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**INSTANT MILK POWDER PRODUCTION:
DETERMINING THE EXTENT OF AGGLOMERATION**

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Anna M Williams

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ABSTRACT

Agglomerated milk powders are produced to give improved properties such as flowability, dispersibility, reduced dustiness and decreased bulk density. A key function of these powders is to dissolve “instantly” upon addition to water and because of this they are also called “instant milk powders”. They are produced by agglomerating the undersized fines that are returned to the top of the spray drier with milk concentrate droplet spray. Interaction occurs in a collision zone, often with multiple sprays and fines return lines. Agglomeration can be a difficult process to control and operators find it hard to fine tune the process to produce specific powder properties. This work aimed to understand the effects of key droplet and fines properties on the extent of agglomeration to allow a mechanistic understanding of the process.

Three scales of spray drier were investigated in this study with different rates of evaporation; a small scale drier ($0.5 - 7 \text{ kg water h}^{-1}$), a pilot scale drier ($80 \text{ kg water h}^{-1}$) and a range of commercial production scale driers ($4 - 15\,000 \text{ kg water h}^{-1}$). A survey of operators of commercial scale driers showed that control of instant milk powder production to influence bulk density is highly intuitive. Fines recycle rates were expected to be important in control of agglomeration processes and were estimated on a specific plant by using the pressure drop measured in the fines return line. A model based on pressure drop along a pneumatic pipeline under-predicted the experimental values for pressure drop due to solids, which means a calibration curve should be generated for each specific drier. Fines recycle rates were predicted to be significantly higher at 95 to 130 % of production rates compared to those expected by operators of 50%.

Experimental measurements agreed with existing models for the effect of temperature on the density and viscosity of milk concentrates. Experimental results showed that the surface tensions of concentrated milks were within the same range as literature values for standard milks below 60°C , but were significantly higher for milk above 60°C . This is thought to be linked to the mechanism of skin formation due to disulphide cross linking at high temperatures and concentrations. Powder properties were also established for selected products produced on the commercial scale driers. These powders were then used in experiments on the two smaller driers. Because collision frequency depends on the velocity and droplet size of sprays; these properties were measured for the small scale drier and estimated, where possible, for the pilot and commercial driers.

The small scale agglomerating spray drier was configured to alter droplet and particle properties when interacting a vertical fines particle curtain with a horizontal spray sheet. An extensive design and improvement process was carried out to ensure the system consistently delivered these streams in a controllable manner. The processes of collision and adhesion occur very quickly inside the spray drier. In order to assess the extent of agglomeration that has occurred, the feed streams must be compared to the final product stream. An ideal way to do this is to use an agglomeration index which

compares the particle size distributions of the feed (fines recycle and spray streams) and the particle size distribution of the product stream (the agglomerated powder). The index described changes between these streams across the particle size distribution and is called an agglomeration efficiency, ζ_g . However, it was found that the presence of fines in the product of the one-pass design obscured the agglomerates formed. The agglomeration efficiency, ζ_g , was modified to become ζ_h which subtracted the fines stream from the agglomerated product distribution. In this way ζ_h models industrial operation where the fines are recycled, by effectively just comparing the spray and product streams entering and leaving the process.

The small scale drier was used for an experimental study on natural and forced agglomeration, where the drier was operated with spray only, then with spray and fines. For natural agglomeration, SEM images of the product powder indicated that little agglomeration occurred between spray droplets. The product yield was unacceptably low (~ 40%) due to adhesion of spray droplets to the drying chamber wall opposing the horizontal spray. When the fines curtain was introduced in the forced agglomeration experiments, product yield increased above 50% because the fines acted as collectors for the spray droplets. However, the agglomeration performance of the modified spray drier was lower than expected. The equipment design was then optimised by considering three key issues; fines dispersion, droplet dispersion and stickiness, and agglomerate breakdown. Final experiments studied agglomeration at low fines to spray mass flux ratios and showed that increasing the fines size had a positive effect on agglomeration efficiency, ζ_h .

The agglomeration study at pilot scale identified the effect of key variables, total solids, concentrate and fines flow rate, and fines size on the agglomeration efficiency. A dimensionless flux approach was used to explain the experimental results. The fines to spray mass flux ratio and the projected area flux ratio (at constant concentrate flow rate) were found to be the most suitable to represent the physical processes during agglomeration. Experimental results showed that a higher dimensionless flux resulted in more agglomeration and as well as small fines size and atomising low solids concentrate. The critical Stokes number highlighted the importance of particle size and collision velocity on the outcome of the collision as well as the importance of stickiness on adherence following the collision. A statistical analysis established a relational model for predicting the agglomeration efficiency based on fines size, total solids and the fines to spray mass flux ratio.

This thesis has gained insight into agglomeration processes during spray drying and knowledge about how to define the extent of agglomeration. Practical findings from this research can have a significant impact on successful spray drying operation for instant powders. There are some practical steps to be taken industrially to promote the control of agglomerating spray driers. The first step is to measure and control the flow of fines recycled to the top of the spray drier. The next step is to validate the findings at industrial scale and link the agglomeration index to the bulk powder properties. However, there are many challenges that remain to be tackled in the area of milk powder agglomeration. Milk powder agglomeration at the top of the spray drier is a complex process involving many different variables. A more detailed study of the micro processes that occur during agglomeration will give increased understanding of the relationships between key operating variables and agglomerate properties.

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“Particles can’t read” so just writing the word agglomeration on a piece of equipment will not necessarily ensure this is the process that will happen inside. This quote certainly sums up my thesis, which would have told quite a different story if only particles could read.

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NOMENCLATURE

| | | |
|------------------------|--|----------------------------------|
| \dot{A} | area flux of powder traversing the spray zone | $\text{m}^2 \text{s}^{-1}$ |
| A | area | m^2 |
| A_t | total cross sectional nozzle area | m^2 |
| A_p | particle surface area | m^2 |
| A_{spray} | spray on foot print area of the nozzle onto the powder bed | m^2 |
| Ca | Capillary number | — |
| C_D | drag coefficient of particle | — |
| C_D' | modified drag coefficient | — |
| C_p | specific heat capacity at constant pressure | $\text{J kg}^{-1} \text{K}^{-1}$ |
| D | diameter | m |
| D_0 | initial droplet diameter | m |
| D_{12} | sum of colliding particle radii | m |
| $D_{3,2}$ | Sauter mean diameter | m |
| $D_{4,3}$ | volume weighted mean diameter | m |
| D_{BET} | BET diameter | m |
| D_{bridge} | diameter of sinter bridge | m |
| D_f | diffusion coefficient | $\text{W m}^{-1} \text{K}^{-1}$ |
| D_{max} | maximum diameter of droplet | m |
| D_{OA} | degree of agglomeration | — |
| $D_{\text{particles}}$ | volume mean particle diameter of the particles | m |
| D_{primary} | diameter of initial particles | m |
| D_v | diffusion coefficient of water vapour in air | $\text{W m}^{-1} \text{K}^{-1}$ |
| D_w | diffusion coefficient of water vapour through the droplet | $\text{W m}^{-1} \text{K}^{-1}$ |
| e | coefficient of restitution | — |
| E_{in} | heat in inlet air | kW |
| E_{loss} | heat loss | kW |
| E_{out} | heat in outlet air | kW |
| f | frequency | — |
| F | finer flow rate | kg s^{-1} |
| FN | flux number | — |
| f_n | number of fines particles | — |
| F_N | total number of fines particles | — |
| f_p | solids-pipe friction factor | — |
| F_{PA} | projected area of fines | m^2 |
| F_{pw} | solids-pipe friction force | Pa m^{-1} |
| F_t | impact force | Pa m^{-1} |
| g | acceleration due to gravity | m s^{-2} |
| h | convective heat transfer coefficient | $\text{W m}^{-2} \text{K}^{-1}$ |
| h_a | height of granule surface asperities | m |
| H_{amb} | enthalpy of ambient air | kJ kg^{-1} |
| H_{fus} | latent heat of fusion | J kg^{-1} |
| H_{in} | enthalpy of inlet air | kJ kg^{-1} |

| | | |
|------------------|--|---------------------------------|
| h_m | convective mass transfer coefficient | $\text{W m}^{-2} \text{K}^{-1}$ |
| H_{out} | enthalpy of outlet air | kJ kg^{-1} |
| k | constant or ratio of specific heats | – |
| K | consistency coefficient | N s m^{-2} |
| KD | constant \times particle diameter | M |
| KE_1 | kinetic energy before impact | J |
| KE_2 | kinetic energy after impact (droplet is at maximum | J |
| k_l | thermal conductivity of liquid | $\text{W m}^{-1} \text{K}^{-1}$ |
| L | length | m |
| L_e | equivalent length | m |
| l_v | friction loss | $\text{m}^2 \text{s}^{-2}$ |
| l_w | wetted length of Wilhelmy plate | m |
| m | mass loading = ratio of M_p to M_g | – |
| M_A | mass rate of air | kg s^{-1} |
| M_{atm} | mass flow rate of air through the atomiser | kg s^{-1} |
| M_c | mass flow rate of concentrate | kg s^{-1} |
| M_{chute} | mass flow rate of air through the chute | kg s^{-1} |
| M_f | mass flow rate of fines | kg s^{-1} |
| M_g | mass flow rate of gas | kg s^{-1} |
| M_{in} | mass flow rate of inlet air | kg s^{-1} |
| M_{leak} | mass flow rate of air in due to leakage | kg s^{-1} |
| m_{obs} | mass of powder added over observed time | kg |
| M_{obs} | mass flow rate of fines (observed) | kg s^{-1} |
| M_{out} | mass flow rate of outlet air | kg s^{-1} |
| M_p | mass flow rate of particles | kg s^{-1} |
| M_{prod} | mass flow rate of powder production | kg s^{-1} |
| M_s | mass flow rate of spray | kg s^{-1} |
| m_t | total mass added | kg |
| n | power law index | – |
| \hat{N} | number concentration of particles | m^{-3} |
| \dot{N} | number flow rate | s^{-1} |
| N | number of particles per kilogram | kg^{-1} |
| Nu | Nusselt number | – |
| Oh | Ohnesorge number | – |
| p | mass proportion in the powder | – |
| P_1 | inlet nozzle pressure | Pa |
| P_2 | exit nozzle pressure | |
| Pr | Prandlt number | – |
| P_w | Wilhelmy force | N |
| ΔP | pressure drop | Pa |
| ΔP_{fg} | pressure drop due to friction of gas | Pa |
| ΔP_{fs} | pressure drop due to solids friction | Pa |
| ΔP_g | pressure drop due to gas | Pa |
| Δp_{hu8} | pressure drop due to hold up of gas | Pa |

| | | |
|------------------|---|----------------------------|
| ΔP_{hus} | pressure drop due to hold up of solids | Pa |
| ΔP_s | pressure drop due to solids | Pa |
| Q_a | volumetric air rate | $\text{m}^3 \text{s}^{-1}$ |
| $Q_{a.c}$ | volumetric flow rate of curtain air | $\text{m}^3 \text{s}^{-1}$ |
| Q_{atm} | volumetric flow rate of atomising air | $\text{m}^3 \text{s}^{-1}$ |
| q_b | binder spray rate | kg s^{-1} |
| Q_l | liquid flow rate | kg s^{-2} |
| r | particle or granule radius | μm |
| Re | Reynolds number | – |
| RH | relative humidity | kg kg^{-1} |
| S | spray flow rate | kg s^{-1} |
| Sc | Schmidt number | – |
| SE_1 | the surface energy before impact | J |
| SE_2 | surface energy after impact | J |
| S_g | geometric standard deviation | – |
| Sh | Sherwood number | – |
| s_n | number of spray droplets | – |
| S_N | total number of spray droplets | – |
| S_{PA} | projected area of spray | m^2 |
| SSA | specific surface area | $\text{m}^2 \text{g}^{-1}$ |
| St^* | critical Stokes' number | – |
| St_e | Stefan number | – |
| St_v | viscous Stokes' number | – |
| T | temperature | $^{\circ}\text{C}$ |
| T_{amb} | ambient temperature | $^{\circ}\text{C}$ |
| T_j | air temperature under sonic conditions | $^{\circ}\text{C}$ |
| t | time | s |
| t_{exp} | exposure time | s |
| T_g | glass transition temperature | $^{\circ}\text{C}$ |
| T_m | droplet melting temperature | $^{\circ}\text{C}$ |
| TS | total solids concentration | – |
| $T_{w.l}$ | initial substrate temperature | $^{\circ}\text{C}$ |
| U | velocity | m s^{-1} |
| u_0 | initial relative granule collision velocity | cm s^{-1} |
| U_0 | droplet impact velocity | m s^{-1} |
| U_A | velocity of air | m s^{-1} |
| U_{A1} | predicted air exit velocity from the nozzle | m s^{-1} |
| U_{A2} | predicted velocity 50mm from nozzle of air and spray | m s^{-1} |
| $U_{air\ only}$ | measured air velocity 50 mm from nozzle without spray | m s^{-1} |
| U_c | collision velocity | m s^{-1} |
| UD | constant, D and overall heat transfer coefficient U | W K^{-1} |
| U_d | droplet velocity | m s^{-1} |
| u_e | excess gas velocity | m s^{-1} |
| U_g | interstitial gas velocity | m s^{-1} |

| | | |
|-------------------|--|-------------------------------|
| U_s | velocity of particles | m s^{-1} |
| U_T | terminal velocity | m s^{-1} |
| u_z | velocity of entrained air | m s^{-1} |
| $u_{z,max\infty}$ | centre velocity of the air in the equilibrium state | m s^{-1} |
| \dot{V} | volumetric spray rate of the binder liquid | $\text{m}^3 \text{s}^{-1}$ |
| v | specific volume of mixture | $\text{m}^3 \text{kg}^{-1}$ |
| V_b^* | dimensionless bridge volume | – |
| V_b | pendular bridge volume | m^3 |
| V_d | droplet volume | m^3 |
| v_f | specific volume of fat | $\text{m}^3 \text{kg}^{-1}$ |
| V_g | volume of gas | m^3 |
| v_i | specific volume of component i | $\text{m}^3 \text{kg}^{-1}$ |
| v_{nw} | specific volume of native whey protein | $\text{m}^3 \text{kg}^{-1}$ |
| v_{rel} | relative velocity between liquid and air | m s^{-1} |
| V_s | volume of solids | m^3 |
| v_s | velocity of a single particle | m s^{-1} |
| $v_{s\infty}$ | terminal velocity of a single particle | m s^{-1} |
| v_z | velocity of the particle in the powder jet | m s^{-1} |
| $v_{z,max\infty}$ | centre velocity of the particle in the equilibrium state | m s^{-1} |
| w | width | m |
| W^* | dimensionless rupture energy | – |
| W | pendular bridge rupture energy | J |
| We | Weber number | – |
| W_{vis} | work done in deforming a droplet against viscosity | J |
| x | curtain width | m |
| X_c | dry matter content of concentrate | kg kg^{-1} |
| X_{casein} | mass concentration of casein | kg L^{-1} |
| X_{dw} | mass concentration of denatured whey protein | kg L^{-1} |
| X_{eq} | equilibrium moisture content | kg kg^{-1} |
| X_{fat} | percentage of fat | – |
| x_i | percentage of component i in the mixture | – |
| x_l | dry matter fraction of lactose | kg kg^{-1} |
| X_{milk} | dry matter content of standard milk | kg kg^{-1} |
| X_{nw} | mass concentration of native whey protein | kg L^{-1} |
| y | curtain length | m |
| Y_g | dynamic yield stress | Pa |
| Z_{12} | collision rate between two particles | $\text{m}^{-3} \text{s}^{-1}$ |

Greek Letters

| | | |
|-------------|---|---------------|
| μ | viscosity | Pa s |
| μ_g | viscosity at the glass transition temperature | Pa s |
| μ_l | viscosity of lactose solution | Pa s |
| μ_{ref} | viscosity of the serum | Pa s |
| α | shape factor | – |

| | | |
|---------------|---|--------------|
| γ | rate of strain | s^{-1} |
| ε | voidage | — |
| θ | contact angle | $^{\circ}$ |
| θ_a | advancing contact angle | $^{\circ}$ |
| ζ_g | agglomeration efficiency using g distribution | — |
| ζ_h | agglomeration efficiency using h distribution | — |
| ρ | density | $kg\ m^{-3}$ |
| σ | surface tension | $N\ m^{-1}$ |
| σ_l | surface tension of saturated lactose | $N\ m^{-1}$ |
| τ | shear stress | Pa |
| Φ | volume fraction of concentrate | — |
| Φ_{max} | the maximum volume fraction | — |
| Φ_{milk} | volume fraction of milk | — |
| ψ | dimensionless time | — |
| Ψ_a | dimensionless spray flux | — |
| δ | thickness of the liquid layer | m |

Subscripts

| | |
|---------|-----------------------|
| 0 | before |
| 1 | after |
| a | air |
| agg | agglomerates |
| c | concentrate |
| d | droplet |
| AE | entrained air |
| f | finer |
| g | gas |
| p | particle or product |
| s | spray, dried droplets |
| $skim$ | skim milk |
| $whole$ | whole milk |

Acronyms

| | |
|--------|------------------------------|
| AF | agglomeration factor |
| AP | agglomeration parameter |
| BET | Brunauer, Emmett, & Teller |
| DoA | degree of agglomeration |
| GAB | Guggenheim-Anderson-de Boer |
| $ISMP$ | instant skim milk powder |
| $IWMP$ | instant whole milk powder |
| PSD | particle size distribution |
| SEM | scanning electron microscopy |
| SMC | skim milk concentrate |
| SSA | specific surface area |
| SSM | standard skim milk |
| SWM | standard whole milk |
| WMC | whole milk concentrate |