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Title

Effect of composition on the mechanical response of agglomerates of infant formulae

{Short title: Effect of composition on the mechanical response of infant formula agglomerates}

Authors

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Abstract

The objective of this paper is to investigate the relationship between the compositions of four typical infant formulae, the evolution of key quality characteristics during pneumatic conveying and the mechanical properties of individual agglomerates. Conveying experiments were conducted using a lab-scale rig. Four quality characteristics were measured before and after conveying: bulk density, volume mean diameter, wettability and percentage free fat. Relative breakages were calculated from particle size distributions. Uniaxial compression experiments were performed on individual agglomerates, giving forces and strains at failure and agglomerate stiffnesses. Coefficients of restitution of the agglomerates were obtained by video analyses of drop tests. The data indicate that bulk densities before conveying and the force and the strain of individual agglomerates at failure were related to the protein content. The force at failure and agglomerate stiffness were strongly correlated, and generally increased with increasing protein to fat ratio while the strain at failure decreased.
Keywords
attrition, drop tests, infant formula, pneumatic conveying, relative breakage, uniaxial compression

1) Introduction
Infant formula is a substitute for human milk which is mostly produced in a powder format using a spray drying process. Agglomeration is widely used to enhance the reconstitution properties of dried powders such as infant formulae (Ortega-Rivas, 2009). Breakage of these agglomerates during in-plant handling and transport is undesirable: implications range from deterioration of the product’s instant properties such as wettability (Hogekamp and Schubert, 2003), to failure of product bulk densities to comply with predetermined quality control limits (e.g., of scoop delivery). The issue of agglomerate breakage is often significant since agglomerates tend to be considerably more fragile, and therefore more liable to disintegrate under mechanical loading, than the primary particles from which they are formed.

Pneumatic conveying is commonly used for transporting infant formula to powder storage silos and packaging lines after spray drying. The
agglomerates experience many transient contacts with other agglomerates and the inner wall of the pipeline during this transport process. It is known that the composition of dairy powders affects quality characteristics such as flowability (Benkovic and Bauman, 2009; Fitzpatrick et al., 2007). However, little information is available in the literature to relate the composition of infant formulae to the macro-scale changes in key quality characteristics that occur during pneumatic conveying.

The forces experienced by individual agglomerates during conveying are very difficult to quantify experimentally. One feasible alternative is to study the breakage behaviour of single agglomerates under controlled laboratory conditions using diametrical compression. Using this approach, detailed information about the force-deformation response is provided (Adams et al., 1994; Antonyuk et al., 2005; Khanal et al., 2008), mechanical properties such as agglomerate strength and toughness may be measured (Bika et al., 2001), and expressions such as those developed by Rumpf (1962) and Kendall (1988) may be applied to relate the agglomerate strength to properties of the primary particles and inter-particle bonds (Samimi et al., 2003). Advances in technology have also made it possible to study individual agglomerates under dynamic loading by recording high-speed video footage of agglomerate impacts (e.g., Samimi et al., 2004; Subero and Ghadiri, 2001). Both the static and dynamic loading cases can provide
valuable insights into the micro-scale behaviour of the agglomerates during loading which influences the macro-scale changes in quality characteristics.

This research had two main objectives:

1) To investigate the relationship between composition and the resulting changes in key quality characteristics, such as bulk density and wettability, when subjected to conveying. The potential relationship will be assessed via pneumatic conveying trials using four formulae of differing compositions.

2) To establish whether the mechanical response characteristics of the individual infant formula agglomerates, including stiffnesses and coefficients of restitution, may be predicted by compositional trends. In order to investigate this, uniaxial compression tests and dynamic drop tests will be used.

2) Materials and Methods

2.1) Infant Formulae

Four types of infant formulae were tested in this work. The compositions of these typical formulae are shown in Table 1 in order of increasing protein to fat ratio. The size range of the agglomerates was restricted for both the
uniaxial compression and drop test experiments, both to control one of the sources of variability and to simplify agglomerate handling. The microstructure of these agglomerates is complex, but may be viewed as a continuous crystalline lactose phase in which all other components are suspended. The fat is localised in globules which are bounded by protein. A sieve shaker was used to isolate the fraction of the infant formula between 710 μm and 850 μm (sieving time of 5 min). Once isolated, the agglomerates were stored in air-tight containers with a large quantity of silica gel desiccant beads to inhibit moisture absorption.

2.2) Pneumatic Conveying Experiments

The pneumatic conveying rig used in this study was described by Hanley et al. (2011), and is illustrated in Figure 1. This rig consisted of three identical straight 650 mm lengths of 316L stainless steel pipeline, two horizontal and one vertical, joined by two 200 mm 90° bends. The nominal diameter of these rig parts was 25 mm. There was a conical expansion section near the end of the rig which doubled the diameter to 50 mm and length of 650 mm before the air stream entered a collection vessel. This vessel incorporated filters to separate the entrained powder from the air.

Dry compressed air was used for conveying and its pressure could be regulated to provide air velocities of up to 40 m/s. All the air velocity data
given in this paper are the maximum superficial air velocities, which can be measured accurately by inserting a 3 mm diameter Pitot-static tube into a small resealable hole in one horizontal length of the rig. Three air velocities were used in this study: one in the dense phase regime (4 m/s) and two in the dilute phase regime (10 m/s and 20 m/s). In the dense phase regime, the conveyed solids were distributed non-uniformly over the pipe cross-section, i.e., the solids were not fully suspended in the gas flow, which was the case for dilute phase conveying. For the latter, the infant formula was poured into the powder-loading funnel shown in Figure 1 at a constant flow rate of 1 g/s while the air flow was switched on. However, for the dense phase conveying, the funnel was removed and the aperture was sealed. A single plug of powder with a 100 mm length was created in the line by filling the first sight glass with infant formula. This plug was not compressed before conveying to avoid breakage of the powder. The rig was then reassembled and the air supply was switched on with a ball valve.

2.3) Measured Product Quality Characteristics for Conveying Experiments

Four product quality characteristics were measured:

1) Bulk density
2) D[4,3] - Volume mean diameter
3) Wettability

4) Percentage free fat

Bulk density is a crucial quality characteristic for manufacturers of infant formulae as this determines the mass of powder which fills a measuring scoop used for dispensing powder on reconstitution. This property was measured using a Stampf volumeter STAV 2003 (J. Engelsmann AG, Luwigshafen, Germany). For each measurement, 100 g of powder was weighed into the graduated cylinder and tapped 1250 times to the extreme powder bulk density (GEA Niro, 2006). The volume mean diameter (D[4,3]) was obtained from laser diffraction using a Malvern Mastersizer S with dry powder feeder (Malvern Instruments Limited, Malvern, Worcestershire, UK). Wettability was measured according to the GEA Niro Analytical Method (GEA Niro, 2009). Note that low wettability times indicate good wettability behaviour, and vice-versa.

The percentage free fat of the infant formulae was determined using the relevant GEA Niro Analytical Method (GEA Niro, 2005). Free fat is an important property for infant formula manufacturers: if it is excessively high, a fatty layer would appear on the surface of the reconstituted formula which is organoleptically undesirable. The free fat was measured by agitating 10 g of infant formula with 50 ml of petroleum ether for 15
minutes using a Stuart SF1 flask shaker (Bibby Scientific Limited, Stone, Staffordshire, UK), filtering the resulting solution through Whatman Grade 113 filter paper and evaporating 25 ml of the filtrate to dryness. Most of the ether was evaporated in a fume hood before the samples were dried in an oven at 105°C for 90 minutes: a more rigorous criterion than the one hour drying time required by the standard (GEA Niro, 2005). The mass of the residue was then measured.

All pneumatic conveying trials were conducted three times. The percentage free fat was measured for only one replicate, while the other three quality characteristics were measured for all replicates. For each replicate, bulk density and wettability were measured once, giving three measurements in total for each pneumatic conveying trial. D[4,3] was measured twice for each replicate, giving six measurements for each trial. Free fat measurements were carried out in duplicate to allow the variability in the measurement to be estimated. In all cases, the order of the trials was completely randomised to minimise the bias in the results.

2.4) Relative Breakage

Rather than using the change in D[4,3] (or any similar definition of average particle size) to quantify particle breakage, the approach proposed by Hardin (1985) which gives insight into the breakage which occurs in different size
fractions of the material under loading was used. This method for quantifying breakage using particle size distributions is commonly used in the field of soil mechanics (e.g., Coop et al, 2004; Donohue et al, 2009). Particle size distributions are often presented as shown in Figure 2a, with percentages of the particles contained in each size category shown on a linear vertical axis. To apply Hardin’s method, it is necessary to present cumulative particle size distributions, where the vertical axis shows the percentage of the particles which is finer than the corresponding particle diameter. The particle diameter must be displayed on a logarithmic axis. This is shown in Figure 2b for the same particle size distributions as considered in Figure 2a.

Hardin defined three measures: breakage potential ($B_p$), total breakage ($B_t$) and relative breakage ($B_r$). $B_p$ and $B_t$ represent the shaded areas on Figure 3. A baseline particle size must be selected; Hardin used 74 µm although other researchers have used different values, e.g., Coop et al (2004) and Donohue et al (2009) both used 63 µm. For this work, 20 µm was chosen as measured changes in the particle size distributions of infant formulae below this threshold were negligible, even where attrition was high. $B_p$ is the area enclosed between the particle size distribution before breakage and the baseline size (Figure 3a), while $B_t$ is the area enclosed between the particle size distributions before and after breakage for particle diameters greater
than the baseline particle size (Figure 3b). Thus, $B_t$ is a measure of the actual breakage that occurs whereas $B_p$ quantifies the breakage if all the particles had final sizes below the baseline size. The relative breakage, $B_r$, may then be calculated as:

$$B_r = \frac{B_t}{B_p}$$  \hspace{1cm} \{\text{Eq. 1}\}

For the analysis of the laser diffraction data, two particle size distributions were averaged since measurements were duplicated for each of the three replicates. Thus, three values of relative breakage were obtained for each combination of air velocity and infant formula. Note that while changing the baseline particle size had a major effect on the magnitude of $B_r$, it did not significantly affect the relative trends observed for $B_r$. For example, selecting Hardin’s baseline size of 74 µm instead of 20 µm would increase $B_t$ results by 107%, on average; however, the minimum and maximum increases would be 74% and 139%. Thus, choosing a different baseline size caused the results to scale proportionally for the infant formulae tested.

2.5) Texture Analysis and Compression Methodology

The uniaxial compression experiments were carried out using a Stable Micro Systems TA.HDplus texture analyser (Stable Micro Systems Ltd.,
Godalming, Surrey, UK). A 5 kg load cell was used which had a force resolution of 0.1 g (≈ 0.001 N) and a displacement resolution of 1 µm. Four variables were recorded: force, distance, strain and time. Individual agglomerates were compressed on a flat, 120 mm square glass plate that was 4 mm thick. Since the agglomerates were not fixed in place, they were always in a stable orientation before compression. A 75 mm compressive platen was used; the diameter of this platen was large enough to ensure that any fragments which broke off the agglomerates remained underneath the platen, where they may have been compressed again subsequently. The configuration of the texture analyser is shown in Figure 4. The load cell and range of the texture analyser were calibrated daily before use, and small shims were inserted underneath the bottom surface of the texture analyser to ensure that the two compressing surfaces were exactly parallel. The settings chosen for the texture analyser are shown in Table 2. Note that the texture analyser begins to record data at the trigger force and continues until the target force is attained.

2.6) Uniaxial Compression Responses

Three responses were taken from the raw data obtained from the texture analyser:

1. The normal force at the point of failure of the agglomerate (N)
2. The strain at the point of failure of the agglomerate (%)

3. The agglomerate stiffness (N m\(^{-1}\))

Identifying one unique point of failure for strain-controlled crushing can be problematic as each plot will feature a number of local maxima, so criteria to identify a failure point must be selected. These issues are illustrated by Figure 5, which shows three representative plots of force versus displacement for compression of infant formula agglomerates. For consistency, an algorithm to identify the points at which agglomerates fail was developed and implemented in MATLAB (v.7.0.1, The MathWorks, Natick, MA, USA). The point of failure is defined to be the first point, ordered by increasing strain, which satisfies the following three criteria:

1. It is a local maximum on a plot of force versus deformation, and has the highest force value within a small (< 2%) strain range surrounding this point.

2. The maximum force attained within a 10% strain range following the point of failure must be less than the force at failure (otherwise, this is indicative of asperity failure, not agglomerate failure).

3. The difference between the force at failure and the force at the subsequent local minimum must be greater than 0.01 N, and when
normalised by the force at failure, the difference must be greater than 25%.

These specific numeric values were chosen as they identified the point of failure reliably when the algorithm was tested using a subset of the data recorded. The circles and dashed lines on Figure 5 identify the points of failure and stiffnesses obtained using this approach for three representative tests. Each agglomerate stiffness was calculated as the slope of the linear region of the force-displacement plot immediately prior to failure. The total numbers of agglomerates considered were 486, 457, 464 and 447 for infant formulae A–D, respectively.

2.7) Drop Tests using High-Speed Camera

Thirty agglomerates of each infant formula were dropped individually onto a flat, horizontal, stainless steel plate. Each agglomerate was gently pushed off a smooth stainless steel platform which was at a fixed elevation of 450 mm above the target plate. This ensured that the impact velocities were consistent at approximately 1.9 m/s: an important consideration since the coefficient of restitution generally decreases with increasing impact velocity (e.g., Schwager, 2007). None of the agglomerates were seen to fail when subjected to this dynamic loading.
The impacts were recorded at 1000 fps and at a resolution of 1280x600 using an AOS X-Motion high-speed camera (AOS Technologies AG, Baden Daettwil, Switzerland). Each resulting .mpg file was analysed subsequently using ProAnalyst (v.1.5.3.0, Professional ed., Xcitex, Inc., Cambridge, MA, USA). This software allowed the position of each agglomerate to be tracked over time, and thus provided the raw data required to calculate the coefficient of restitution, i.e., the magnitude of the normal velocity after impact divided by the normal velocity before impact. The mean normal velocities were calculated for six consecutive frames directly before and after impact. These velocities, averaged over a 0.005 s period, were used to calculate the coefficient of restitution. This was more accurate than using only two frames. Figure 6 shows four non-consecutive cropped sample frames for one drop test with a mm scale in the background.

3) Results and Discussion

3.1) Pneumatic Conveying Experiments

The variations in bulk density, mean particle diameter (D[4,3]), wettability and percentage free fat with conveying velocity for the four formulae tested are given in Figure 7. For all four infant formulae, there was a steady increase in bulk density with conveying velocity, as expected. It is
interesting to compare the magnitudes of the bulk density increases. The densities of formulae B and D showed the least variation: the percentage differences between the bulk densities before conveying and after conveying at 20 m/s were 8.9% and 7.7%, respectively. The corresponding differences for formulae A and C were much larger at 18.4% and 25.5%. Even within the dense phase regime, differences between the densities before conveying and after conveying at 4 m/s were more pronounced for A and C: the percentage differences were 2.8%, 0.9%, 1.6% and 0.4% respectively for formulae A–D. This means that attrition is potentially of greatest concern for formulae A and C. The bulk densities of the formulae before conveying were related to the percentage of protein in their compositions. Infant formula A contained the least protein (10.7%) and had the lowest bulk density before conveying of 452.6 kg/m$^3$; C contained the second-lowest amount of protein and had the second-lowest bulk density of 465.1 kg/m$^3$; etc.

The bulk density increased due to attrition of the powder, which is also shown by the decreasing trends in D[4,3] on Figure 7. This is as expected from results in literature (e.g., Kalman and Goder, 1998). The differences between mean diameters of the formulae before conveying and following passage through the rig at 4 m/s were low: the difference of 2.1% for formula A was the largest. The percentage differences following transport at
20 m/s were 17.8%, 13.5%, 22.0% and 14.1% respectively for formulae A–D. The marked differences between D[4,3] values before conveying were attributable to factors such as spray dryer and fluidised bed configurations.

These D[4,3] results may be compared to the relative breakage results shown in Figure 8. B_r generally increased with air velocity, except for infant formula A for which B_r remained constant at around 0.06. For any particular velocity, the trends in B_r were related to the percentage differences in D[4,3], e.g., at 20 m/s, formula C had the largest percentage difference in D[4,3] compared to the value before conveying of 22%, and this formula correspondingly had the highest B_r of 0.096. Similarly, the smallest percentage difference of 13.5% was for formula B which had the lowest B_r of 0.050.

One of the primary disadvantages of infant formula attrition is deterioration of the product's rehydration characteristics. Figure 7 shows the increase in wettability time that is commensurate with increased attrition. Infant formula B had a very poor wettability. The minimum wettability recorded for this formula (22.6 s, measured before conveying) was higher than any of the measurements for the other three formulae, even where attrition was high. Note that wettability is related to particle size (Ortega-Rivas, 2009), and B also had the smallest D[4,3] values of the four formulae tested.
Formula A had a very high free fat content, which increased from 0.75% before conveying to a maximum of 1.46% after conveying at 20 m/s. This increasing trend in percentage free fat was not observed for the other formulae; however, this may be due to their relatively low values of free fat compared to formula A. The progressive reduction in percentage free fat as the protein to fat ratio increased reflected a similar trend in the overall fat content of these powders: powder A contained the most fat (28.8%) while powder D contained the least (15.2%). It is hypothesised that the regions which contained high concentrations of fat coincided with the points of structural weakness for agglomerates of infant formula A. This explanation accounts for the observed free fat results as breakage of the agglomerates during conveying exposed surfaces containing high levels of fat.

3.2) Uniaxial Compression

The distributions in force at failure, strain at failure and agglomerate stiffness showed a pronounced positive skew. Both Weibull and lognormal distributions were fitted to these data sets for each infant formula using MATLAB. The lognormal distribution gave a better fit of the data for each response. The associated probability density functions are compared on Figures 9–11, while Table 3 shows means and standard deviations of the raw data and the parameters of the lognormal distributions.
By comparing the means, a number of general trends became apparent. In general, the force at failure and agglomerate stiffness increased with the protein to fat ratio while the strain at failure decreased. For all responses and formulae, the standard deviations were very large, exceeding 50% of the corresponding means, which illustrates the inherent variability between individual spray-dried agglomerates. There was a clear correspondence between the force at failure and the percentage of protein in the formulae: the lowest of 10.7% for formula A resulted in the lowest mean force at failure of 0.0530 N, whereas the maximum mean force at failure of 0.0905 N was for formula D which contained 16.7% protein. Protein content was also inversely related to strain at failure. As the percentage of protein increased, the strain at failure decreased. Since the percentage of protein was related to the bulk densities of the formulae before conveying, formulae with low bulk densities before conveying had low mean forces at failure and high mean strains at failure. There is an interesting link between mean forces at failure of the agglomerates and changes in bulk density due to conveying. It is intuitively sensible that formulae containing strong agglomerates with high forces at failure would break less under mechanical loading, and thus exhibit less change in bulk density, than a formula containing weaker agglomerates. Infant formulae B and D had the largest mean forces at failure, and also showed the least change in bulk density on
Figure 7. The agglomerates of formulae A and C were comparatively weak, and their bulk densities varied the most with conveying velocity.

The mean strain at failure was also related to the differences between bulk densities before conveying and after conveying at 4 m/s: the smallest bulk density difference (0.4%) and mean strain at failure (13.4%) were both for infant formula D, while the highest values (2.8% and 15.1% respectively) were recorded for formula A. There was a relationship between carbohydrate content and agglomerate stiffness, although this was not as clear as the others. It is true that increasing the percentage of carbohydrate increased the agglomerate stiffness. However, formulae C and D had identical carbohydrate contents but markedly different stiffnesses, which indicated that the observed relationship may be caused by other factors. It must be noted that all of these observations were based on only four data points and more formulae would need to be tested to confirm these general trends.

It was instructive to determine whether or not significant correlations existed between any pairs of responses from a data set. Since the distributions were not normal (verified using the Shapiro-Wilk W test), the degree of relationship between the responses was evaluated in STATISTICA (v.7.1, StatSoft, Inc., Tulsa, OK, USA) using Spearman R
rank correlation coefficients. All correlations were found to be positive as shown in Table 4. There was a strong positive correlation between force at failure and agglomerate stiffness, with infant formula A having the highest Spearman R of 0.8511. Spearman R coefficients for the other two combinations of responses were much lower (maximum of 0.2444).

3.3) Drop Tests

Table 5 shows means and standard deviations of the coefficients of restitution which were obtained by video analyses of agglomerate drop tests. Formula A had the lowest mean coefficient of restitution of 0.2460 and formula B had the highest value of 0.3258. There was an interesting relationship between the coefficient of restitution and the relative breakage results for low velocity conveying at 4 m/s. Where coefficients of restitution were low, this implies that extensive breakage of either bonds or primary particles occurred which absorbed energy. Since these agglomerates were particularly susceptible to damage, it might be expected that the infant formula would be more friable than other formulae under similar loading conditions. Infant formula A, which had the lowest coefficient of restitution, had the highest \( B_r \) for conveying at 4 m/s (0.058), formula D had the second-lowest coefficient of restitution and the second-highest \( B_r \) of 0.026, while the \( B_r \) results for formulae B and C were transposed, but were very close as seen on Figure 8.
None of the agglomerates were seen to fail during the drop tests. There are two reasons for this. Firstly, the impact velocity was around 1.9 m/s, and it was shown that attrition was low using a considerably higher conveying velocity of 4 m/s. Secondly, the agglomerates for the uniaxial compression experiments and the drop tests were isolated using a sieve shaker which applied significant mechanical loading to the powder. This process is likely to have broken any friable agglomerates, so those remaining with a size between 710 μm and 850 μm were likely to be particularly resistant to breakage.

4) Conclusions

In this paper, the relationships between the compositions of infant formulae, the changes in key quality characteristics when transported by pneumatic conveying and the mechanical properties obtained by uniaxial compression and drop testing of individual agglomerates were investigated. As conveying velocity increased, so too did the bulk density, although the increases in bulk densities for some formulae (A and C) were considerably greater than for others (B and D). As the percentage of protein increased, the bulk densities before conveying of the four formulae increased accordingly. The wettability of formula B was poor, which may be caused by its
generally smaller particle size. The free fat decreased progressively as the protein to fat ratio increased, which is commensurate with the decreasing fat contents of these powders.

The force at failure and agglomerate stiffness generally increased with increasing protein to fat ratio while the strain at failure decreased. Strong positive correlations existed between force at failure and agglomerate stiffness. Both force and strain at failure were related to the percentage of protein: increasing the protein content caused an increase in the mean force at failure and a decrease in the mean strain at failure. Agglomerates of infant formulae B and D had the largest mean forces at failure, which explains why these formulae showed the least variation in bulk density when pneumatically conveyed. The strain at failure was also related to the differences between bulk densities before conveying and after conveying at 4 m/s. When drop tests were conducted at around 1.9 m/s, infant formula A had the lowest mean coefficient of restitution and B had the highest (0.3258). The coefficients of restitution decreased as the relative breakage results increased, which is likely to be due to the increased number of bonds or primary particles broken during loading.
5) Acknowledgements

Kevin Hanley would like to acknowledge the receipt of funding from the Irish Research Council for Science, Engineering and Technology (IRCSET).

6) References


7) Figures

Figure 1: An isometric diagram of the pneumatic conveying rig

Figure 2: Alternative presentations of the same particle size distributions for infant formula B before conveying and following pneumatic transport at 20 m/s
Figure 3: Particle size distributions with shaded areas indicating: a) the breakage potential, $B_p$, and b) the total breakage, $B_t$, according to the definitions of Hardin (1985).

Figure 4: Configuration of the texture analyser used for agglomerate compression.
Figure 5: Plots of force (N) versus displacement ($10^{-4}$ m) for experimental compression of three agglomerates of infant formula B, indicating points of failure of the agglomerates using circles and agglomerate stiffnesses using dashed lines.

Figure 6: Four cropped frames (numbers 50, 69, 72 and 83) extracted from the video of a drop test of one agglomerate (infant formula C), where the agglomerate is circled and its direction of motion is indicated by arrows.
Figure 7: Bar charts comparing the bulk density (kg/m$^3$), D[4,3] (μm), wettability (s) and percentage free fat of infant formulae A–D before conveying, and following pneumatic transport at 4, 10 and 20 m/s, where the error bars indicate one standard deviation.
Figure 8: Plot of relative breakage against air velocity (m/s), where the error bars indicate one standard deviation.

Figure 9: Probability density functions of lognormal distributions fitted to force at failure data (N) for the four infant formulae tested.
Figure 10: Probability density functions of lognormal distributions fitted to strain at failure data (%) for the four infant formulae tested

Figure 11: Probability density functions of lognormal distributions fitted to agglomerate stiffness data (N m$^{-1}$) for the four infant formulae tested
8) Tables

Table 1: Compositions of the infant formulae used in terms of their major components, in order of increasing protein to fat ratio

<table>
<thead>
<tr>
<th>Infant Formula</th>
<th>Carbohydrate</th>
<th>Fat</th>
<th>Protein</th>
<th>Ash</th>
<th>Moisture</th>
<th>Protein:Fat</th>
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</thead>
<tbody>
<tr>
<td>A</td>
<td>56.5</td>
<td>28.8</td>
<td>10.7</td>
<td>2.0</td>
<td>2.0</td>
<td>0.372</td>
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<tr>
<td>B</td>
<td>56.1</td>
<td>22.1</td>
<td>15.4</td>
<td>4.4</td>
<td>2.0</td>
<td>0.697</td>
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<tr>
<td>C</td>
<td>58.9</td>
<td>17.1</td>
<td>14.8</td>
<td>5.2</td>
<td>4.0</td>
<td>0.865</td>
</tr>
<tr>
<td>D</td>
<td>58.9</td>
<td>15.2</td>
<td>16.7</td>
<td>5.2</td>
<td>4.0</td>
<td>1.099</td>
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</table>

Table 2: Settings chosen for the configurable parameters of the texture analyser

<table>
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<th>Parameter</th>
<th>Setting</th>
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</thead>
<tbody>
<tr>
<td>Trigger force</td>
<td>0.2 g (= 0.002 N)</td>
</tr>
<tr>
<td>Target force</td>
<td>30 g (= 0.29 N)</td>
</tr>
<tr>
<td>Pre-test speed</td>
<td>0.01 mm/s</td>
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<tr>
<td>Test speed</td>
<td>0.01 mm/s</td>
</tr>
<tr>
<td>Post-test speed</td>
<td>5 mm/s</td>
</tr>
<tr>
<td>Data acquisition rate</td>
<td>500 points/s</td>
</tr>
</tbody>
</table>
Table 3: Means and standard deviations of the results obtained for force at failure (N), strain at failure (%), and agglomerate stiffness (N m⁻¹), and the parameters of the associated fitted lognormal distributions. 

μ and σ are the means and standard deviations of the corresponding normal distributions

<table>
<thead>
<tr>
<th></th>
<th>Force (N)</th>
<th>Strain (%)</th>
<th>Stiffness (N m⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Formula A</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean {Std. Dev.}</td>
<td>0.05301 {0.04156}</td>
<td>15.1448 {11.4917}</td>
<td>4695.79 {2964.44}</td>
</tr>
<tr>
<td>μ</td>
<td>σ</td>
<td>-3.20912</td>
<td>0.74351</td>
</tr>
<tr>
<td><strong>Formula B</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean {Std. Dev.}</td>
<td>0.07140 {0.05275}</td>
<td>14.4088 {10.4331}</td>
<td>4648.45 {2921.23}</td>
</tr>
<tr>
<td>μ</td>
<td>σ</td>
<td>-2.87162</td>
<td>0.68387</td>
</tr>
<tr>
<td><strong>Formula C</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean {Std. Dev.}</td>
<td>0.05603 {0.04372}</td>
<td>14.9298 {10.8541}</td>
<td>5232.35 {3169.15}</td>
</tr>
<tr>
<td>μ</td>
<td>σ</td>
<td>-3.11709</td>
<td>0.67391</td>
</tr>
<tr>
<td><strong>Formula D</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mean {Std. Dev.}</td>
<td>0.09047 {0.06005}</td>
<td>13.3854 {10.2184}</td>
<td>7358.42 {4201.11}</td>
</tr>
<tr>
<td>μ</td>
<td>σ</td>
<td>-2.60941</td>
<td>0.65754</td>
</tr>
</tbody>
</table>

Table 4: Spearman R rank correlations for all combinations of the force at failure (N), strain at failure (%), and agglomerate stiffness (N m⁻¹) responses for all four infant formulae tested

<table>
<thead>
<tr>
<th></th>
<th>Spearman R {p-value}</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Force ↔ Strain</td>
</tr>
<tr>
<td><strong>Formula A</strong></td>
<td>0.1418 {0.0017}</td>
</tr>
<tr>
<td><strong>Formula B</strong></td>
<td>0.2363 {0.0000}</td>
</tr>
<tr>
<td><strong>Formula C</strong></td>
<td>0.2067 {0.0000}</td>
</tr>
<tr>
<td><strong>Formula D</strong></td>
<td>0.2368 {0.0000}</td>
</tr>
</tbody>
</table>
### Table 5: Means and standard deviations of coefficients of restitution

<table>
<thead>
<tr>
<th>Infant Formula</th>
<th>Mean</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula A</td>
<td>0.2460</td>
<td>0.0950</td>
</tr>
<tr>
<td>Formula B</td>
<td>0.3258</td>
<td>0.0979</td>
</tr>
<tr>
<td>Formula C</td>
<td>0.3009</td>
<td>0.1030</td>
</tr>
<tr>
<td>Formula D</td>
<td>0.2768</td>
<td>0.1089</td>
</tr>
</tbody>
</table>