%).

There were very strong correlations between these energy terms specifically at the point of failure of the agglomerates. Figs. 9(a) and 9(c) show that linear relationships exist between the frictional work or bond energy dissipated, and the boundary work when all energy terms were quantified at the point of failure of the agglomerate. However, there is no discernable relationship between the bond energy stored at the point of failure and the total boundary work at the failure point (Fig. 9(b)). The linear relationship between boundary work and dissipated energy presented in Fig. 9(d) is to be expected since the sole energy input is boundary work, which is approximately equal to the energy dissipated from the system at all strains, i.e., relatively little energy is stored in the system.
Figure 9: Four plots of energy terms at the point of failure against the boundary work at failure (both in µJ) for uniaxial compression of all simulated agglomerates. (a) shows the frictional work, (b) the bond energy, (c) the bond energy dissipated and (d) the total energy dissipated. Linear trendlines are shown for three of the plots.

4.2. Mode of failure of the agglomerates

Fig. 8(b) shows that bonds were failing as the agglomerates were compressed and that the macro-scale response is directly related to bond ruptures. The total number of bonds which failed could be subdivided by the type of failure, i.e., whether the bond failed as the result of its normal or shear strength being exceeded. In fact, very few of the bonds failures (< 2%) were caused by excessive shear stresses being generated. The progressive breakage of parallel bonds with increasing axial strain caused the agglomerates to break into multiple daughter agglomerates. The total number of agglomerate fragments were recorded at each 0.5% increment of strain for each simulation. Fig. 10(a) presents these data for each agglomerate as a thin grey line, with the median response shown as a thicker blue line. Each particle fragments into a relatively large number of daughter agglomerates as the compression progresses, although there is a large amount of scatter in the data. Fig. 10(b) presents the corresponding data for the size of the largest daughter agglomerate present in the system at each increment of strain. This data appears to show some convergence, with the largest daughter agglomerate at 50% strain containing between 54 and 189 spheres (median of 99), a huge reduction from a median of almost 700 before compression. Clearly the specific geometry of each agglomerate governs the number of bond breakages, but
each agglomerate contains a relatively robust cluster of between 50 and 200 bonded particles that resists fracture, even at a strain level of 50%.

![Figure 10](image_url)

**Figure 10:** Plots of (a) the median number of agglomerate fragments and (b) the median number of spheres remaining in the largest intact agglomerate fragment against axial strain (%) for all simulated agglomerates subjected to uniaxial compression, where the data for each agglomerate are shown as a light grey line.

## 5. Conclusions

The quasi-static, uniaxial compression of individual infant formula agglomerates was simulated using the discrete element method. The model was calibrated using experimental data: a difficult process since it was necessary for the simulated agglomerates to capture the large variability apparent in the natural agglomerates. Hanley, O’Sullivan, et al. (2011) proposed a DEM calibration approach based on the Taguchi method of experimental design. In this paper, the method was shown to be effective when applied to real, physical test data. The final calibrated parameters included particle and parallel bond stiffnesses of $4 \times 10^7$ N/m and $2.4 \times 10^{15}$ N/m$^3$, respectively, and parallel bond strengths of $1 \times 10^9$ N/m$^2$. The mean force at failure, strain at failure and agglomerate stiffness were 0.075 N, 19.3% and 7.00 kN/m, respectively, while the corresponding means obtained by experiment for the physical agglomerates were, respectively, 0.071 N, 14.4% and 4.64 kN/m. When lognormal distributions were fitted to the experimental and DEM results and their probability density functions were compared, the results for force at failure were very close. While the DEM results for strain at failure and stiffness were skewed compared to the experimental results, they still gave a good representation of the experimental data. The Weibull moduli for the simulation results and the experimental data were almost identical. An analysis of the key energy terms during the simulations shows that the bond energy and the normal force exerted on the platens were strongly correlated, and bond breakage events coincided with large energy dissipation rates. There were clear linear relationships between both the frictional work and boundary work at failure, and the bond energy dissipated and boundary work at the point of failure of the agglomerate. It should be noted that since this model was calibrated using solely
quasi-static compression data, it is unlikely to be applicable to dynamic loading conditions without some modification.

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**Nomenclature**

\( d \) a suitable measure of agglomerate size, taken as the distance between the compression platens at the point of failure of the agglomerate, m

\( F \) force at failure for agglomerate compression, N

\( i \) index of any particular agglomerate when ordered by increasing \( \sigma \)

\( m \) Weibull modulus

\( n \) total number of data points for \( \sigma \)

\( P_s \) survival probability of an agglomerate

\( \lambda \) bond radius multiplier in the parallel bond model

\( \sigma \) uniform tensile stress exerted on an agglomerate, N/m²

\( \sigma_0 \) characteristic stress at which 37% of agglomerates survive when loaded by a stress \( \sigma \), N/m²

**References**


