

Title	Effect of pneumatic conveying parameters on physical quality characteristics of infant formula
Authors	Hanley, Kevin J.;Byrne, Edmond P.;Cronin, Kevin;Oliveira,, Jorge C.;O'Mahony, James A;Fenelon, Mrk
Publication date	2011-05-09
Original Citation	Hanley, K.J., Byrne, E.P., Cronin, K., Oliveira, J.C., O'Mahony, J.A. and Fenelon, M.A. (2011) 'Effect of pneumatic conveying parameters on physical quality characteristics of infant formula', Journal of Food Engineering, 106(3), pp. 236–244. Available at: https://doi.org/10.1016/j.jfoodeng.2011.04.029
Type of publication	Article (peer-reviewed)
Link to publisher's version	https://doi.org/10.1016/j.jfoodeng.2011.04.029 - 10.1016/j.jfoodeng.2011.04.029
Rights	© 2011, Elsevier Ltd. All rights reserved. This manuscript version is made available under the CC BY-NC-ND 4.0 license. - https://creativecommons.org/licenses/by-nc-nd/4.0/
Download date	2025-03-25 12:45:58
Item downloaded from	https://hdl.handle.net/10468/15340

Title

Effect of pneumatic conveying parameters on physical quality characteristics of infant formula

{Short title: Effect of pneumatic conveying parameters on infant formula properties}

Authors

Kevin J. Hanley^{a*}, Edmond P. Byrne^a, Kevin Cronin^a, Jorge C. Oliveira^a, James A. O'Mahony^b, Mark A. Fenelon^c

Affiliations

a) Dept. of Process and Chemical Engineering, University College Cork, Cork, Ireland

b) School of Food and Nutritional Sciences, University College Cork, Cork, Ireland

c) Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland

* Corresponding author: k.hanley@umail.ucc.ie

Abstract

The geometry and operating conditions of a pneumatic conveying rig for infant formula were varied according to an L_{18} orthogonal array, with the goal of minimising variations in four product quality characteristics: bulk density, volume mean diameter, particle density and wettability. A modular pneumatic conveying rig was fabricated from 316L stainless steel components. The factors that were varied in these experiments included mode of conveying, air velocity, number of rig passes, bend radii and vertical rig section length. A factorial analysis of variance showed that the mode of conveying, air velocity and number of passes had a statistically-significant effect on bulk density. The optimum settings to minimise variability were dense phase conveying with a 50 mm plug length, 960 mm vertical section, 3 m/s air velocity, 2 passes and 50 mm bend radii, assuming a linear model. The bulk density change at these optimum settings was negligible at 0.9%.

Keywords

attrition, bulk density, experimental design, Taguchi robust engineering design, wettability

Nomenclature

a_0	offset
a_1	linear model parameter for mode of conveying
a_2	linear model parameter for length of vertical rig section
a_{22}	quadratic model parameter for length of vertical rig section
a_{31}	linear model parameter for air velocity in dense phase
a_{32}	linear model parameter for air velocity in dilute phase
a_{331}	quadratic model parameter for air velocity in dense phase
a_{332}	quadratic model parameter for air velocity in dilute phase
a_4	linear model parameter for number of passes through rig
a_5	linear model parameter for radii of 90° bends
a_{55}	quadratic model parameter for radii of 90° bends
a_{61}	linear model parameter for plug length
a_{62}	linear model parameter for solids feed rate
a_{662}	quadratic model parameter for solids feed rate
F_1	coded mode of conveying factor (-1, 1)

F ₂	coded length of vertical rig section factor (-1, 0, 1)
F ₃	coded air velocity factor (-1, 0, 1)
F ₄	coded number of passes through rig factor (-1, 0, 1)
F ₅	coded radii of 90° bends factor (-1, 0, 1)
F ₆	coded plug length / solids feed rate pseudo-factors (-1, 0, 1)
R	model response

1) Introduction

Infant formula is a substitute for human milk designed to satisfy the normal nutritional requirements of infants until the introduction of appropriate complementary feeding (Codex Alimentarius, 2007). Globally, most infant formula is sold in powdered form, and typically these powders are agglomerated for improved reconstitution.

Powdered infant formula is usually dispensed on a volume basis using a scoop before it is reconstituted with water. It is essential that the caregiver measures powders accurately to achieve the correct liquid formula composition which fully meets the nutritional needs of the infant. Legislation specifies the essential composition of infant formula when reconstituted in accordance with manufacturer's preparation instructions (e.g., European Commission, 2006; U.S. Food and Drug Administration,

2010). Therefore, powder bulk density is a key quality parameter for infant formula manufacturers, as this affects the quantity of powder that fills the scoop. It is necessary for manufacturers to factor in an allowance in bulk density during spray drying to compensate for powder breakage, which inevitably occurs during in-plant handling, transport and packaging. If this breakage estimation is inaccurate, product bulk densities may be out of compliance with in-plant specifications. Other effects of infant formula attrition must also be taken into account. For example, attrition tends to deteriorate the product's instant properties, such as wettability (Hogekamp and Schubert, 2003).

Dense-phase pneumatic conveying is often used in plants for transporting infant formulae from the drying operation to the filling line in order to minimise breakage of the agglomerates. Previous studies have demonstrated conclusively that increasing the air or particle velocity in a pneumatic conveying system increases product attrition (Kalman and Goder, 1998; Taylor, 1998; Zhang and Ghadiri, 2002). Most attrition in pneumatic conveying systems occurs around bends (Kalman, 1999), which makes the selection of bend type critical (Aarseth, 2004; Chapelle et al., 2004; Salman et al., 2002; Wypych and Arnold, 1993). Other influential factors include the number of passes (Kalman, 2000; Konami et al., 2002), the mass flow

ratio (Han et al., 2003; Kalman, 2000) and structural characteristics of the conveyed product (Samimi et al., 2003).

The objective of this paper was to analyse the influence of seven pneumatic conveying parameters that may affect key quality characteristics of infant formulae, such as bulk density, particle size and wettability. A lab-scale modular pneumatic conveying rig was constructed, which allowed the selected parameters to be varied according to a Taguchi experimental design, and four responses were measured. The aim was to identify which parameters have the most significant effect on these responses, as it is more important to control these parameters accurately than those which are less influential. In practice, while the settings chosen by the system designer for some of the tested parameters (e.g., the vertical conveying length) would be constrained by physical restrictions, it is still important to understand how these parameters influence the key quality characteristics of infant formula. Another objective was to determine the optimum pneumatic conveying parameters to minimise variations in the measured product quality characteristics. This research also investigates whether the significant parameters are the same for different responses, and if any of the responses are relatively unaffected by conveying.

2) Materials and Methods

2.1) Overview of the Taguchi Method

The Taguchi method was developed as an integrated process improvement technique by Genichi Taguchi during the 1950s, and has since become popular due to its experimental efficiency when compared to alternatives such as response surface methodology. However, one disadvantage of this approach is the creation of intricate confoundings between effects of factors and of interactions (Montgomery, 2009). Taguchi recommended a method with three steps (Ross, 1988):

(i) System design; (ii) Parameter design; (iii) Tolerance design

System design involves determining which process parameters have the greatest influence on the response and selecting suitable levels for those factors. Parameter design is then applied to find the optimum levels for each factor from those identified by the system design. This requires the selection of a suitable orthogonal array which depends on the number of factors and levels to be tested. Each control factor is assigned to a column of the array, and the number of trials for each set of experiments is given by the number of rows. Finally, tolerance design is used to tighten tolerances on statistically-significant product or process parameters to reduce variation (Ross, 1988).

When applying the Taguchi method, it is usual to interpret the data using Analysis of Variance (ANOVA). This statistical tool quantifies how much of the variability of the response can be attributed to each factor or interaction. If data from multiple replicates is available, Taguchi recommends combining repetitions of each trial into signal-to-noise (S/N) ratios to find the optimum combination of parameters. The advantage of analysing with S/N ratios is that the mean response may be optimised while simultaneously taking the reduction of process variability into account. Three different equations are used for calculating the S/N ratio depending on the objective of the optimisation. For this analysis, the lower-is-best (or smaller-the-better) quality characteristic is used, which is calculated as:

$$S/N_L = -10 \log_{10} \left(\frac{1}{r} \sum_{i=1}^r y_i^2 \right)$$

where r is the number of repetitions of each trial and y_i is the quality characteristic value for trial i (Ross, 1988). After transforming the experimental results into S/N ratios, the objective is to maximise the response. Once the optimum response has been found, a confirmation experiment should be carried out unless the factor levels which maximise

the S/N ratio correspond exactly to one of the rows of the orthogonal array; this is an unlikely event due to the highly fractioned nature of these designs.

2.2) Raw Materials and Pneumatic Conveying Rig

A commercial infant formula was used for these trials containing approximately 57% carbohydrate, 28% fat, 11% protein, 2% ash and 2% moisture. The pneumatic conveying rig was assembled from hygienically-designed modular components using tri-clover clamps. The components were manufactured from 316L stainless steel with a 2B internal surface finish, except for the powder collection vessel, which was made of polypropylene. The basic rig configuration was invariant, with two horizontal sections linked to a vertical section by two 90° bends. This configuration was selected to provide a close analogue of a real industrial system, in which inclined pipe sections are seldom used and a vertical conveying section is required to transport formulae from the spray dryer outlet to the silo inlet. Three different bend radii (50, 200, 300 mm) and three different vertical section lengths (340, 650, 960 mm) were available. The rig incorporated two in-line sight-glasses to allow visual observation of the powder transport operation. Dry compressed air entered the rig at the point indicated on Figure 1 with its pressure regulated to provide different air velocities. The air velocity could be measured by inserting a 3 mm diameter Pitot-static tube into a small resealable hole in one horizontal

length. All air velocities in this paper are stated in terms of the maximum superficial air velocities in the pipeline containing no powder, which could be measured more accurately than average velocity. After measuring the velocity, the Pitot-static tube was removed and the measurement hole sealed before commencing powder conveying to avoid disruption of the flow.

For dilute phase operation, the powder was poured into the air stream using a funnel. To simulate dense phase (plug flow) operation, the funnel was removed and the opening sealed off with a blank. The first sight glass was filled with infant formula to create a single plug in the line before reassembling the rig and switching on the air supply with a ball valve.

The pipe diameter was constant at 25 mm, except for a 50 mm diameter terminal diverging section. This section of larger diameter minimised breakage at the capture point by reducing the velocity of the air stream immediately before the capture vessel. The typical pipe diameter in an infant formula manufacturing plant is approximately 100 mm. Although the ratio of pipe diameter to particle diameter remained very large for the lab-scale rig, there are often issues when scaling up results obtained using small-scale pneumatic conveying systems to industrial-scale systems. This possible limitation must be remembered for the results given in this paper. The end vessel incorporated filters for separation of powder from the air,

and air stream entry into the vessel was offset to create a swirling fluid motion and further reduce powder breakage.

2.3) Experimental Design

Seven factors were varied, with all others held constant. Noise factors were not included in these experiments so only an inner array was used. All factors were controlled at three levels only, except mode of conveying (dilute or dense phase), which must be a 2-level factor. The control factors and levels used for this experiment are shown in Table 1. Note that the number of passes for this experimental system was analogous to the length of an industrial conveying system, i.e., increasing the number of passes increased the effective length.

An L_{18} orthogonal array was chosen; this mixed-level array allows for one 2-level factor and up to seven 3-level factors to be tested. The blocking of the design must be noted. For dense phase, the factor 'plug length' exists but 'solids feed rate' does not, and vice-versa for the dilute phase. Furthermore, the mode of conveying determines the levels used for air velocity: if dense phase is specified, air velocity must be less than the saltation velocity for horizontal flow (Marcus et al., 1990), which was found during preliminary experiments. This situation may be interpreted using the trans-factor technique (a specific type of pseudo-factor design) to assign the orthogonal

array (Taguchi, 1987). Mode of conveying is a branching factor, while plug length and solids feed rate are pseudo-factors or nested factors. A narrow range of velocities was used for dense phase conveying (3–5 m/s) as the velocity had to be less than the saltation velocity, yet also sufficient to ensure reliable conveying.

This array provides seventeen degrees of freedom for studying effects. The vertical rig section length was assigned to column two of the array to allow its interaction with the 2-level factor to be studied; this is the only interaction which can be studied (Maghsoodloo et al., 2004) as others are partially confounded with the remaining 3-level columns (Taguchi, 1992). All other interactions must therefore be assumed to be negligible, or the intricate confounding would not permit a unique interpretation of the data. Sufficient degrees of freedom are available to allow this interaction to be tested for statistical significance without pooling. After assigning the remaining factors to columns, the array in Table 2 was obtained.

2.4) Product Quality Characteristics and Experimental Procedure

Four product quality characteristics were measured:

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

3) Particle density

4) Wettability

For infant formula manufacture, bulk density is one of the most critical quality characteristics as this determines the quantity of powder which fills the scoop before reconstitution. This was measured using a Stampfvolumeter STAV 2003 (J. Engelsmann AG, Ludwigshafen, Germany). One hundred grams of powder was weighed into the graduated cylinder for each measurement and 1250 taps were used.

The particle size distribution of the infant formula agglomerates was measured by laser diffraction using a Malvern Mastersizer S with dry powder feeder (Malvern Instruments Limited, Malvern, Worcestershire, UK). The particle size distribution was measured twice for each trial to increase accuracy, and the mean result was used for analysis. The volume mean diameter / De Brouckere mean diameter ($D[4,3]$) was used as the single measure to compare particle size results.

Agglomerates of infant formula contain internal cavities; however, it was not known whether or not these voids were impermeable to air for the formula tested. Particle density was found by nitrogen pycnometry using a Micromeritics Multivolume Pycnometer 1305 (Micromeritics Instrument

Corporation, Norcross, GA, USA). By assessing the changes in particle density upon conveying, inferences could be made about the permeability of the subsurface cavities to air. The particle density was determined three times for each trial using the same sample and the mean result was used.

Wettability was measured using the GEA Niro Analytical Method (GEA Niro, 2009) based on the International Dairy Federation standard for determining the dispersibility and wettability of instant dried milk (International Dairy Federation, 1979). Ten grams of infant formula were placed inside a steel ring on top of a flat stainless steel plate. The plate was placed on top of a 600 ml beaker containing 250 ml of water at 25°C. The infant formula was scattered on the water surface by slowly withdrawing the plate. The wettability was recorded as the time required for all particles to become wetted, by visual inspection, from when the plate began being withdrawn. Note that low wettability times are desirable, indicating good wettability behaviour.

Three complete sets of replicates were carried out, giving 54 trials in total. Complete randomisation was used to minimise bias in the results. Wettability was measured for only one replicate, while bulk density, particle density and D[4,3] were measured for all three replicates. These four quality characteristics were also measured prior to conveying: measurements were

taken for four samples of infant formula per replicate, e.g., bulk density was measured for twelve samples before conveying. The mean bulk density, $D[4,3]$, particle density and wettability of the infant formula before conveying were 462 kg/m^3 , $312 \text{ }\mu\text{m}$, 1107 kg/m^3 and 18 s, respectively.

2.5) Analytical Procedure

All data analysis was conducted using the percentage change in each measured powder quality characteristic before and after passage through the rig. The data were analysed using STATISTICA v.7.1 (StatSoft, Inc., Tulsa, OK, USA). The complete analysis may be divided into two parts:

- 1) The pseudo-factors were treated as categorical and an ANOVA was done using S/N ratios for each quality characteristic.
- 2) A polynomial model was fitted to the data, taking into account the nesting of factors.

2.5.1) Categorical ANOVA

As recommended by Taguchi, the trial replicates were combined into S/N ratios for each of the four responses. This was done for wettability even though data were available for only a single replicate. S/N ratios were calculated using the lower-is-best definition, since the smaller the change in

a quality characteristic during infant formula handling, the easier it is to control final product properties in the manufacturing plant. Ideally, the properties of the infant formula immediately after drying would be unchanged when the consumer reconstitutes the product. In that case, the spray drying conditions could be controlled to give an optimal product rather than needing to add estimated allowances for subsequent powder breakage.

An ANOVA was performed on the S/N ratios for each quality characteristic to identify statistically-significant factors. All factors were analysed without the inclusion of nesting in the array to avoid intricate confoundings which would prevent certain factor effects from being uniquely distinguished. For instance, no distinction was made between air velocities of 5 m/s (the highest value for dense phase) and 30 m/s (the highest for dilute phase), as both have the meaning “velocity as high as possible” in terms of the categorical classification. The same is true for equivalent levels of plug length and solids feed rate (both as high as possible, or as low as reasonable, or the mid-point).

Effects were calculated and contributions found by subtracting the global mean of the S/N ratios for each quality characteristic from the marginal means. These effects were tested for statistical significance. Contributions

plots were drawn for each factor which show the average effect of selecting any factor level on the responses. A second ANOVA was performed for four selected factors, taking nesting into account: air velocity, mode of conveying, plug length and solids feed rate. By omitting all other factors, confounding was avoided. The main purpose of the second ANOVA was to obtain contributions plots for air velocity and the pseudo-factors which show the required dependence on mode of conveying. It is noted that this procedure does not lead to any loss of information, because the impact of ignoring the nesting in the first ANOVA was that the importance of the nested factors may have been overestimated. Thus, the main outcome of the first ANOVA was to identify those factors which were not statistically-significant. The second ANOVA then gave a more accurate and real view of the results by considering the nesting.

Optimum factor levels were determined using the maximum S/N ratio contributions, and estimates of each product quality characteristic at these optimum levels were calculated. A verification experiment was done using the optimum settings to check the results of the analysis.

2.5.2) Polynomial Models

It is likely that the influence of certain factors on a characteristic depends on the mode of conveying, i.e., the air velocity might be expected to have far

greater importance for dilute phase conveying than for dense phase. However, the ANOVAs did not consider this when evaluating the statistical significance of factors, including the air velocity and pseudo-factors. A complementary analysis was performed in which a polynomial model was fitted to the data. This allows the statistical significance of most factors to be evaluated while taking nesting into account, i.e., distinguishing between effects for dilute and dense phase conveying. This polynomial model was fitted to the raw data for each quality characteristic: bulk density, D[4,3], particle density and wettability. This model took the form of Eq. 1, where F_n represents the levels of factor n when coded between -1 and 1 and R is the model response:

$$\begin{aligned}
 R = & a_0 + a_1F_1 + a_2F_2 + a_{22}F_2^2 + 0.5a_{31}F_3(1 - F_1) + 0.5a_{32}F_3(1 + F_1) \\
 & + 0.5a_{331}F_3^2(1 - F_1) + 0.5a_{332}F_3^2(1 + F_1) + a_4F_4 + a_5F_5 + a_{55}F_5^2 + 0.5a_{61}F_6(1 - F_1) \\
 & + 0.5a_{62}F_6(1 + F_1) + 0.5a_{662}F_6^2(1 + F_1)
 \end{aligned}
 \tag{Eq. 1}$$

By omitting quadratic effects of the number of passes and the plug length, a unique set of model parameters could be determined for each quality characteristic by least-squares regression. The linear and quadratic terms for F_3 and F_6 in Eq. 1 are multiplied by $(1 \pm F_1)$ to ensure that certain parameters are present in the models only if dense phase conveying is used,

i.e., if $F_1 = -1$, while those parameters that are multiplied by $(1 + F_1)$ appear only if dilute phase conveying is used.

In the model fitting, the residual sums of squares for bulk density, D[4,3] and particle density were partitioned into pure error and lack of model fit components. This was not possible for wettability due to the lack of replicated measurements. Plots of model predicted values against experimental data were drawn to highlight bias, if present. Standardised effects were calculated for these parameters and these were shown on Pareto charts using a 95% significance level.

3) Results and Discussion

3.1) Categorical ANOVA

Table 3 is the partial ANOVA table using S/N ratios as the response, and ignoring nesting in the array. In the combined ANOVA table, ν is degrees of freedom, S is the sum of squares and p is the p-value. Factors are statistically-significant at the 90%, 95% or 99% levels if their p-values are less than 0.1, 0.05 or 0.01, respectively. Mode of conveying (99%), air velocity (95%) and number of passes (90%) had a statistically-significant effect on bulk density. For D[4,3] and wettability, mode of conveying was

the only factor which was significant. Air velocity had the second-highest sum of squares for both of these responses, but was not significant at the 90% level in either case. None of the factors had a significant influence on particle density. Since dense phase conveying is invariably used for pneumatic transport of infant formula, the air velocity is the most critical factor which manufacturers must control to avoid attrition issues.

Since S/N ratios are not always straightforward to interpret, a simpler way of visualising these results was to partition the raw percentage data into six categories: one for each combination of mode of conveying and air velocity. The mean percentage changes in bulk density, $D[4,3]$ and wettability were calculated for each of these combinations, and are plotted on Figure 2. For example, using dilute phase conveying and an air velocity of 30 m/s caused the bulk density to increase by 25.3%, on average, whereas selecting the same conditions reduced $D[4,3]$ by an average of 21.8%.

Figure 3 shows the ANOVA plot for S/N ratios using contributions, which shifts all data to be centred on the x-axis. Note that air velocity and plug length / solids feed rate factors showed a strong dependence on the mode of conveying. The optimum level for each factor was selected as that which gave the maximum S/N ratio contribution. Figure 4 compares the percentages of the total sums of squares attributed to each factor, and

identifies the optimum level for each factor. For example, mode of conveying accounted for more than 90% of the total sum of squares for bulk density and the optimum level of this factor was 1, or dense phase by reference to Table 1. This sum of squares was calculated from Table 3 by dividing 1892.06 by 2063.01, and the level was selected from Figure 3.

Figure 4 was used to select the optimum settings. It is clear that dense phase conveying, the lowest air velocity of 3 m/s and the shortest plug length of 50 mm should be chosen. The number of passes was set at 2, since this was the optimum for all responses except particle density, where it was not statistically-significant. The fact that 2 passes was selected as the optimum rather than 1 pass implies that the additional breakage caused by multiple passes through the conveying rig was negligible. The ANOVA confirms this: the number of passes was not significant for any quality characteristic except bulk density, where it was barely significant at the 90% level. A small amount of experimental variability could have caused 2 passes to be identified as being preferable to 1 pass, whereas there was minimal difference between the results obtained using these levels in reality. A vertical section length of 960 mm was selected; this factor made the dominant contribution to the total sum of squares for particle density. Finally, a bend radius of 50 mm was chosen. This was the optimum setting for both $D[4,3]$ and wettability. However, neither the radii of the bends nor

the vertical conveying length were significant factors. The estimated percentage bulk density and D[4,3] changes at these optimum settings were negligible at 1.15% and -1.90%, based on S/N ratios. These were obtained by summing the contributions corresponding to the optimum settings (including the interaction) to the global mean of the S/N ratios for each quality characteristic.

Since the combination of settings identified as the optimum was not among the 18 experiments in the Taguchi design, a verification test was required. The bulk density and particle size distribution were measured before and after conveying a 600 g sample of infant formula through the rig using the optimal rig geometry and operating parameters. Bulk densities of this sample before and after were 457.1 kg/m³ and 461.2 kg/m³ respectively and the corresponding D[4,3] results were 310.36 µm and 306.91 µm. These were changes of 0.90% and -1.11%. These results were close to the predicted results and were essentially equivalent within the limits of experimental error.

3.2) Polynomial Models

The parameters obtained by fitting the polynomial model shown as Eq. 1 to the raw data for bulk density, D[4,3], particle density and wettability are shown in Table 4. Table 5 contains the R² and R²_{adjusted} values for the model

fits, along with the percentages of the total model sum of squares attributed to error, subdivided into pure error and lack of model fit components. The model fit was particularly good for bulk density, where less than 5% of the total sum of squares was due to error, and less than 1% of this total was due to lack of model fit. However, the model fit for particle density was extremely poor: more than 80% of the total sum of squares was due to error. This result, in conjunction with the lack of any statistically-significant factors in Table 3, confirmed that the particle density of the infant formula tested was not affected by pneumatic conveying. Since agglomerates of such formulae contain internal cavities, there must be sufficient surface fissures or pores to permit the ingress of nitrogen into most of these voids when measuring particle density by pycnometry. Therefore, when the agglomerates were damaged during conveying, the change in particle density was negligible.

In all cases, some of the sum of squares attributed to error was caused by the omission of two quadratic effects from the model; however, the proportion of the sum of squares caused by lack of model fit was always low, indicating that neither quadratic effect was very important. The contributions plot for the plug length factor confirmed this, since it did not exhibit a marked curvature for any response. However, the contributions plot for the number of passes was notably curved for bulk density with the

maximum S/N occurring at the intermediate level. This factor was also significant at a 90% level in the categorical analysis, yet the omission of the quadratic effect did not significantly affect the model fit: a result which demonstrated the dominance of the mode of conveying factor for the bulk density response. Plots of model predicted values against experimental data are shown in Figure 5. No bias was apparent for bulk density or wettability. The model for D[4,3] showed a slight tendency to under-predict the experimental results at high attrition levels. There was a more obvious bias in the model for particle density. This model under-predicted large changes in particle density and overestimated small density changes.

Pareto charts for the four quality characteristics are shown in Figure 6. Mode of conveying was significant for both bulk density and D[4,3], while the linear parameter for air velocity in dilute phase was significant for all quality characteristics except particle density. It is interesting to note that the equivalent parameter for dense phase conveying was not close to significance in any case. Therefore, if dense phase conveying is used for infant formula transport, air velocity is not likely to have a significant impact on measured quality characteristics. However, the choice of air velocity remains critical to ensure consistent, reliable flow through the pneumatic conveying system. It should be noted that there are considerations other than the effect on product quality characteristics which

influence the choice of settings of a factor. For example, the air velocity and solids feed rate or plug length affect the throughput of a conveying system, and if a certain mass flow rate is required, this imposes restrictions on the settings chosen for these parameters. Other influential parameters were the quadratic parameter for solids feed rate for bulk density and the linear parameter for length of the vertical rig section. The latter was statistically-significant only for D[4,3], but had the largest standardised effect for particle density and the second-largest for wettability.

4) Conclusions

A lab-scale modular pneumatic conveying rig with a diameter of 25 mm was used to investigate the effect of varying seven factors on four product quality characteristics: bulk density, volume mean diameter, particle density and wettability. The main conclusions of this work are summarised as follows:

- 1) Mode of conveying (99%), air velocity (95%) and number of passes (90%) all had a statistically-significant effect on bulk density. For D[4,3] and wettability, mode of conveying was the only significant factor, while none of the factors had a statistically-significant influence on particle density.

- 2) Optimum settings for this system were dense phase conveying with a 50 mm plug length, 960 mm vertical section, 3 m/s air velocity, 2 passes and 50 mm bend radii. Estimated bulk density and $D[4,3]$ changes at these optimum settings were 1.15% and -1.90%. A verification test obtained similar changes of 0.90% and -1.11%.
- 3) By fitting a polynomial model to the raw data, it was shown that air velocity is significant for only dilute phase conveying, and not for dense phase. These results imply that the resulting changes in infant formula quality characteristics caused by conveying will be negligible, irrespective of the settings chosen for the other conveying parameters, provided that the air velocity is selected to ensure dense phase conveying.

5) Acknowledgements

The authors would like to thank technical staff of the Department of Process and Chemical Engineering at UCC, and Kevin Hanley would like to acknowledge the receipt of funding from the Irish Research Council for Science, Engineering and Technology (IRCSET).

6) References

Aarseth, K.A. (2004). Attrition of feed pellets during pneumatic conveying: the influence of velocity and bend radius. *Biosystems Engineering*, 89(2), 197-213.

Chapelle, P., Abou-Chakra, H., Christakis, N., Bridle, I., Patel, M.K., Baxter, J., Tuzun, U., & Cross, M. (2004). Numerical predictions of particle degradation in industrial-scale pneumatic conveyors. *Powder Technology*, 143-144, 321-330.

Codex Alimentarius (2007). *Codex Alimentarius official standard 72*, Standard for infant formula and formulas for special medical purposes intended for infants. Rev. 1. Codex Alimentarius Commission, Rome, Italy.

European Commission (2006). EC Directive L401: 30.12.2006, Commission Directive 2006/141/EC of 22 December 2006 on infant formulae and follow-on formulae and amending Directive 1999/21/EC. *Official Journal of the European Union*, L401, 1-33.

GEA Niro (2009). GEA Niro Method No. A 5 b: Wettability IDF method. Retrieved from

<http://www.niro.com/niro/cmsdoc.nsf/WebDoc/ndkw6u9bd3> on 20
September 2009.

Han, T., Levy, A., & Kalman, H. (2003). DEM simulation for attrition of salt during dilute-phase pneumatic conveying. *Powder Technology*, 129(1-3), 92-100.

Hogekamp, S., & Schubert, H. (2003). Rehydration of food powders. *Food Science and Technology International*, 9(3), 223–235.

International Dairy Federation (1979). *International IDF Standard 87*, Instant dried milk - Determination of the dispersibility & wettability. International Dairy Federation, Brussels.

Kalman, H. (1999). Attrition control by pneumatic conveying. *Powder Technology*, 104(3), 214-220.

Kalman, H. (2000). Attrition of powders and granules at various bends during pneumatic conveying. *Powder Technology*, 112(3), 244–250.

Kalman, H., & Goder, D. (1998). Design criteria for particle attrition. *Advanced Powder Technology*, 9(2), 153-167.

Konami, M., Tanaka, S., & Matsumoto, K. (2002). Attrition of granules during repeated pneumatic transport. *Powder Technology*, 125(1), 82-88.

Maghsoodloo, S., Ozdemir, G., Jordan, V., & Huang, C-H. (2004). Strengths and limitations of Taguchi's contributions to quality, manufacturing, and process engineering. *Journal of Manufacturing Systems*, 23(2), 73-126.

Marcus, R.D., Leung, L.S., Klinzing, G.E., & Rizk, F. (1990). *Pneumatic conveying of solids: a theoretical and practical approach* (1st ed.). Chapman and Hall, London, UK.

Montgomery, D.C. (2009). *Design and Analysis of Experiments [International Student Version]* (7th ed.). John Wiley & Sons, New York.

Ross, P.J. (1988). *Taguchi techniques for quality engineering: loss function, orthogonal experiments, parameter and tolerance design* (1st ed.). McGraw-Hill, New York.

Salman, A.D., Hounslow, M.J., & Verba, A. (2002). Particle fragmentation in dilute phase pneumatic conveying. *Powder Technology*, 126(2), 109–115.

Samimi, A., Ghadiri, M., Boerefijn, R., Groot, A., & Kohlus, R. (2003). Effect of structural characteristics on impact breakage of agglomerates. *Powder Technology*, 130(1-3), 428–435.

Taguchi, G. (1987). *System of experimental design: engineering methods to optimize quality and minimize costs*. UNIPUB/Kraus International Publications, New York.

Taguchi, G. (1992). *Taguchi methods, research and development*. Quality engineering series: v.1. American Supplier Institute Inc., Michigan, US.

Taylor, T. (1998). Specific energy consumption and particle attrition in pneumatic conveying. *Powder Technology*, 95(1), 1-6.

U.S. Food and Drug Administration (2010 version). Federal Food, Drug, and Cosmetic Act, Sec. 412. [21 USC §350a] Requirements for Infant Formulas. Retrieved from <http://www.fda.gov/RegulatoryInformation/Legislation/FederalFoodDrugandCosmeticActFDCA/default.htm> on 11 January 2010.

Wypych, P.W., & Arnold, P.C. (1993). Minimizing wear and particle damage in pneumatic conveying. *Powder Handling and Processing*, 5(2), 129–134.

Zhang, Z., & Ghadiri, M. (2002). Impact attrition of particulate solids. Part 2: Experimental work. *Chemical Engineering Science*, 57(17), 3671–3686.

7) Figures

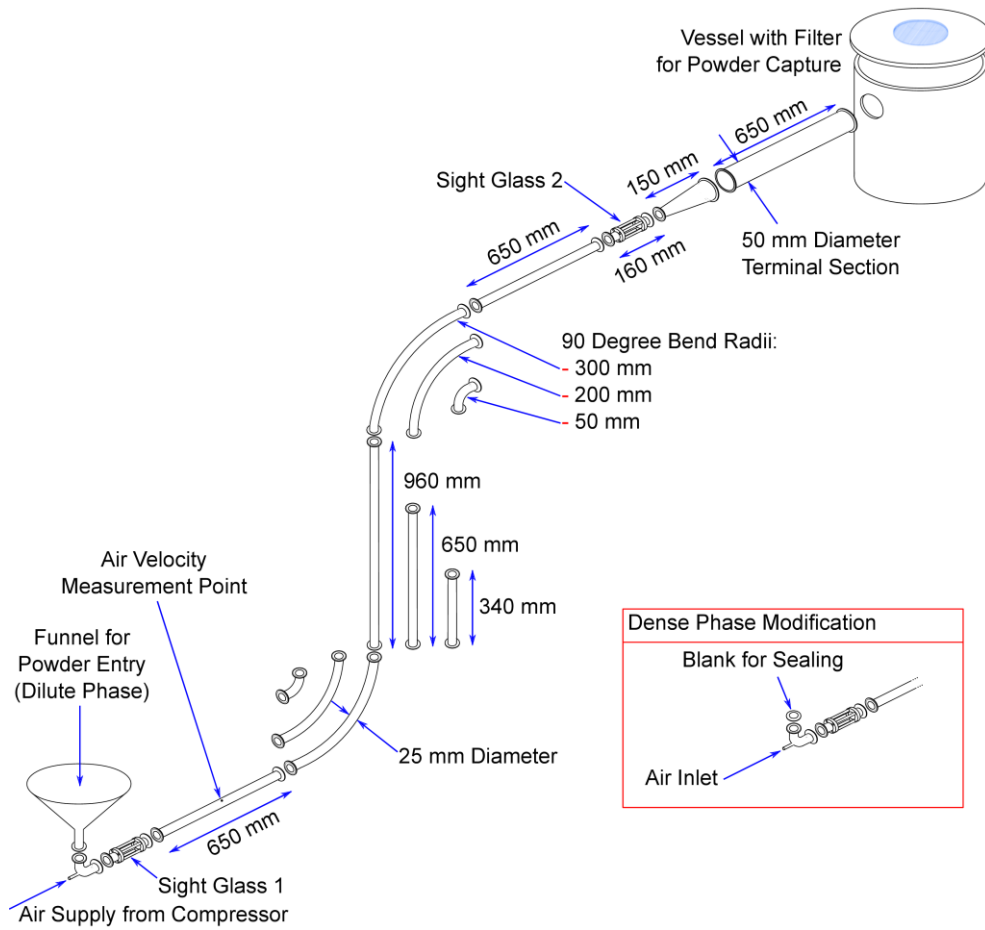


Figure 1: An isometric view of the conveying rig in dilute phase configuration, with an inset showing the modification for dense phase conveying

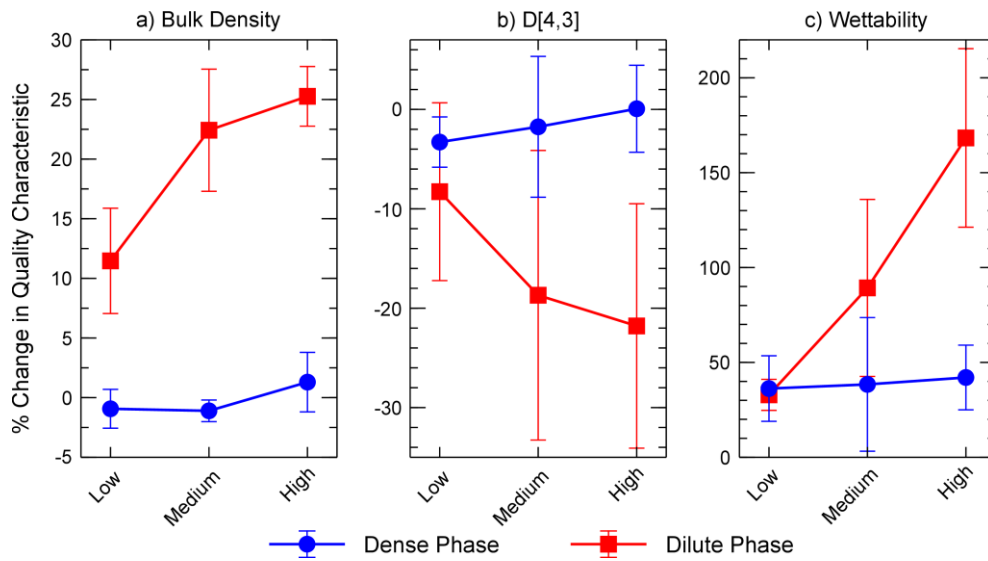


Figure 2: Line graphs showing the average percentage changes in a) bulk density, b) D[4,3] and c) wettability caused by dense phase pneumatic conveying at 3, 4 and 5 m/s and dilute phase conveying at 10, 20 and 30 m/s. The error bars indicate one standard deviation

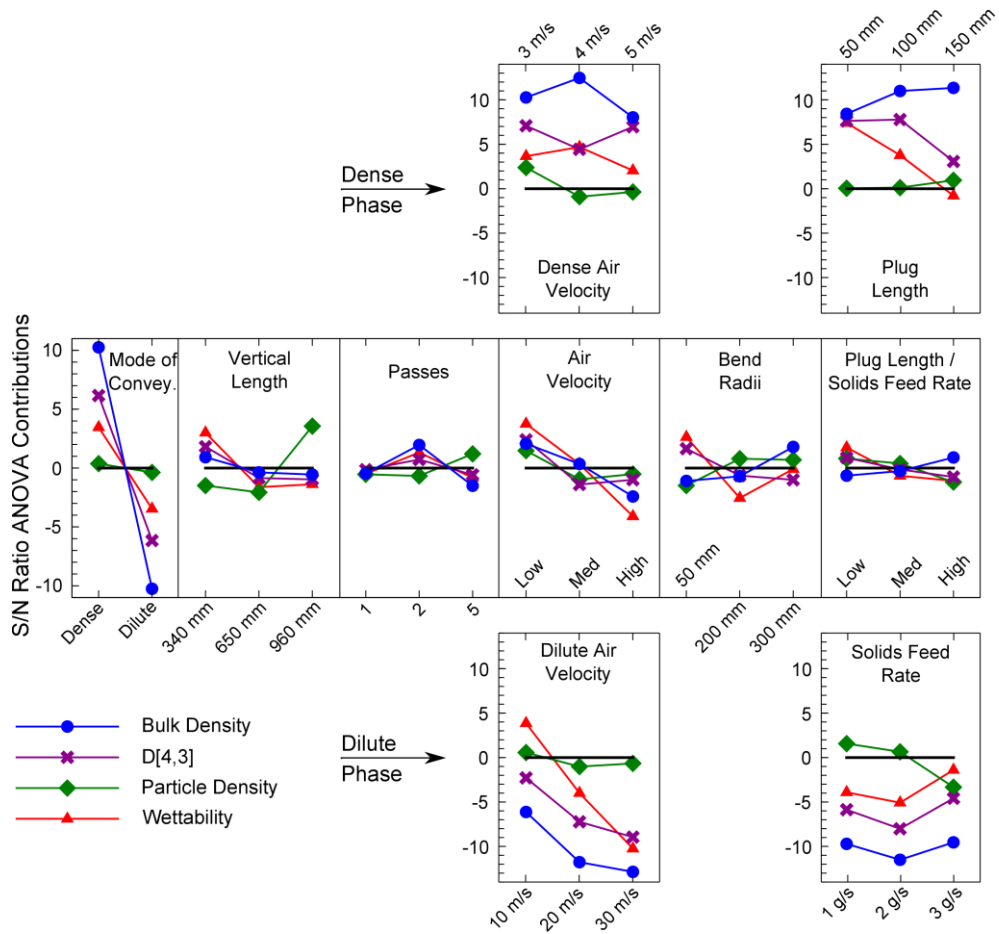


Figure 3: ANOVA contributions plot for S/N ratio responses

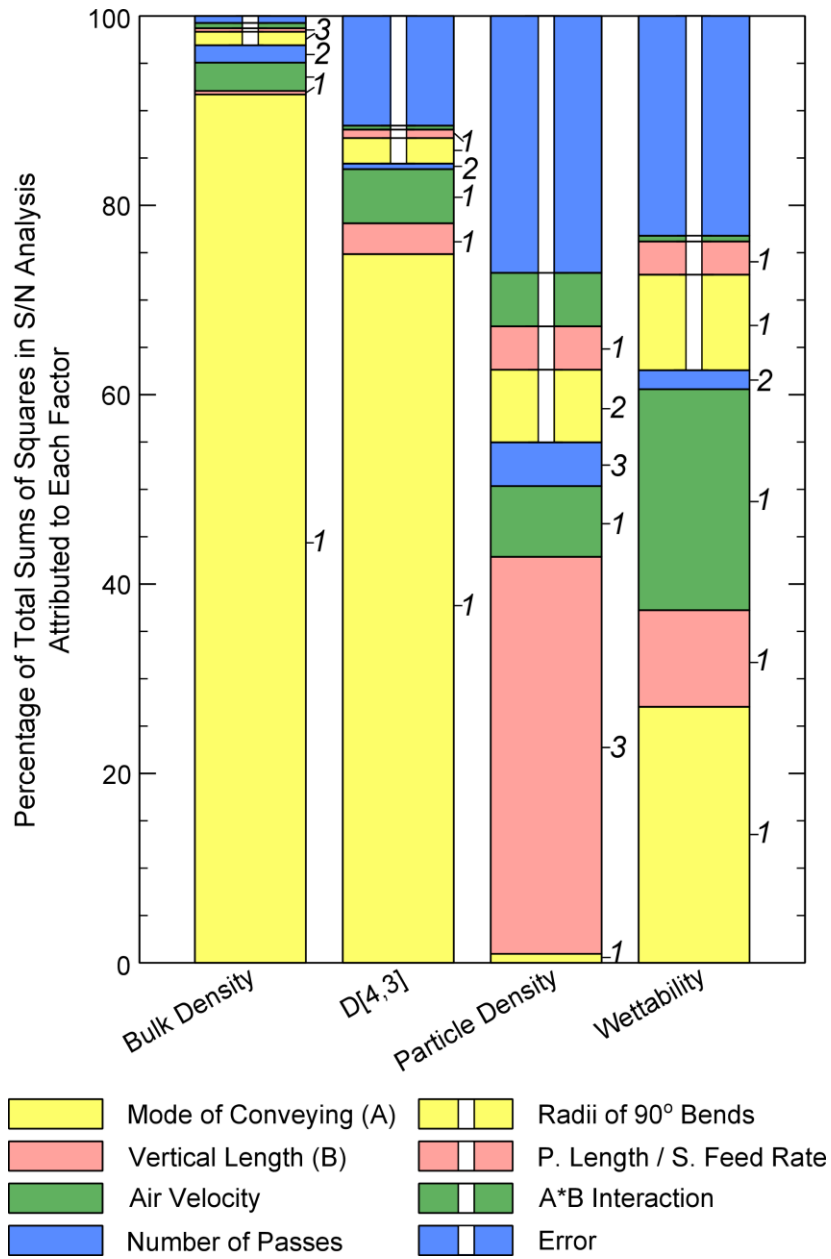


Figure 4: Stacked bar plot showing the distribution of the total sums of squares for the S/N ratio analysis as percentages allocated to each factor, interaction or error and the optimum level of each factor

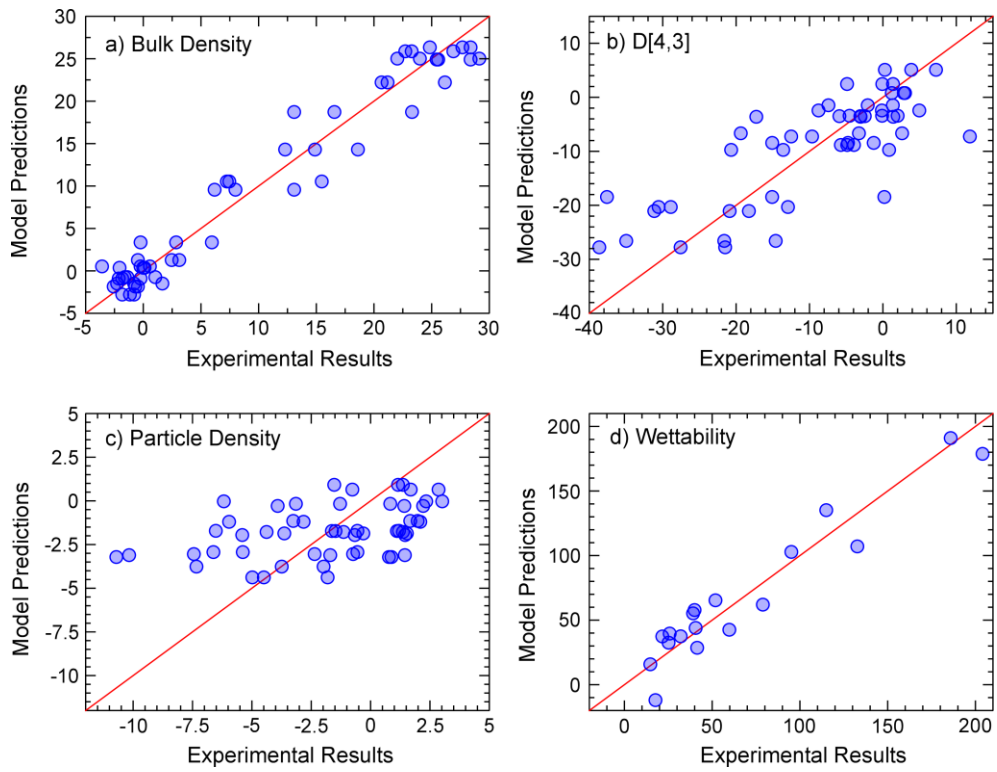


Figure 5: Plots of model predictions versus experimental results for four responses: a) bulk density, b) D[4,3], c) particle density and d) wettability

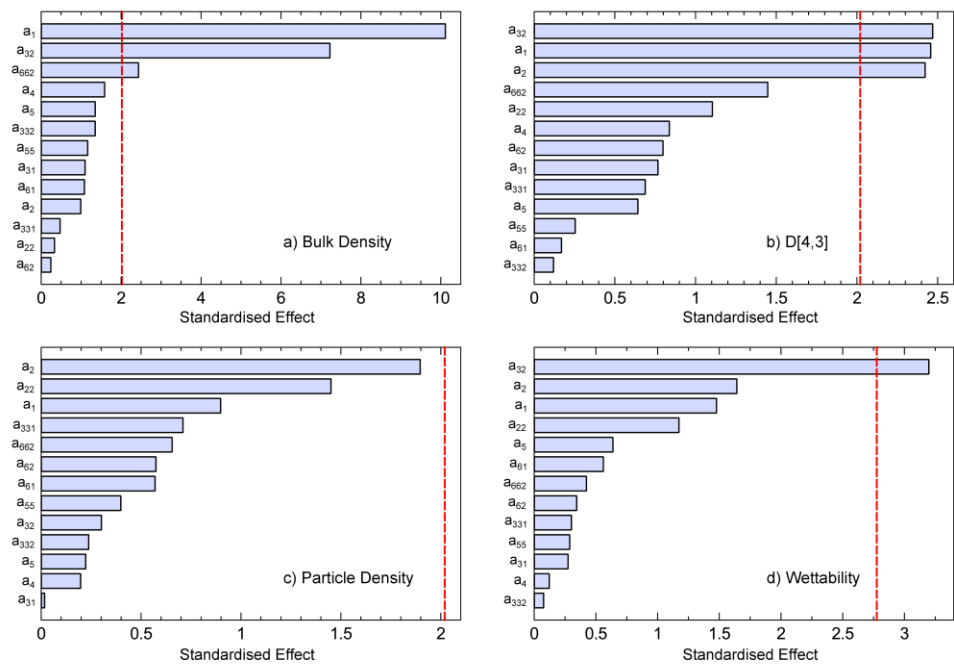


Figure 6: Pareto charts showing the standardised effects for each term in the fitted model for a) bulk density, b) D[4,3], c) particle density and d) wettability, where the dashed lines indicate the 95% significance level

8) Tables

Table 1: Control factors and levels used for the L₁₈ experimental design

Control Factors		Levels		
		1	2	3
Mode of Conveying		Dense	Dilute	
Plug Length	Dense	50 mm	100 mm	150 mm
Solids Feed Rate	Dilute	1 g/s	2 g/s	3 g/s
Air Velocity	Dense	3 m/s	4 m/s	5 m/s
	Dilute	10 m/s	20 m/s	30 m/s
Number of Passes through Rig		1	2	5
Length of Vertical Rig Section		340 mm	650 mm	960 mm
Radii of 90° Bends		50 mm	200 mm	300 mm

Table 2: L₁₈ array showing the columns used after assigning all factors to the array

Row	Column							
	Mode	Vertical Length	Unused Columns		Air Velocity	No. of Passes	Bend Radii	Plug Length / Solids Feed Rate
1	Dense	340 mm	1	1	3 m/s	1	50 mm	50 mm
2	Dense	340 mm	2	2	4 m/s	2	200 mm	100 mm
3	Dense	340 mm	3	3	5 m/s	5	300 mm	150 mm
4	Dense	650 mm	1	1	4 m/s	2	300 mm	150 mm
5	Dense	650 mm	2	2	5 m/s	5	50 mm	50 mm
6	Dense	650 mm	3	3	3 m/s	1	200 mm	100 mm
7	Dense	960 mm	1	2	3 m/s	5	200 mm	150 mm
8	Dense	960 mm	2	3	4 m/s	1	300 mm	50 mm
9	Dense	960 mm	3	1	5 m/s	2	50 mm	100 mm
10	Dilute	340 mm	1	3	30 m/s	2	200 mm	1 g/s
11	Dilute	340 mm	2	1	10 m/s	5	300 mm	2 g/s
12	Dilute	340 mm	3	2	20 m/s	1	50 mm	3 g/s
13	Dilute	650 mm	1	2	30 m/s	1	300 mm	2 g/s
14	Dilute	650 mm	2	3	10 m/s	2	50 mm	3 g/s
15	Dilute	650 mm	3	1	20 m/s	5	200 mm	1 g/s
16	Dilute	960 mm	1	3	20 m/s	5	50 mm	2 g/s
17	Dilute	960 mm	2	1	30 m/s	1	200 mm	3 g/s
18	Dilute	960 mm	3	2	10 m/s	2	300 mm	1 g/s

Table 3: Partial ANOVA table for S/N ratio responses

Factor	v	Bulk Density		D[4,3]		Particle Density		Wettability	
		<i>S</i>	<i>p</i>	<i>S</i>	<i>p</i>	<i>S</i>	<i>p</i>	<i>S</i>	<i>p</i>
Mode of Conveying (A)	1	1892.06	0.000**	682.17	0.007**	2.51	0.731	215.70	0.097[#]
Vertical Length (B)	2	7.75	0.439	29.65	0.610	114.88	0.153	81.37	0.483
Air Velocity	2	61.52	0.039*	52.16	0.448	20.12	0.618	186.24	0.249
Number of Passes	2	37.82	0.082[#]	5.28	0.907	13.09	0.722	16.01	0.847
Radii of 90° Bends	2	29.41	0.116	24.62	0.658	19.95	0.620	80.51	0.486
Plug Length / Solids	2	7.75	0.439	8.14	0.862	13.31	0.718	27.95	0.755
Feed Rate	2	7.75	0.439	8.14	0.862	13.31	0.718	27.95	0.755
A*B Interaction	2	11.47	0.325	3.80	0.932	15.30	0.687	4.87	0.949
Error	4	15.23		105.54		73.93		185.16	
Total	17	2063.01		911.36		273.08		797.81	

[#] 90% significance

* 95% significance

** 99% significance

Table 4: Parameters and p-values of polynomial models fitted to percentage data for four quality characteristics, where factors which are statistically-significant at a 95% level are indicated by an asterisk and are highlighted in bold

Factor	Bulk Density		D[4,3]		Particle Density		Wettability	
	<i>Param.</i>	<i>p</i>	<i>Param.</i>	<i>p</i>	<i>Param.</i>	<i>p</i>	<i>Param.</i>	<i>p</i>
a ₀	13.221	0.000*	-15.715	0.002*	-2.104	0.287	87.653	0.057
a ₁	11.746	0.000*	-8.827	0.018*	1.301	0.375	36.204	0.214
a ₂	0.462	0.330	-3.513	0.020*	1.110	0.065	16.249	0.176
a ₂₂	0.267	0.744	2.773	0.276	1.470	0.155	-20.099	0.306
a ₃₁	1.054	0.281	2.284	0.448	0.019	0.987	5.561	0.798
a ₃₂	-0.715	0.642	3.245	0.496	1.351	0.483	9.663	0.779
a ₃₃₁	6.962	0.000*	-7.361	0.018*	0.362	0.765	65.061	0.033*
a ₃₃₂	-2.056	0.186	0.560	0.906	-0.451	0.814	2.416	0.944
a ₄	1.272	0.121	-2.080	0.408	-0.197	0.845	2.046	0.910
a ₅	-1.049	0.185	1.545	0.525	0.216	0.825	10.463	0.559
a ₅₅	-1.678	0.253	1.132	0.802	-0.718	0.693	-8.759	0.789
a ₆₁	-0.901	0.288	-0.435	0.867	0.595	0.572	9.880	0.606
a ₆₂	-0.199	0.813	2.064	0.430	-0.599	0.570	6.067	0.749
a ₆₆₂	-3.950	0.020*	7.269	0.156	-1.328	0.517	-14.480	0.695

Table 5: Metrics quantifying goodness of fit of the polynomial models

Response	R^2	R^2_{adjusted}	% S [Error]	% S [Pure Error]	% S [Lack of Fit]
Bulk Density	0.954	0.939	4.616	3.911	0.705
D[4,3]	0.620	0.497	37.976	34.223	3.753
Particle Density	0.192	-0.070	80.775	75.908	4.867
Wettability	0.915	0.639	8.484	-	-