Optimization of Spray Drying Parameters for Yogurt-Ice Cream Mix

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The effects of spray drying operating variables such as inlet air temperature, pump speed and feed temperature on final moisture content, particle size and bulk density of yoghurt-ice cream mix powder, and the powder recovery and the drying rate of the drying process were evaluated. Final moisture content decreased with an increase in inlet air temperature and a decrease in pump speed while particle size increased with an increase in both inlet air temperature and pump speed. Bulk density was inversely proportional to particle size. Drying rate increased with an increase in pump speed and feed temperature. Powder recovery increased with an increase in inlet air temperature and feed temperature and a decrease in pump speed. All response models generated through response surface methodology were adequate, having $R^2$ values higher than 0.60 and a coefficient of variation below 10%. The optimum spray drying conditions found for yoghurt-ice cream mix were: inlet air temperature of 180 °C, pump speed of $12 \times 10^3$ rpm, feed temperature of 43 °C and air pressure of 1–2 bars.

Key Words: response surface methodology, spray drying, yoghurt-ice cream


INTRODUCTION

For years, yoghurt has been considered by consumers as a healthy product. This trend was shown by the continuous increase in the sale of yoghurt in the form of drinkable yoghurts, yoghurt-ice cream, yoghurt mousse, probiotic yoghurts and low-fat yoghurts (Dello Staffolo et al. 2004).

Loyola (1984) conducted a study to introduce yoghurt-ice cream and determine the most acceptable proportion of yoghurt to ice cream. They found that 75:25 yoghurt-to-ice cream proportion was the most preferred formulation while the 85:15 proportion was the least preferred. Lately, frozen yoghurt and yoghurt-ice cream outlets have proliferated as consumers who did not particularly prefer yoghurt have now developed a liking for it, in frozen form as ice cream. Some of the most popular foreign franchise brands of frozen yoghurt in the Philippines include Red Mango (South Korea, http://www.redmango.com.ph), Californiaberry (USA), Tutti Frutti (USA, http://www.tuttifrutti.com.ph), and White Hat (Italy, http://www.thewhitehat.com.ph) with 10, 13, 21, and 32 stores nationwide, respectively. Most of the outlets that sell frozen yoghurt utilize imported frozen yoghurt powder mixes. Three of the leading manufacturers and international wholesalers of soft-serve frozen yoghurt powder mix that distribute their products to more than 10 countries are Cielo (USA, Frozen Yogurt Co., http://www.cielousa.com), YoCream (USA, http://www.yocream.com) and Yoflavor (USA, Wellspring, http://www.yoflavor.com). They supply frozen yoghurt powder mixes to stores in over 15, 18 and 30 countries worldwide, respectively.

Although the technology for making yoghurt is well established and simple, the production of yoghurt-ice cream mix (combination of yoghurt and ice cream mix) powder/frozen yoghurt mix powder is more complicated. This is the reason why frozen yoghurt or yoghurt-ice cream outlets in the Philippines use imported frozen yoghurt mix powders. Drying conditions must preserve the viability of the yoghurt bacteria and, at the same time, yield an acceptable frozen yoghurt/yoghurt-ice cream product when reconstituted and frozen.


Spray drying is one method used to preserve yoghurt by converting it into a stable powder form. This method is a continuous operation which is adjustable to full automatic control. Drying rates range from a few kilos at a time to 100 ton h⁻¹ (Gharsallaoui et al. 2007). In this drying technique, there is minimal thermal damage due to extremely short exposure time and cooling effects in a critical drying period, thus making it suitable for heat-sensitive products (Bhandari et al. 1997). Producing yoghurt powder using spray drying requires a more careful step to preserve the viability of the lactic acid bacteria. The particle size of spray dried powder ranges from 1 to 5 μm, with narrow particle size distribution. Another advantage of spray drying technology is the fact that it is a simple and rapid process.

Spray drying generally produces powder with reduced moisture content that could easily be transported and stored. However, the quality, acceptability and other physico-chemical properties of spray dried products may vary depending on different process variables such as feed viscosity, flow rate, drying air temperature as well as the type of atomizer. It is therefore important to optimize these spray drying parameters to produce powders with better yield and physico-chemical properties (Tee et al. 2012).

This study aimed to establish optimum spray drying conditions for yoghurt-ice cream mix and to develop a spray dried yoghurt-ice cream mix powder that can be reconstituted and frozen into an acceptable yoghurt-ice cream product.

**MATERIALS AND METHODS**

This study was conducted from June to December 2011. Mother and bulk starter culture preparation, microbiological analyses and spray drying of yoghurt-ice cream mix were conducted at the Food Microbiology Laboratory of the Food Science Cluster, University of the Philippines Los Baños (UPLB); and the preparation of yoghurt-ice cream mix and some chemical analyses of reconstituted yoghurt-ice cream mix were conducted at the Dairy Training and Research Institute (DTRI) of the Animal and Dairy Sciences Cluster (ADSC), UPLB.

**Milk, Cream and Yoghurt Culture**

Raw cow’s milk, cream and yoghurt consisting of mixed *Streptococcus thermophilus* and *Lactobacillus delbrueckii* ssp. *bulgaricus* used in the experiment were obtained from DTRI. Raw milk and cream samples were analyzed for fat content using the Gerber method (British Standard Institution, BS 696, 1955) wherein milk fat was separated from proteins by adding sulfuric acid (1.82–1.83 sp. gr. at 15.5 °C). The separation was facilitated by adding amyl alcohol (0.82–0.83 sp. gr.) and by centrifugation. The fat content was read directly through a special calibrated butyrometer. Milk solids nonfat (MSNF) was determined by the equation:

\[
\% \text{ MSNF} = \left(100 - \% \text{ milk fat}\right) \times 0.09
\]

where 0.09 is a constant value since the milk serum (MSNF + water) is always assumed to contain 9% MSNF (Arbuckle 1986).

The freeze-dried yoghurt culture was revived in 10 mL sterile 12% reconstituted skimmed milk powder (RSMP) and incubated at 42 °C until it has clotted. Subsequent transfers at 2.5% inoculation rate were carried out using the same medium and incubation temperature for intermediate and bulk starter cultures.

**Preparation of Yoghurt Mix**

The composition of yoghurt mix used in this study consisted of 2.0% fat, 14% MSNF, 6.5% sugar, 2.0% cassava flour and 2.5% lactic acid starter, for a total of 24.5% total solids. This composition was based on FAO/WHO (2000) standard composition of partially skimmed yoghurt consisting of maximum milk fat of < 3.0% and minimum MSNF of 8.2%. Cassava flour was dissolved in a little amount of cold water and then heated until gelatinized. Dry mixture of sugar and skimmed milk powder were dissolved in the pre-heated (50 °C) mixture of milk and water for 10 min. The mixture, including the gelatinized cassava flour solution, was heated up to 65–70 °C and then homogenized at 13,790 kPa.

After homogenization, the mix was pasteurized at 88–90 °C for 10 min, then cooled to around 40 °C before addition of 2.5% yoghurt starter. The yoghurt mix was then incubated at 42 °C until it has clotted. The yoghurt was kept in the refrigerator for 2 d to allow for further acidity development. It was analyzed for pH using Pocket Pen Type pH meter and acidity (% LA) wherein a known volume of milk was titrated with 0.1 N NaOH (1.03 sp. gr.) to the pale pink endpoint of an indicator such as phenolphthalein. Lactic acid was computed using Eq. 2:

\[
\% \text{ lactic acid} = \left[\frac{\text{Vol. of } 0.1 \text{ NaOH} \times 0.009}{\text{Wt. of sample}}\right] \times 100
\]

where wt. of sample = volume of milk × specific gravity (Wehr and Frank 2004).

**Preparation of Ice Cream Mix**

The ice cream mix used in this study consisted of 10% fat, 11% MSNF, 13% sugar, 0.5% cassava flour (food grade) and 0.5% emulsifier and stabilizer mix (Cremodan®SE 709, Denmark), for a total of 35% total solids. Cremodan® and cassava flour were used as the emulsifier-stabilizer blend. Both Cremodan® and cassava flour were dissolved in a little amount of cold water and then heated until gelatinized. Cream (DTRI, UPLB) was added to the milk base (DTRI pasteurized raw milk, UPLB) at temperature of 25 °C while the dry materials were added to the mix.
were added at 45–50 °C. The temperature was increased to 65–70 °C for 2 min, after which, the mix was homogenized at 13,790 kPa. The mix was then pasteurized at 85 °C for 5 min and then cooled to below 4 °C. The mix was aged at 4 °C for at least 24 h.

Preparation of Yoghurt and Ice Cream Mix
The yoghurt-ice cream mix for the experiment consisted of 75% yoghurt and 25% ice cream mix. This proportion of yoghurt to ice cream mix was based on the most preferred formulation found by Loyola (1984). The ratio of yoghurt to ice cream may vary from 5%–70% depending on the preferred level of acidity (Schmidt et al. 1997). Table 1 shows the composition of yoghurt-ice cream mix before spray drying. The lots, consisting of 1.5 L each of yoghurt-ice cream mix, were subjected to different spray drying treatments. The yoghurt-ice cream mix was also analyzed for pH and acidity (% LA). The percentage of total soluble solids of the feed was made constant in all the spray drying treatments in this study. Values for total soluble solids (TSS) of the feed prior to spray drying were validated using a Hand-held Optical Refractometer IP65 to make sure that TSS values did not vary from each other significantly.

Spray Drying of Yoghurt-Ice Cream Mix
The Niro Mobile Spray Dryer (Niro SEA Pte. Ltd., Singapore, Type: MobileMinor™ - Basic Model) was used to spray dry the yoghurt-ice cream mix. The spray drying unit converted the feed into powder in a continuous one-step operation. The standard procedure of the Niro Mobile Spray Dryer for spray drying was followed. Prior to the spray drying procedure, the feed was weighed and samples for pH and acidity were collected. The pH and acidity values of the feed were later compared with the values obtained from the reconstituted spray dried yoghurt-ice cream mix. During the spray drying operation, ambient air temperature, outlet temperature and the drying time were closely monitored. The drying time of each run was recorded to calculate the drying rate. The feed temperature, one of the operating variables, was held constant at 30, 40 and 50 °C, respectively, during the spray drying procedure by placing the feed in a water bath at a temperature of 65–100 °C depending on the desired feed temperature. The temperature of the water bath was maintained using an electric heater (Hanabishi single electric stove with low, medium and high heat settings) and was closely monitored. Dried yoghurt-ice cream mix powder was collected in the receiving bottle at the base of the cyclone. The powdered mix was weighed and packed in a polypropylene plastic bag, refrigerated at 4 °C for 24 h, and then analyzed. The final weight of the powder from each run was used to calculate percentage powder recovery.

<table>
<thead>
<tr>
<th>Component</th>
<th>% In Yoghurt Ice Cream Mix</th>
<th>% In Yoghurt Ice Cream Mix</th>
<th>% In Yoghurt Ice Cream Mix</th>
<th>% In Yoghurt Ice Cream Mix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fat</td>
<td>2</td>
<td>1.5</td>
<td>10</td>
<td>2.5</td>
</tr>
<tr>
<td>SMP</td>
<td>14</td>
<td>10.5</td>
<td>11</td>
<td>2.75</td>
</tr>
<tr>
<td>Sugar</td>
<td>6.5</td>
<td>4.88</td>
<td>13</td>
<td>3.25</td>
</tr>
<tr>
<td>Cassava flour</td>
<td>2</td>
<td>1.5</td>
<td>0.5</td>
<td>0.13</td>
</tr>
<tr>
<td>Cremodan</td>
<td>--</td>
<td>--</td>
<td>0.5</td>
<td>0.13</td>
</tr>
<tr>
<td>SMP—skimmed milk powder</td>
<td></td>
<td></td>
<td>1.63</td>
<td>0.13</td>
</tr>
<tr>
<td>% total solids</td>
<td>27.13</td>
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<td></td>
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</tr>
</tbody>
</table>

Table 1. Composition of yoghurt-ice cream mix before spray drying.

Experimental Design
Response surface methodology (RSM) was used to optimize the spray drying conditions. A Box and Behnken factorial design (Montgomery 1991) with three factors and three levels was used. The independent variables were: inlet air temperature (X1), pump speed (X2) and feed temperature (X3) while the measures of spray dryer performance or responses (dependent variables) were: final moisture content (Y1), particle size (Y2) and bulk density (Y3) of yoghurt-ice cream mix powder, powder recovery (Y4) and drying rate (Y5). The experimental design consisted of 15 runs. The levels of the independent variables were coded as −1, 0 and +1, with 0 as the center point. The variables, symbols and levels are shown in Table 2 and the spray drying treatments in Table 3. Each run was replicated three times.

The final moisture content, particle size and bulk density of the powder recovered from each run were determined.

Statistical Analysis
The second-order polynomial equation (Eq. 3) with k independent variables was fitted to the experimental values using the response surface regression of Design Expert (Version 8.0.7.1).

\[
Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ij} x_i x_j + \sum \beta_{ijk} x_i x_j x_k,
\]

where \( \beta_0, \beta_i, \beta_{ij} \) and \( \beta_{ijk} \) are constant coefficients, and \( x_i \) and \( x_j \) are the coded independent variables.

The three independent variables were studied and the quadratic model for each response was expressed in Eq. 4:

\[
Y = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_12 x_1 x_2 + b_13 x_1 x_3 + b_23 x_2 x_3 + b_{12} x_1 + b_{13} x_1 + b_{23} x_2 + b_{123} x_1 x_2 x_3
\]

\[
(4)
\]

where \( b_0 \) is the intercept, \( b_1, b_2 \) and \( b_3 \) are the linear terms, \( b_{11}, b_{22} \) and \( b_{33} \) are the quadratic terms, \( b_{12}, b_{13} \) and \( b_{23} \) are the cross product regression terms, and \( x_1, x_2 \) and \( x_3 \) are the coded independent variables.
Regression analysis was done using the response surface regression procedure of Statistical Analysis System (SAS Version 9.1). The coefficient of determination ($R^2$), the coefficient of variation (CV), and regression constants were obtained and analysis of variance (ANOVA) was also done. Lack of fit measures the variation of the data around the fitted model. Lack of fit becomes significant once the model does not fit the data well. On the other hand, $R^2$ measures the amount of variation around the mean explained by the model. CV, which is the standard deviation expressed as a percentage of the mean, is a measure of reproducibility of the models. A CV of less than 10% means that the model is highly reproducible (Rustom et al. 1991). Acceptable significance level was at 5%. Surface plots were obtained using Design Expert (Version 8.0.7.1). Overlay plots wherein critical response contours were overlaid on a contour plot to visually search for the optimum region were obtained.

Table 2. Variables and their levels for the Box and Behnken factorial design.

<table>
<thead>
<tr>
<th>Independent Variables</th>
<th>Symbols</th>
<th>Coded-Variable Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Coded Uncoded</td>
</tr>
<tr>
<td>Inlet temperature, °C</td>
<td>IT</td>
<td>-1 140 160 180</td>
</tr>
<tr>
<td>Pump speed, rpm * 10^3</td>
<td>PS</td>
<td>10 13 16</td>
</tr>
<tr>
<td>Feed temperature, °C</td>
<td>FT</td>
<td>30 40 50</td>
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</tbody>
</table>

Table 3. Spray drying treatments.

<table>
<thead>
<tr>
<th>Runs</th>
<th>Inlet Air Temperature, °C</th>
<th>Pump Speed, rpm * 10^3</th>
<th>Feed Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>160</td>
<td>13</td>
<td>40</td>
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<tr>
<td>2</td>
<td>140</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>3</td>
<td>160</td>
<td>16</td>
<td>50</td>
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<tr>
<td>4</td>
<td>180</td>
<td>10</td>
<td>40</td>
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<tr>
<td>5</td>
<td>180</td>
<td>13</td>
<td>30</td>
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<td>6</td>
<td>180</td>
<td>13</td>
<td>50</td>
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<td>7</td>
<td>160</td>
<td>13</td>
<td>30</td>
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<td>8</td>
<td>140</td>
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<td>160</td>
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</tr>
<tr>
<td>15</td>
<td>160</td>
<td>13</td>
<td>40</td>
</tr>
</tbody>
</table>

Physico-chemical and Microbiological Analyses

Moisture content (IDF Standard 26A:1993). One gram of the prepared test sample, transferred into a pre-weighed dish and weighed to the nearest 0.1 mg, was placed inside an oven with a controlled temperature of 102 ± 2 °C for 2 h. Heating was repeated until the difference in mass between two successive weighings did not exceed 0.5 mg. The lowest mass was recorded and moisture content-wet basis (MCwb) was calculated using Eq. 5:

$$\text{MCwb} = \left(\frac{S_1 - S_2}{S_1}\right) \times 100 \quad (5)$$

where $MC_{wb} = \%$, $S_1 =$ weight of the sample before drying and $S_2 =$ weight of the dried sample.

Particle size. An optical microscope equipped with a calibrated micrometer eyepiece was used to measure the particle size of the yoghurt-ice cream mix powder. A drop of petroleum ether was mounted on the slide to prevent individual powder particles from sticking to each other. The average size of 50 particles was obtained and the results were reported in micrometer ($\mu$m).

Bulk density. One hundred grams of powder was measured in a graduated cylinder. The cylinder was manually tapped 100 times with the same intensity. The volume of the powder was then recorded after tapping. Bulk density was calculated by dividing the weight of the powder (100 g) by the volume obtained after tapping (Haugaard et al. 1978).

Spray Drying Performance

Powder recovery. Powder recovery was determined based on the total solids of the feed. It was computed using Eq. 6:

$$\% \text{ Recovery} = \frac{\text{Weight of powder}}{\text{Initial weight of the feed}} \times \left(\frac{\% \text{ total solids}}{100}\right) \quad (6)$$

where % total solids = 100 – moisture content of the feed.

Drying rate. The average drying rate ($DR_{ave}$, kg water h⁻¹) was computed using Eq. 7:

$$DR_{ave} = \frac{m_f - m_p}{t} \quad (7)$$

where $m_f =$ amount of water in the feed (kg), $m_p =$ amount of water in the powder (kg) and $t =$ drying time (h).

RESULTS AND DISCUSSION

Spray Drying on the pH and Acidity of Reconstituted Yoghurt-Ice Cream Mix

Spray drying significantly increased the pH and decreased (p<0.05) the acidity of reconstituted yoghurt-ice cream mix (Table 4). Fresh milk, which is a major component of yoghurt, contains 200 mg CO₂. About 50% of the CO₂ is driven off on standing, with additional losses during heating. This process results in an increase in pH and a decrease in titratable acidity. Upon heating, colloidal Ca₃(PO₄)₂ is formed, which compensates for the loss of CO₂. The reaction is partially reversible following a severe heat treatment. When the milk becomes concentrated, the shifts in Ca₃(PO₄)₂ and pH increase (Fox and McSweeney 1998).
Table 4. Acidity (% lactic acid, LA) and pH of yoghurt-ice cream mix before spray drying (A) and the reconstituted spray dried yoghurt-ice cream mix powder (B).

<table>
<thead>
<tr>
<th>Runs</th>
<th>pH A</th>
<th>pH B</th>
<th>Acidity (LA) A</th>
<th>Acidity (LA) B</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>5.2</td>
<td>0.66</td>
<td>0.6</td>
</tr>
<tr>
<td>2</td>
<td>5.2</td>
<td>5.2</td>
<td>0.59</td>
<td>0.57</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>5</td>
<td>0.66</td>
<td>0.61</td>
</tr>
<tr>
<td>4</td>
<td>5.1</td>
<td>5.3</td>
<td>0.66</td>
<td>0.61</td>
</tr>
<tr>
<td>5</td>
<td>5.2</td>
<td>5</td>
<td>0.61</td>
<td>0.59</td>
</tr>
<tr>
<td>6</td>
<td>5.2</td>
<td>5.3</td>
<td>0.74</td>
<td>0.68</td>
</tr>
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<td>7</td>
<td>5.2</td>
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<td>0.5</td>
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<tr>
<td>8</td>
<td>5.1</td>
<td>5.3</td>
<td>0.59</td>
<td>0.6</td>
</tr>
<tr>
<td>9</td>
<td>5.1</td>
<td>5.3</td>
<td>0.6</td>
<td>0.57</td>
</tr>
<tr>
<td>10</td>
<td>5.2</td>
<td>5.2</td>
<td>0.63</td>
<td>0.62</td>
</tr>
<tr>
<td>11</td>
<td>5.2</td>
<td>5.3</td>
<td>0.63</td>
<td>0.6</td>
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<tr>
<td>12</td>
<td>5.2</td>
<td>5.3</td>
<td>0.65</td>
<td>0.61</td>
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<tr>
<td>13</td>
<td>5.3</td>
<td>5.5</td>
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<td>14</td>
<td>5</td>
<td>5.1</td>
<td>0.64</td>
<td>0.59</td>
</tr>
<tr>
<td>15</td>
<td>5.2</td>
<td>5.2</td>
<td>0.65</td>
<td>0.58</td>
</tr>
</tbody>
</table>

Mean 5.15 5.24 0.63 0.60

The difference between the means of A and B was significant at p ≤ 0.05 using t-test.

Spray Drying Conditions on Characteristics of Yoghurt-Ice Cream Mix Powder

Inlet air temperature significantly affected all the responses except drying rate while pump speed significantly affected all the responses (Table 5). Feed temperature significantly affected particle size and powder recovery but did not affect drying rate, final moisture content and bulk density.

Model Fitting

The regression coefficients for second-order polynomials for each response variable are shown in Table 6. Adequacy and degree of fit were validated for each response variable by ANOVA (Table 7).

All the models generated for moisture content, particle size, bulk density, powder recovery and drying rate were adequate and R² values were satisfactory (Table 7). The lack of fit was not significant except for the model for moisture content, which had a significant lack of fit at 1% level. However, considering the R² value (0.88) and the coefficient of variation, which is below 10% (3.70%), and model significance, the model developed for moisture content can still be considered acceptable. All the models developed for each of the responses were found to be statistically significant with R² values higher than 0.60 and CV values below 10%.

Since the models developed for all the responses were significant, then the response surface plots based on the said models can be plotted accurately.

Operating Variables on Yoghurt-Ice Cream Mix Powder Characteristics, Drying Rate and Powder Recovery

The characteristics of the resulting powder could be affected by different spray drying operating variables and the feed properties as well. The powder produced was

Table 5. Analysis of variance showing the effect of treatment variables, as a linear, quadratic, or interaction term on each of the responses.

<table>
<thead>
<tr>
<th>Process Variables</th>
<th>Moisture Particle Content Size μm (Y1)</th>
<th>Bulk Density g mL⁻¹ (Y2)</th>
<th>Powder Recovery % (Y3)</th>
<th>Drying Rate kgwater h⁻¹ (Y4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>df</td>
<td>% (Y1)</td>
<td>% (Y2)</td>
<td>% (Y3)</td>
<td>% (Y4)</td>
</tr>
<tr>
<td>Intercept</td>
<td>3.763</td>
<td>37.984</td>
<td>0.711</td>
<td>76.123</td>
</tr>
<tr>
<td>X₁</td>
<td>-0.397 ''</td>
<td>1.075 ''</td>
<td>-0.013 ''</td>
<td>6.791 ''</td>
</tr>
<tr>
<td>X₂</td>
<td>0.167 ''</td>
<td>2.168 ''</td>
<td>-0.055 ''</td>
<td>-4.130 ''</td>
</tr>
<tr>
<td>X₃</td>
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<td>0.017 ns</td>
<td>0.004 ns</td>
<td>1.392 ''</td>
</tr>
<tr>
<td>X₁*X₂</td>
<td>0.053 as</td>
<td>0.894 as</td>
<td>-0.008 as</td>
<td>-5.337 ''</td>
</tr>
<tr>
<td>X₁*X₃</td>
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<td>1.289 ''</td>
<td>0.002 ''</td>
<td>-3.037 ''</td>
</tr>
<tr>
<td>X₂*X₂</td>
<td>-0.073 ns</td>
<td>0.707 ns</td>
<td>-0.001 ns</td>
<td>-2.685 ''</td>
</tr>
<tr>
<td>X₂*X₃</td>
<td>0.018 as</td>
<td>-0.063 ns</td>
<td>0.002 ns</td>
<td>-2.550 ''</td>
</tr>
<tr>
<td>X₃*X₃</td>
<td>0.008 as</td>
<td>-0.426 ns</td>
<td>0.002 ''</td>
<td>1.245 ns</td>
</tr>
<tr>
<td>X₁*X₁</td>
<td>0.107 as</td>
<td>-1.091 as</td>
<td>0.007 ns</td>
<td>0.642 ns</td>
</tr>
</tbody>
</table>

significant at 1% level; X₁ = inlet air temperature
significant at 5% level; X₂ = pump speed
not significant; X₃ = feed temperature

Table 6. Regression coefficients based on coded data of the second-order polynomials representing the relationships between the responses and independent variables.

<table>
<thead>
<tr>
<th>Process Variables</th>
<th>Moisture Particle Content Size μm (Y₁)</th>
<th>Bulk Density g mL⁻¹ (Y₂)</th>
<th>Powder Recovery % (Y₃)</th>
<th>Drying Rate kgwater h⁻¹ (Y₄)</th>
</tr>
</thead>
<tbody>
<tr>
<td>df</td>
<td>% (Y₁)</td>
<td>% (Y₂)</td>
<td>% (Y₃)</td>
<td>% (Y₄)</td>
</tr>
<tr>
<td>Intercept</td>
<td>4.92‡</td>
<td>192.43</td>
<td>0.08</td>
<td>2159.54†</td>
</tr>
<tr>
<td>Linear</td>
<td>4.47‡</td>
<td>140.60</td>
<td>0.06</td>
<td>1562.62‡</td>
</tr>
<tr>
<td>Quadratic</td>
<td>3.23‡</td>
<td>29.66</td>
<td>0.001**</td>
<td>289.64**</td>
</tr>
<tr>
<td>Cross product</td>
<td>2.23‡</td>
<td>22.17</td>
<td>0.001**</td>
<td>207.29**</td>
</tr>
</tbody>
</table>

significant at 1% level; X₁ = inlet air temperature
significant at 5% level; X₂ = pump speed
not significant; X₃ = feed temperature

Table 7. Analysis of variance showing the effect of treatment variables, as a linear, quadratic, or interaction term on each of the responses.

<table>
<thead>
<tr>
<th>Process Variables</th>
<th>Moisture Content % (Y₁)</th>
<th>Particle Size μm (Y₂)</th>
<th>Bulk Density g mL⁻¹ (Y₃)</th>
<th>Powder Recovery % (Y₄)</th>
<th>Drying Rate kgwater h⁻¹ (Y₅)</th>
</tr>
</thead>
<tbody>
<tr>
<td>df</td>
<td>% (Y₁)</td>
<td>% (Y₂)</td>
<td>% (Y₃)</td>
<td>% (Y₄)</td>
<td>% (Y₅)</td>
</tr>
<tr>
<td>Model</td>
<td>9</td>
<td>4.92</td>
<td>192.43</td>
<td>0.08</td>
<td>2159.54†</td>
</tr>
<tr>
<td>Linear</td>
<td>3</td>
<td>4.47</td>
<td>140.60</td>
<td>0.06</td>
<td>1562.62‡</td>
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<td>3</td>
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</tr>
<tr>
<td>Cross product</td>
<td>3</td>
<td>2.23</td>
<td>22.17</td>
<td>0.001**</td>
<td>207.29**</td>
</tr>
</tbody>
</table>

significant at 1% level
significant at 5% level
not significant

---

**Note:** This text is a natural representation of the content as it appears on the page, without altering or reformatting the original layout or structure.
fine and dusty and did not reconstitute well in water at room temperature. Thus, the temperature of water used was 50 ºC for better reconstitution.

**Moisture Content**
The effects of inlet air temperature and pump speed on the final moisture content of yoghurt-ice cream mix powder were highly significant (p<0.01) while feed temperature had no effect on final moisture content (Table 5). The model equation (Eq. 8) generated for moisture content was:

\[
\text{Moisture Content} = 3.76 - 0.40X_1 + 0.17X_2 - 0.026X_3 + 0.14X_2X_1 + 0.019X_2X_1 + 0.0068X_3X_2 + 0.05X_1^2 + 0.068X_2^2 + 0.10X_3^3
\]  

(8)

Final moisture content increased with a decrease in inlet air temperature and an increase in pump speed as reflected in the slopes obtained from the model equation (Eq. 8) for moisture content: (-) 0.40 and (+) 0.17 for inlet air temperature and pump speed, respectively.

The model was found to be significant at 1% level (Table 7). The coefficient of determination obtained for the model (R²=0.88) was acceptable, which suggests that 88% of the total variation was accounted for by the model.

Moisture content is known to be affected by temperature and humidity levels in the dryer. Differences in the humidity levels in the dryer inlet air due to seasonal variability in ambient air conditions play a significant role in the standardization of the incoming air in the dryer. Dryer capacity is dependent on the total moisture in the air leaving the spray drying chamber. Air with too much moisture will result in a powder which tends to be sticky. Outlet air temperature condition, on the other hand, is the resulting temperature of the heat and mass balance in the drying chamber and thus cannot be controlled. It is also the manifestation of the relative humidity in the outlet air (http://www.niroinc.com). As a general rule, outlet air temperature is equivalent to the maximum product temperature. Outlet temperature is also a result of the combined effects of inlet temperature, aspirator flow rate (quantity of air), peristaltic pump setting and the concentration of the feed being sprayed (Meuri 2002).

Feed rate is regulated by adjusting pump speed. The feed pump used has adjustable speed settings that can produce a range of 5 to 20 × 10³ rpm. However, a constant flow rate is quite difficult to achieve and control since flow rate depends on many factors including the diameter, length and material of the tube, the force pushing on the fluid and the type and viscosity of the spray solution, which may vary from one experiment to another. The speed of the pump, which directly corresponds to the inlet mass, has a significant effect on the temperature difference between the inlet temperature and the outlet temperature. Generally, decreasing the pump speed while holding the inlet temperature constant increases the proportion of dry matter of the final product. Conversely, increasing the pump speed decreases the outlet temperature, thereby increasing the difference between the inlet temperature and the outlet temperature.

A decrease in the outlet temperature may also be due to the higher quantity of the solution since more energy is necessary to evaporate the droplets to particles. Increasing the temperature difference while the inlet temperature is held constant results in an increase in the residual moisture content in the final powder. For the resulting powder with relatively smaller amount of residual moisture, it is advisable to hold the inlet temperature as high as possible and the temperature difference as small as possible. The viscosity of the feed and the diameter of tubing also affect the pump throughput (Meuri 2002). Coefficients for X₁ (inlet air temperature) and X₂ (pump speed) were significant at 1% level (Table 6).

**Particle Size**
The effects of inlet air temperature, pump speed and feed temperature on particle size were significant at 1% level (Table 5). The model equation (Eq. 9) obtained for particle size was:

\[
\text{Particle size} = 37.98 + 1.07X_1 + 2.17X_2 + 0.019X_3 + 1.29X_2X_1 - 0.062X_3X_1 - 0.42X_2X_2 + 0.90X_1^2 + 0.71X_2^2 - 1.09X_3^3
\]  

(9)

Particle size increased with an increase in inlet air temperature and pump speed as reflected in the positive signs of the slopes for inlet air temperature (1.07) and pump speed (2.17), obtained from the model equation (Eq. 9) for particle size.

The model has an R² of 0.90, which suggests that 90% of the total variation was accounted for by the model. The model was also found to be significant at 1% level (Table 7). Coefficients obtained for inlet air temperature and pump speed were significant at 1% level (Table 6).

The effect of drying temperature is dependent on the drying characteristics of the product. An increase in drying air temperature resulted in large particle size of the powder for droplets that have a tendency to expand during drying. However, if the drying air temperature increases to the extent that moisture evaporates too fast that the droplets disintegrate, a powder with smaller particle sizes will be produced (Masters 1972).

An increase in feed rate may slightly cause an increase in the particle size of the powder, which was also evident in the results obtained in this particular study. Faster drying rate, high inlet air temperature and low temperature difference between inlet and outlet air temperatures may result in a powder with particle size...
that is a little larger than the particle size of powder produced under conditions that result in slow drying (Finney et al. 2002).

Other important factors which directly affect the particle size of the final product are mass flow, viscosity, total solids content and surface tension of the feed, milk characteristics, processing conditions and the type of the equipment used in the drying process. For example, condensed milk with higher total solids content produces larger particle size while a low concentrate viscosity results in smaller particle size. Variation in the morphology of the particles results from different drying conditions to which the individual particles were exposed (Caric 1994).

In order to reduce the particle size at a particular feed rate, the nozzle must be replaced by a smaller orifice. Because one of the goals of this optimization study was to maximize the particle size for better reconstitution and flow characteristics of the powder, a nozzle was used instead of a smaller orifice. Only one nozzle was used for all the spray drying treatments.

Particle size also increased with an increase in feed temperature as reflected in the positive slope for feed temperature (0.019), obtained from the model (Eq. 9) for particle size. The results were contrary to those of Trullinger (1997) which showed that particle size was inversely proportional to feed temperature such that low feed temperature resulted in large particle size. This relationship can be explained by the effect of viscosity of the feed on atomization. As the feed temperature decreased, the viscosity increased, resulting in a greater energy supplied in the nozzle to overcome larger viscous forces. As a result, the energy needed to break up the particles decreases, thus producing larger particle size. Figure 1 shows the normal distribution curve for particle size of spray dried yoghurt-ice cream mix.

Bulk Density
ANOVA revealed that both inlet air temperature and pump speed significantly influenced bulk density at 1% level while the effect of feed temperature was not significant (Table 5). The model equation (Eq. 10) obtained for bulk density was:

\[
\text{Bulk density} = 0.71 - 0.014X_1 - 0.055X_2 + 6.40 \times 10^{-5} X_3 - 1.75 \times 10^{-3} X_1 X_3 - 3.75 \times 10^{-4} X_2 X_3 - 1.28 \times 10^{-3} X_1 X_2 - 6.98 \times 10^{-3} X_1^2 - 1.43 \times 10^{-4} X_3^2 + 7.65 \times 10^{-5} X_3^3
\]  

(10)

Bulk density increased with a decrease in inlet air temperature and pump speed as reflected in the negative slopes for inlet air temperature (0.014) and pump speed (0.055), obtained from the model equation (Eq. 10) for bulk density.

The model for bulk density was significant at 1% level with \(R^2\) of 0.89, which suggests that 89% of the total variation was accounted for by the model (Table 7).

Drying Rate
ANOVA (Table 5) showed that pump speed had the greatest influence on drying rate at 1% level. The effects of inlet air temperature and feed temperature were not significant. Response surface regression yielded Eq. 11 for the drying rate:

\[
\text{Drying rate} = 1.02 - 0.026X_1 + 0.19X_2 + 0.049X_3 + 0.029X_2X_1 + 0.021X_3X_1 + 0.023X_2X_3 - 0.043X_1^2 + 0.030X_2^2 + 4.30 \times 10^{-3} X_3^3
\]  

(11)

Pump speed and feed temperature were directly proportional to drying rate as reflected in the positive slopes for pump speed (0.19) and feed temperature (0.049), obtained from the model equation (Eq. 11) for drying rate.

The model was significant at 1% level with \(R^2\) value of 0.79 (Table 7). Lack of fit was not significant. The highest coefficient was that corresponding to \(X_2\) (pump speed) and therefore had the greatest influence on the response value of drying rate (Table 6). This result was somehow expected since an increased pump speed resulted in faster feed injection, thus a shorter drying time was obtained.
The feed temperature, on the other hand, primarily affected the viscosity of the feed. Higher feed temperature resulted in less viscous feed, thus less pressure and time were required to suck the feed from the container into the dryer.

**Powder Recovery**
Proper handling and packaging of powder must be considered because most powders are both thermoplastic and hygroscopic. This common nature of powder could possibly give rise to problems such as adhesion to dryer walls, difficult handling and caking (Mani et al. 2002).

ANOVA showed that all the operating variables (inlet air temperature, pump speed and feed temperature) significantly affected powder recovery (Table 5). The model equation (Eq. 12) obtained for powder recovery was:

\[
\text{Powder recovery} = 76.12 + 6.79X_1 + 4.13X_2 + 1.39X_1^2 - 3.04X_2X_1 - 2.55X_3X_1 + 1.25X_3X_2 - 2.69X_2^2 + 0.64X_3^3
\] (12)

Powder recovery increased with an increase in inlet air temperature and feed temperature, and a decrease in pump speed as reflected in the signs of the slopes obtained from the model equation (Eq. 12) for powder recovery: (+) 6.79, (+) 1.39 and (−) 4.13 for inlet air temperature, feed temperature and pump speed, respectively.

The model was significant at 1% level with \( R^2 \) of 0.87 (Table 7). Lack of fit was not significant. The highest coefficients were those corresponding to \( X_1 \) (inlet air temperature) and \( X_2 \) (pump speed) (Table 6).

By reducing the pump speed or the feed injection, the time of exposure of the atomized particles in the heated air was longer, resulting in a less sticky final product and high powder recovery. Higher feed temperature resulted in less viscous feed, thus increasing the feed rate. Consequently, an increase in feed rate resulted in large particle size of the powder. Because of this large particle size, lesser fines were carried away by the exhaust air, thus powder recovery also increased (Miranda 2006).

As explained earlier, as the pump speed or the feed rate is reduced, the outlet temperature increased, thus resulting in a small difference in temperature between inlet temperature and outlet temperature. Consequently, the residual moisture in the final product is reduced (Meuri 2002).

If the outlet temperature is very low, the product particles in the drying chamber will not dry sufficiently within the time allowed, causing sticky particles which may cause blocking or plugging (http://www.niroinc.com). Aside from adhesion of the product particles in the chamber wall, high residual moisture may also give rise to poor powder recovery.

Drying chamber design and air flow rate determine the droplet residence time in the chamber wall. Once the desired moisture removal from the droplets is completed, the product is removed from the dryer before the temperature of the product reaches the outlet drying air temperature. Consequently, the product is less likely to be damaged by heat. The two common systems used to separate the product from the drying medium include the primary separation of the drying product, which takes place at the base of the drying chamber, and the total recovery of the final product in the separation equipment. The most common separation equipment is the cyclone, which was used in this optimization study. Separation is based on inertial forces wherein the particles are separated from the cyclone wall and removed (Meuri 2002).

**Optimization of Spray Drying Conditions for Yoghurt-Ice Cream Mix**
Design Expert 8.0.7.1 was used to generate an overlay graph, which is composed of the contour plots (Fig. 2–6) from each property laid on top of each other.

A criterion was set for each of the properties measured. Both lower and upper limits were filled up to assure that values were within the specified range. Table 8 shows the goals for each response and the optimum spray drying parameters for yoghurt-ice cream mix.

For final moisture content and bulk density, the goal was to minimize their values in the resulting powder. Minimum values of 2.0% (http://nzic.org.nz) and 0.50 g mL$^{-1}$ (Pisecky 1997) were set for moisture content and bulk density of regular spray dried powder, respectively. However, for particle size, drying rate and powder recovery, the goal was to maximize their values. Maximum values of 250 μm, 1.55 kg water h$^{-1}$ and 85% were set for particle size, drying rate and powder recovery, respectively. Spray dried powder particles are usually spherical in shape with diameters ranging from 10 to 250 μm (Caric 1994). High levels of drying rate and powder recovery are indicators of good spray dryer performance. Powder recovery of 100% is somewhat impossible to achieve since there is a possibility that exhaust air could carry away some powder particles while some might adhere to the chamber wall and pipes of the dryer. Drying rate, on the other hand, could easily be controlled by adjusting the pump speed and the viscosity of the feed.

Moisture content, powder recovery and particle size were considered to be the most important properties, while bulk density and drying rate were regarded as the least important ones. Moisture content, particle size and bulk density affect quality of the final product, while drying rate and powder recovery affect economic performance of the spray drying process.

Figure 7 shows the overlay plots used to visually search for the best compromise. Regions that do not
Fig. 2. Contour plot of final moisture content of spray dried yoghurt-ice cream mix as a function of feed temperature and pump speed (left) and pump speed and inlet air temperature (right).

Fig. 3. Contour plot of particle size of spray dried yoghurt-ice cream mix as a function of feed temperature and pump speed (left) and pump speed and inlet air temperature (right).

Fig. 4. Contour plot of bulk density of spray dried yoghurt-ice cream mix as a function of feed temperature and pump speed (left) and pump speed and inlet air temperature (right).
Fig. 5. Contour plot of drying rate of spray dried yoghurt-ice cream mix as a function of feed temperature and pump speed (left) and pump speed and inlet air temperature (right).

Fig. 6. Contour plot of powder recovery of spray dried yoghurt-ice cream mix as a function of feed temperature and pump speed (left) and pump speed and inlet air temperature (right).

Fig. 7. Overlay plots showing the optimum spray drying parameters and predicted values as a function of feed temperature and pump speed (left) and pump speed and inlet air temperature (right).
satisfy the optimization criteria are shaded gray. Any region that is not gray-shaded satisfies the goals for each property. In this case, the overlay plot, which is colored yellow, shows the region satisfying the optimization criteria.

Experiments were then performed using these optimum conditions to validate the adequacy of the models generated. The predicted and experimental values are summarized in Table 9. In most cases, a percentage error of 5% or below is acceptable. The experimental values obtained for bulk density and moisture content were closest to the predicted values of the model since they have the smallest percentage errors: 2.95% and 5.31%, respectively. However, experimental values for particle size, powder recovery and drying rate were not that close to the predicted values of the model because of high percentage errors: 8.43%, 9.22% and 13.52%, respectively. Average percentage errors obtained from this study are relatively higher than the values obtained from similar optimization studies. In the study of Ghanbarzadeh et al. (2013), which used RSM in the development of Sirolimus liposomes prepared by thin film hydration technique, the average percentage error obtained was 3.59%. Higher prognostic ability of RSM was shown in the study of Mandal et al. (2007), which used RSM in the formulation and optimization of the sustained release matrix tablet Metformin HCl 500 mg with an average percentage error of (± S.D.) 0.0437 ± 0.3285 out of 8 confirmatory runs performed.

CONCLUSION

All the models generated for all the properties were significant at 1% level. Values for coefficient of determination (R²) and coefficient of variation (CV) of all the models were higher than 0.60 and below 10%, respectively, which are good indicators of an adequate model. In all the models, lack of fit was not significant, except for final moisture content, thus second-order polynomial (quadratic model) was sufficient for particle size, bulk density, drying rate and powder recovery. A polynomial of higher degree could be employed for final moisture content model since its lack of fit was significant. However, considering its R², which is greater than 0.60 (0.88), its CV, which is below 10% (3.7%), and model significance, the model developed for final moisture content was considered acceptable.

Response surface plots revealed that decreasing the pump speed and increasing inlet air temperature increased the dry matter of the resulting powder. Particle size increased with an increase in inlet air temperature, pump speed and feed temperature. Pump speed had the greatest influence on drying rate at 1% level. Powder recovery increased with an increase in inlet air temperature and feed temperature, and a decrease in pump speed. The optimum spray drying parameters found were 180 ºC for inlet air temperature, 12 × 10³ rpm for pump speed, 43 ºC for feed temperature and 1–2 bars air pressure.

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