International Dairy Journal 97 (2019) 65–70

Contents lists available at ScienceDirect

International Dairy Journal

journal homepage: www.elsevier.com/locate/idairyj

Slowly dissolving particles in instant whole milk powder – Characterisation and quantitative analysis

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ARTICLE INFO

Article history:
Received 24 January 2019
Received in revised form
28 May 2019
Accepted 28 May 2019
Available online 8 June 2019

ABSTRACT

Instant milk powders are graded after manufacture using functional tests that gauge the performance of the powder in a way that is relevant to consumer usage, but limited with respect to optimisation of the manufacturing plant. One such test is the slowly dissolving particle test that assesses undissolved particulate material adhering to a glass surface after reconstitution. This particulate material can be comprised of two subclasses – bulk particles and surface particles – which have different origins, natures and compositions and require different approaches for their measurement. However, a combination of an image analysis technique developed to quantify bulk particles and a gravimetric approach to assess the surface particle mass, enables more objective powder grading and better understanding of the source of solubility issues that may arise. Plant operators can use the result to make more specific adjustments to optimise plant performance.

1. Introduction

The apparently simple process of reconstituting milk powder belies a complex series of steps involving wetting, sinking, dispersion and, finally, dissolution of the powder. The result is a homogeneous stable colloidal suspension that resembles pasteurised homogenised fresh milk. Ideally, this process occurs rapidly and completely and is one of the key functional expectations of consumer milk powders. In practice, however, when instant whole milk powder (IWMP) is reconstituted with water, there is almost always some undissolved or undispersed residue (Písecký, 2012).

Instant milk powders are assessed using a range of functional tests that usually attempt to mimic domestic reconstitution and gauge the performance of the powder at each of the steps in the dissolution process. Consequently, many of the tests are based on visual assessment and grading against, for example, a series of photographic standards. The slowly dissolving particle (SDP) test is one such consumer-based test that is used routinely in the dairy industry to grade IWMP.

It should be noted that the term 'slowly dissolving particle' may in some instances be ambiguous as some of the material contributing to the grade assigned may actually be insoluble. Furthermore, the terms dispersible and dissolving are also sometimes used interchangeably, despite referring to different steps in the reconstitution process. However, a 'dispersed' agglomerate that has broken down to its primary particles may, in some cases, still be visible to a consumer; hence we have chosen to define SDP as 'slowly dissolving particles'.

The SDP test assesses the amount of undissolved material adhering to the walls of a test tube after a standard reconstitution and draining process and a short standing time, much as a consumer might observe similar material adhering to a glass as the reconstituted product is consumed. The assessment is made by comparing the tube with a series of five photographs (Fig. 1). As it is generally accepted that the limit of resolution of the human eye is about 55–75 μm, the SDP test, by this definition, is a measure of particulate residue above this size that adheres to the glass surface of a test tube and remains visible at the point of assessment.

Inevitably, there will be situations where the combination of slowly dissolving particle size and opacity, along with other factors such as fat coating or smearing, will make assigning a grade a very subjective process. Eyesight and environmental conditions such as lighting can also influence the assigned grade. In addition, producing a photographic standard that represents all the possible combinations of surface coating that might be noticed by an analyst is a difficult task. Thus, in practice, it is often difficult to standardise

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https://doi.org/10.1016/j.idairyj.2019.05.015
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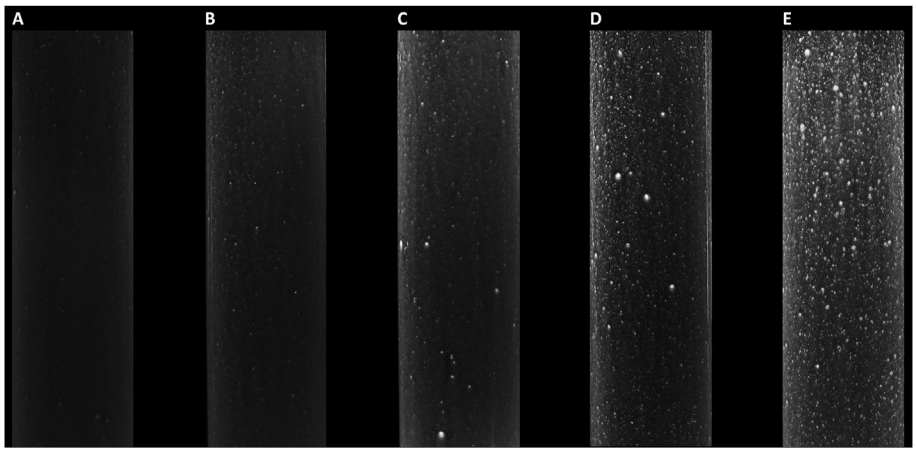

Fig. 1. SDP photographic standard. A reconstituted sample is poured into a test tube, the tube is inverted and after a standardised time, the material adhering to the tube is compared with this photographic standard.

grading between different analysts or laboratories. Furthermore, because the SDP standard is comprised of only five discrete photographs, the grading data generated are insensitive to small changes in processing conditions. Therefore, the test is of limited value in helping to optimise the operation of a manufacturing plant.

McKenna, Lloyd, Munro, and Singh (1999) suggested that the structure of slowly dispersible particles is analogous to that of powder particles and that either agglomerate structure or surface composition is responsible for their slow dispersion. White flecks are another type of particle known to cause a visual defect. White flecks are typically a few hundred micrometres in diameter and either remain suspended in a thin layer on top of the reconstituted milk or adhere to the walls of the reconstitution vessel. These particles are insoluble and are therefore likely to have a composition and structure different from that of powder particles (Toikkanen, Outinen, Malafronte, & Rojas, 2018). Meyer, Rajendram, and Povey (2006) used the terms surface particles and bulk particles to describe and differentiate the types of particle seen after reconstitution. Bulk particles are dispersed throughout the bulk of the fluid, whereas surface particles are found on the surface of the milk and the walls of the container and are formed by clustered fat aggregates. In this work, it is recognised that both particle types can adhere to the walls of the test tube during the SDP test and contribute to the observed defect. Consequently, there are at least two possible causes of SDP, which are probably driven by different mechanisms. Therefore, in addition to addressing the subjectivity issues of the SDP test noted earlier, there is also a need to develop methods that can measure the surface and bulk particles separately.

Techniques are available to measure the breakdown of particles during their dissolution. Usually, these measurements are made on a recirculated sample at very low solids concentrations, for example using laser diffraction particle size analysers. However, to our knowledge, there are no instrumental techniques available that incorporate a standardised, mechanised consumer-based mixing approach and the analysis of the solubility of the reconstituted milk bulk particles at a realistic total solids level and at a specific, known time after reconstitution. Thus, we believe a new approach is required.

Attempts to provide a more quantitative measure of surface particles have included the development of an ultrasound spectroscopy technique (Meyer et al., 2006) and the use of an ultrasonic flaw detector (Hauser & Amamcharla, 2016). Although providing a quantitative measure, ultrasonic techniques require more specialised equipment and the cost may inhibit use in powder grading laboratories. More recently, a defined volume of surface material has been pipetted off the sample surface and transferred to a black plate to count the particles (Toikkanen et al., 2018). The pipetting method is quantitative and simple, but relies on consistent transfer of the surface material by analysts. Consistency of the transfer may be influenced by factors such as where and how deep the pipette is placed in the sample and the amount of surface material present. Finally, the white flecks number (ISO/IDF, 2009) was developed to indicate the amount of white flecks in a sample. The test is based on the clogging of a 63 μm mesh filter by white fleck material. However, the method cannot distinguish between white flecks and SDP, or between samples with a low number of white flecks (Toikkanen et al., 2018).

In a simpler approach, Litman and Ashworth (1957) used a separatory funnel to isolate and characterise "surface scum" particles. Milk powder was reconstituted in the funnel and then slowly drained out, leaving scum on the walls of the funnel. The scum material was redispersed with water and then allowed to rise to the surface. The water was again slowly drained out. The remaining scum residue was then rinsed out of the funnel and collected for analysis. This simple approach involving minimal equipment presented an opportunity for further development into a gravimetric technique for the measurement of surface particles in the current work.

This study presents the development and validation of methods that both differentiate between and measure surface and bulk particles. Firstly, the different types of particle are isolated and characterised. Quantitative methods developed for the analysis of the different particle types are then described. Finally, the ability of a combination of the two methods to predict the SDP test result and to provide a more complete picture of powder solubility is discussed.

2. Materials and methods

2.1. Materials

IWMP samples were sourced from various dairy plants in New Zealand. Reverse osmosis water was used for reconstitution and analysis unless otherwise stated.

2.2. Isolation and characterisation of particles

2.2.1. Isolation of bulk particles

IWMP (12.5 g) was reconstituted in 100 mL of water at 25 °C using a spoon. Approximately 5 min after reconstitution, a

representative sample of bulk particles were isolated by dipping a microscope slide into the mixed solution, removing it and wiping clean the back of the slide prior to examination.

2.2.2. Isolation of surface particles

IWMP (22 g) was reconstituted in 150 mL of water at 40 °C using a 300 mL mixing tube and a rotating mixer (Mixer for the Determination of Miscibility and Clumping; Gerber Instruments AG, Effretikon, Switzerland). The sample was mixed at a rate of one revolution per second for a total of 15 s, after which the reconstituted milk was poured through an 850 μm sieve into a 500 mL separatory funnel. After 5 min, the separatory funnel was drained, leaving 4 mL of milk in the neck of the funnel. Water (150 mL; 20–25 °C) was added and similarly drained after a further 2 min. The material coating the walls and in the neck of the separatory funnel was isolated by filtration through a 5 μm-aperture polycarbonate membrane filter (Isopore TMTP04700, Merck, Dublin, Ireland). The material was scraped off the filter and used for compositional analysis.

2.2.3. Light and confocal microscopy

The bulk particles present on the microscope slide after isolation were examined under light microscopy at 10X magnification (BX60; Olympus, Tokyo, Japan). The same sample was then stained with a 15 μL premix of 0.5% Nile Red and 0.2% Fast Green to highlight the fat and protein components, and was analysed by confocal microscopy (LSM 510; Zeiss, Munich, Germany).

An aliquot (approximately 5 mL) of the sample reconstituted for surface particle analysis was also stained with a 15 μL premix of 0.5% Nile Red and 0.2% Fast Green and the milk surface analysed by confocal microscopy (LSM 800; Zeiss).

2.2.4. Compositional analysis

The proportion of fat in the isolated surface particle material was estimated gravimetrically. The sample was mixed with 10 mL of hexane in a test tube. The tube was centrifuged (200×g, 5 min) and the upper hexane layer was transferred to another tube containing sodium sulphate, mixed again and centrifuged (200×g, 5 min). The hexane layer was transferred to another container and evaporated, and the mass of residue, assumed to be fat, was calculated. The sample was then methylated and the fatty acid composition was determined by gas chromatography (IDF, 2001, 2002a,b).

The fatty acid composition of the milk powder from which the surface particles were isolated was also determined. Fat was extracted from the milk powder and methylated, and the fatty acid composition was determined by gas chromatography (IDF, 2001, 2002a,b).

2.3. Development of a quantitative method for bulk particle analysis

A method using image analysis to determine the amount of undispersed milk powder particles present in the bulk solution after reconstitution was devised.

2.3.1. Principle of operation

Samples were reconstituted to 4% solids by means of a proprietary mechanical mixer (Fig. 2a), which was designed to impart a similar level of shear as experienced in a typical domestic reconstitution process. The typical domestic reconstitution process was determined via an in-market study in which consumers were asked to reconstitute a range of milk powders. The mechanical mixing sequence was then adjusted to obtain a similar level of residue remaining after reconstitution to that obtained by consumers in market.

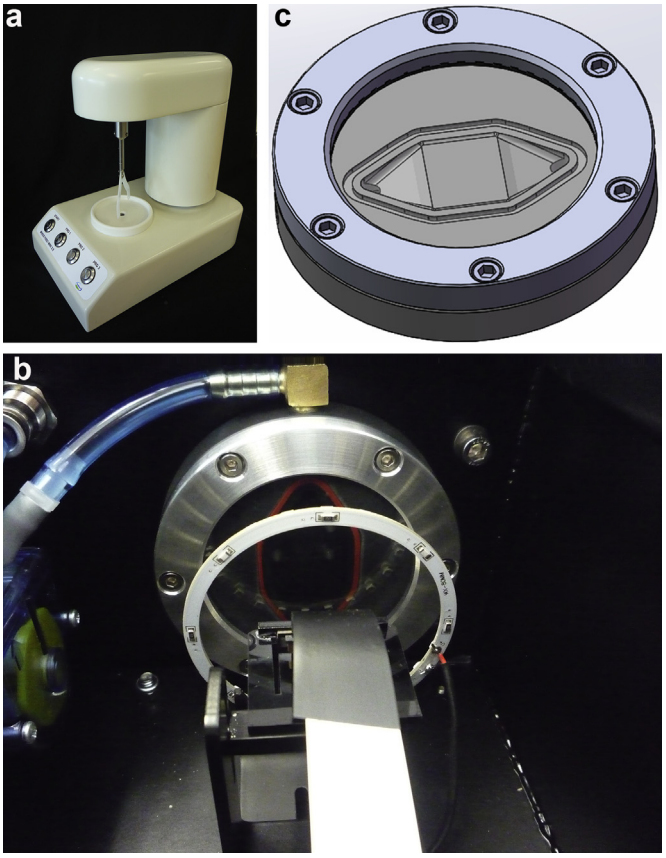

Fig. 2. Low-shear mixer designed to mimic domestic reconstitution (a) and flow cell design (b,c): external diameter 100 mm, measurement zone path length 0.4 mm.

The mixing paddle of the mechanical mixer revolved 13 times clockwise followed by 13 times anticlockwise over a total period of 8 s. The sample was left to stand for 2 min and was then gently mixed via one paddle revolution to resuspend any settled particulate material. The milk was then passed through a laminar flow optical cell (Fig. 2b, c) using a small downstream peristaltic pump that withdrew the reconstituted milk from the lower half of the sample, and thereby limited the analysis to 'bulk particles,' by definition. The optical cell had a shallow (0.4 mm) path length, enabling the visualisation and analysis of undissolved particles against a black background using a camera and image analysis software.

2.3.2. Image analysis procedure

A Raspberry Pi camera and computer (Raspberry Pi Foundation, Cambridge, UK) was used to capture a greyscale image using dark field illumination to give good contrast for the particles. The resolution was approximately 10 μm per pixel. The image was cropped to select the central region (15 mm × 15 mm) and the background was then normalised to reduce any brightness gradient across the flow cell. This was achieved by estimating the background through a sequence of filters: a minimum filter with a 5 pixel radius to remove small bright points (particles smaller than a 5 pixel radius); a mean filter with a 22 pixel radius to average the background and remove noise (large enough to blur details and to estimate the local background pixel value); a maximum filter with a 5 pixel radius (to restore the positions of any blurred edges or gradients shifted by the first minimum filter). The background image was subtracted

from the original image. The normalised image was then converted to a binary image by comparing each pixel with a global intensity threshold value, which could be adjusted manually in the graphical user interface. An example of original and processed images is shown in Fig. 3.

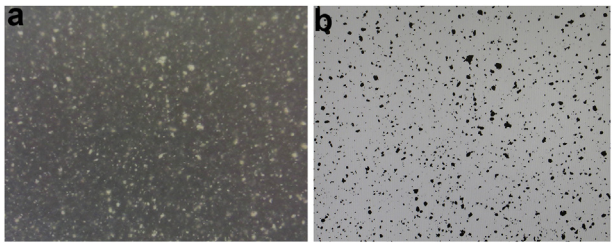

Fig. 3. Conversion of grey-scale image (a) to binary image (b).

Connected components analysis was then used to identify each individual particle. Particles with an area smaller than a pre-set threshold were eliminated. The area threshold was set at 40 pixels (approximately 70 μm diameter), corresponding approximately to the limit of resolution of the human eye. The area distribution of the remaining particles was accumulated. The output of the approach was the number of particles above 40 pixels and their mean size. To increase the confidence in the result, three independent sequential analyses were performed on the sample, with a pumping step between each to refresh the contents of the flow cell.

2.4. Development of a quantitative method for surface particle analysis

The method outlined for the isolation of surface particles was extended for use as a quantitative method for surface particle analysis. The mass of material retained on the 5 μm-aperture membrane filter was measured, after 60 min of desiccation, to obtain a measure of the level of surface particles in a sample.

Studies of linearity, robustness, repeatability and reproducibility were carried out to confirm the ability of the method to reliably measure the surface particles.

2.5. Measurement of surface and bulk particles and comparison with SDP

Sixty-four IWMP samples were analysed using the two new methods. The samples had been previously graded using the SDP test at 25 °C using one analyst in one laboratory to minimise variability. The mass of surface particles and the number of bulk particles were then compared with the results from the SDP test. A model for predicting SDP at 25 °C was then developed using the combined results from both new methods along with the original SDP score.

3. Results and discussion

3.1. Isolation and characterisation of particles

Light microscopy (Fig. 4a) showed that the bulk particles comprised many smaller subunits typical of the structure of agglomerated milk powder. Confocal microscopy showed that these particles were composed of protein and fat globules with a proportion and distribution of components typical of milk powder particles (Fig. 4b). Thus, in agreement with McKenna et al. (1999), who suggested SDP are analogous to intact powder particles, the bulk particles isolated in this study are probably slowly dissolving powder agglomerates.

Confocal microscopy of the surface of reconstituted milk (Fig. 4c) showed clumps of unstructured, non-membrane-bound fat amongst fat globules and background protein. The observation of unstructured fat contrasts with numerous findings that clusters, flocculates or aggregates of oil globules are responsible for most white flecks or surface particles (Drapala, Mulvihill, & O'Mahony, 2018; McKenna et al., 1999; Meyer et al., 2006; Toikkanen et al., 2018). For example, Drapala et al. (2018) demonstrated by confocal microscopy that white flecks are made up of a porous protein network densely packed with individual oil globules. Suggestions are that the flocculation is protein-mediated and/or heat-induced (Drapala et al., 2018; Toikkanen et al., 2018). In support of the presence of protein in surface particles, Meyer et al. (2006) reported a chemical composition containing 87% fat, 7% protein and 2% lactose. Toikkanen et al. (2018) reported a composition of 75% fat and 22% protein in white flecks isolated from reconstituted infant milk formula. Meanwhile, we measured a fat content of 98–100% for the surface particles. Differences in isolation procedures may be partly responsible for the higher fat content, but the difference may also indicate an alternative dominant mechanism of surface particle formation.

Lactose crystallisation in powders during storage can disrupt oil globules, leading to fat leakage and the formation of a different kind of surface particle (Drapala et al., 2018). Toikkanen et al. (2018) showed that smaller, sharper edged particles with shapes corresponding to lactose crystals were formed in this case. It is difficult to resolve whether such particles are present in the confocal image of the surface of the reconstituted milk (Fig. 4c). However, our observation of unstructured fat better aligns with this mechanism. Further work will be carried out on a greater variety of samples, imaging the isolated particles in addition to the reconstituted surface and capturing higher resolution images to better assess the structure of the fat present. Along with the quantitative data from the new isolation method, this may better help to determine the source of surface particles and make better adjustments to plant operating conditions to reduce their occurrence.

The isolated material in the current work also contained elevated levels of palmitic acid (C16:0). The increase in the palmitic acid fraction compared with the original milk powder showed that fractionation of fat had occurred. A decrease in the lower melting point fraction was also observed, probably because of melting and removal of this fraction during the isolation of surface particles at 40 °C. The presence of higher melting point fat fractions in surface material was also observed by Litman and Ashworth (1957) and Regost (2016).

3.2. Quantitative analysis of bulk particles by image analysis

The solubility image analyser measures the number of particles suspended after reconstitution and a predetermined standing time. The number of particles measured is affected by the reconstitution conditions, including the temperature, the time of standing after reconstitution and the total solids. The last property changes the contrast. As the solids level is increased, the number of particles increases but, concomitantly, the background whiteness increases, so that fainter particles fall below the intensity threshold and are no longer counted. Similarly, aspects of the analyser configuration, including the pump speed (liquid flow rate) through the analyser and the lighting intensity, were standardised.

The reproducibility of the solubility image analyser method was determined by analysis of the same set of IWMP samples by two analysts. The samples covered a wide range of solubility performance. An R^2 of 0.98 for the results of one analyst plotted against

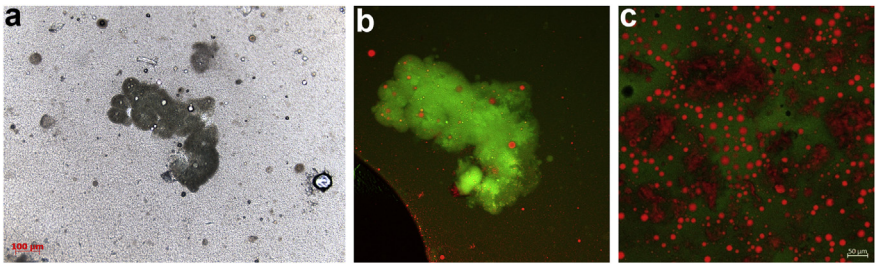

Fig. 4. Light micrograph (a) and confocal micrograph (b) of isolated bulk particles. Confocal micrograph of the surface of reconstituted milk (c). Protein stained green and fat stained red in confocal micrographs.

the results of the second analyst showed that the analyser provides a high level of reproducibility.

Correlation of the number of particles, as measured by image analysis, with the SDP score at 25 °C was found to be poor ($R^2 = 0.57$; data not shown). There are several likely reasons for the discrepancy between the two methods. Meyer et al. (2006) points out that visual reconstitution tests such as the SDP approach have poor repeatability and precision; likewise, our own SDP analysis has found a poor correlation (R^2 of 0.56) when comparing the results of two trained analysts using the same equipment. The SDP score also has a very small number of discrete classes compared with the much broader range of the analyser. Thus, a perfect data fit would not be expected. Finally, the solubility image analyser takes aliquots from the bulk of the sample and therefore measures only bulk particles and ignores surface particles. Therefore, it tests only a subset of the particles seen with the SDP test.

Potential improvements to the solubility image analyser method have been identified. These could include: (i) further automation of sample preparation and image analysis; (ii) an increased number of discrete measurements per sample to provide a more reliable average particle number; (iii) further analysis and interpretation of the particle size data, which may highlight an important role of the particle size data in grading and problem solving; (iv) using the solubility image analyser to also measure the rate of particle dissolution if measurements are made over a range of powder hydration times.

With respect to further analysis and interpretation of the particle size data, in some cases a high bulk particle number was determined for samples with low SDP scores. It may be that the particles counted were near the lower size threshold of the solubility image analyser. Although these particles would be present on the tube in the visual SDP test, they would be difficult to resolve by eye and therefore might be missed. Optimising the threshold settings of the image analyser may be necessary to produce more consumer-relevant results and will be investigated in future work on a wider range of samples. In regard to measuring the rate of particle dissolution over a range of powder hydration times, this would enable a picture of the dissolution properties of the bulk particles with time and provide an indication of what proportion of the initially detected particles remain insoluble after lengthy hydration.

3.3. Quantitative gravimetric analysis of surface particles

The gravimetric surface particle analysis isolates and measures the fat-based material floating on the surface of the milk after reconstitution.

A very good linear response ($R^2 = 0.99$; data not shown) between the mass of IWMP reconstituted and the level of surface particles recovered was found. The linear response was shown to be true for samples that were both low and high in surface particles.

The mass of isolated surface particles was significantly affected by the mixing procedure, the temperature of the water used to reconstitute the sample, the type of membrane filter, the length of the drying time and the interaction between the type of filter and the drying time. Therefore, these aspects of the test were standardised.

Repeatability and reproducibility of the method within our laboratory were determined based on the analysis of nine samples by three analysts (data not shown). The sample set covered the range of 2.8–48.6 mg of surface particles. The average repeatability was 2.8 mg and the average reproducibility was 3.2 mg, indicating that good precision can be achieved.

As observed for bulk particles, the correlation between the surface particle and SDP score was poor ($R^2 = 0.52$; data not shown). Again, this poor correlation was not surprising as the method does not consider the contribution from the bulk particles. Hence, combining the results obtained using these two quantitative methods may enable better prediction of the SDP score.

3.4. A combined surface particle and bulk particle approach to IWMP solubility evaluation

The SDP test provides a means of grading the solubility properties of IWMP in a way that is consumer relevant. However, it fails to deliver sufficient resolution or sensitivity for it to be used to optimise plant operation and it does not identify the source of any observed defect. As SDP can be composed of bulk particles and surface particles, it was considered likely that the overall consumer-relevant solubility performance of a powder was a combination of the measured results for both bulk and surface particles. A modelling approach was taken to determine the most appropriate way to combine the data. All models of numeric bulk particles versus surface particles, their first-order interactions and their squared terms were tested using exhaustive subset selection to find the best model to predict SDP. A three-variable model was the optimal model according to the subset selection search. The adjusted R^2 value of this model was 0.75, and all the predictor variables were highly significant ($p < 0.001$). The predicted versus actual plot is shown in Fig. 5. Although the model is unlikely to be used to predict SDP in our laboratories, generating the model enabled interesting insights to be obtained. This is because the SDP test still has merit in that it indicates the overall fitness-for-purpose from the consumer perspective and thus remains the 'benchmark'.

To improve the practical use of the combined approach in grading laboratories, a chart of the particle number as a function of the mass of surface particles was created (Fig. 6). Although setting the thresholds for failure is ongoing and may also be specific to the product being manufactured, the basis of the chart is that it can be divided into quadrants based on the threshold of failure for each component. For example, the upper right quadrant includes powders that fail in terms of both components, while the lower left

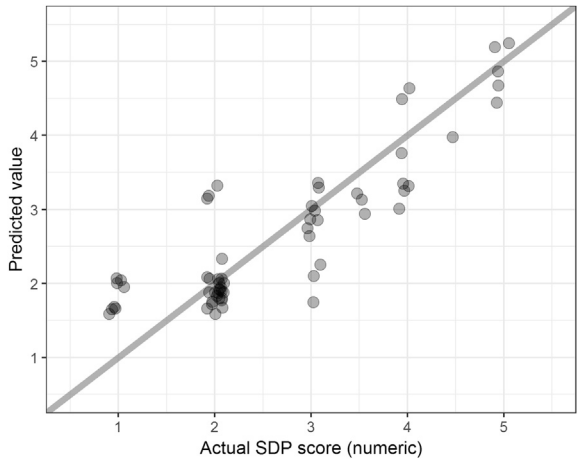

Fig. 5. Predicted versus actual SDP scores for the for three-variable model (numeric SDP ~ IACount + GravWS40 + IACount.sq); the actual SDP scores are jittered horizontally by up to 0.1 to separate them for more clarity.

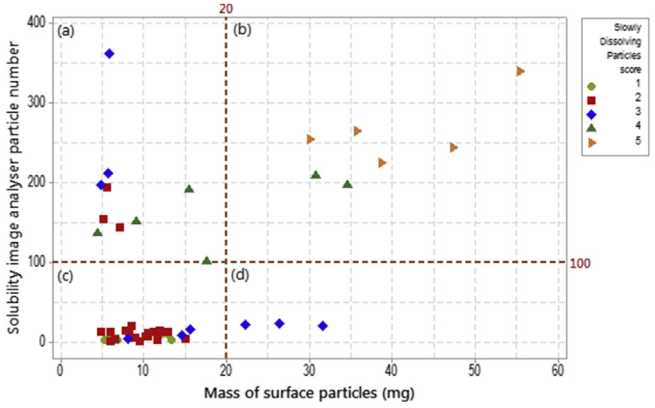

Fig. 6. Plot of image analysis result (particle number) versus surface defects results (mass of surface particles). SDP scores are: ●, 1; ■, 2; ◆, 3; ▲, 4; ▶, 5. Quadrants are: A, fails on particle number; B, fails on particle number, fails on surface defects; C, passes on particle number, passes on surface defects; D, fails on surface defects.

quadrant includes powders that pass in terms of both components. Thus, the chart provides a means not only to grade powders more objectively, but also to better understand the source of the failure. Plant operators can use the quantitative result to make more specific adjustments to optimise plant performance.

In fact, small changes in drying parameters such as inlet temperature and dryer throughput have been shown to affect the number of bulk particles measured by the image analysis technique. Thus, the increased sensitivity of the new method is allowing fine tuning of dryer operation where it was not previously possible. Similarly, the surface particle test has allowed trends to be observed with, for example, length of dryer run and dryer fouling, as well as chamber and ambient humidity (the details of which are commercially sensitive). The combined effect of these small incremental improvements, the fine tuning of dryer operation based on surface and bulk particle levels, is leading to noticeable improvement in the consumer-relevant SDP score in commercial plants.

4. Conclusions

Two subclasses of SDP were identified: bulk particles and surface particles. These subclasses had different origins, natures and compositions. Bulk particles largely comprised undissolved or slowly dissolving powder particles or agglomerates. Surface particles were composed largely of unstructured fat. Therefore, different approaches were required for their measurement.

An image analysis approach was devised to quantify the number of bulk particles suspended in reconstituted milk after defined standing times. A gravimetric approach was used to assess the mass of surface particles. A combination of these approaches provided a means not only to more objectively grade powders as suitable for consumer reconstitution, but also to better understand the source of any solubility issues that may arise. Plant operators can use the quantitative result to make more specific adjustments to optimise plant performance.

Acknowledgements

We acknowledge Michael Loh for performing the microscopic analyses, Richard Croy for creating the SDP prediction model, Maree Luckman for carrying out the statistical analysis and Angela Pike and Andrew Todd for analytical support during method development and validation. We also are very thankful to Jaques Janse van Vuuren for coding and prototype construction for the solubility image analyser method.

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