

# Rapid Particle Size Measurements Used as a Proxy to Control Instant Whole Milk Powder Dispersibility\*

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## Abstract

Dispersibility is a key quality variable for instant whole milk powder (IWMP) measured manually using a complex and laborious dissolution method. This is thus normally carried out post production at industrial dryers. However, this means there is no immediate feedback so the functional quality cannot be controlled in real-time. This work proposes the idea of applying a simpler, surrogate measurement that can be implemented in the plant in order to have useful real-time information regarding the quality of the product being produced. This we term is a proxy measurement. The functional property dispersibility was used as a case study, with particle size being investigated as a proxy at an industrial IWMP plant. It was found that particle sizing could be used to provide useful information regarding the powder, with the proxy measurement being able to predict in-specification powder 97% of the time. Although the test was not as effective for predicting out-of-specification results, with a false-positive rate of 50%, the fact that out-of-specification events are rare in the industry setting means that the overall proxy measurement is still between 78–87% accurate, and thus useful for predicting the dispersibility quality of the IWMP. Furthermore, these proxy measurements can then be combined with on-line plant information using multivariate techniques to further improve their accuracy

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and understand how the quality can be controlled by changing the plant processing conditions.

**Keywords:** quality, dairy, real-time, proxy measurement, functional properties

## 1 Introduction

Process analytical technology (PAT) conveys the concept of systems and tools used for achieving end product quality [1, 2]. Historically, much of the focus has been on spectroscopic measurement and the associated modelling of the results, without necessarily closing the feed-back loop to ensure this information reaches the process in real-time [1]. However, there are many challenging aspects that need to be addressed in order to be able to close the loop and control the quality during manufacture, when process adjustments can still be made. These key challenges are defining the term ‘quality’, being able to measure the relevant quality variables, and linking these back to manipulated process variables for control.

What constitutes a quality product is not always straight forward, and this is especially true of food products where a multitude of variables are measured. For example dairy powders have a number of physical, microbiological, functional and sensory characteristics that are all important in determining the final product quality. A failure in any one of these categories can mean a failure of the product to meet customer specifications, and having individual tests for measuring all possible variables on-line is unlikely to be industrially feasible, if it were even technically possible. This means many tests are carried out post-production, at an external laboratory, once the product quality is fixed.

The consequence of this delayed feedback and the system being sufficiently complex and multi-variable in nature is that it is not immediately obvious what plant inputs affect the different quality outputs. It makes it difficult to correlate cause and effect, especially when data is either missing or incomplete, or the processing and quality data are not

time-aligned and stored in separate databases.

Some measurements, such as composition, can be potentially made on-line or at at-line, using sophisticated instruments such as near infra-red spectroscopes (NIR) [3, 4]. However, other quality attributes can be very complex and determined by the interaction of both physical and chemical properties. For example the dissolution properties of dairy powders (e.g wettability, dispersibility, solubility) are driven by both composition and surface chemistry, as well as physical properties such as the particle size distribution and particle density [5, 6, 7]. Such tests tend to be highly dependent on the measurement technique and grading scale [8], with industry often using their own modified methods for manufacturing purposes or as per customer requirements.

Thus this work looks at using an alternate, or proxy, measurement for a complex quality functional test. Such an alternative should be easier to implement, and thus lower the barrier to implementation in the plant. Although a proxy measurement would be imperfect (as by definition it is not the actual test result) it can still be useful for large-scale industrial manufacturing. It can provide operators with an informed route for making changes to the plant operating conditions due to the availability of immediate feedback regarding the consequence of those changes, which is not possible with post-production testing.

The novelty of this work is in developing the idea of using a proxy measurement to infer a complex product variable for quality control at an industrial plant. The dispersibility of IWMP produced at large scale industrial plants is used as a case-study to show how a proxy measurement can be applied in working towards real-time quality.

## 2 Proxy Measurement

Establishing differences between different powders is easy, but within the same type of powder the differences are much more refined. When industry production is considered,

large scale plants are often originally designed to manufacture a narrow specification of product, for example whole milk (WM) dryers are not normally used for skim milk powder (SMP) manufacture. This means that the product specifications fit within a narrow band, and the variation within some of the physical and/or chemical properties is reduced.

This narrow difference can be used as an advantage. It opens the possibility that only one physical or chemical property drives the main difference in quality within the same type of powder manufactured at the same plant i.e. that most of the variation can be attributed to a very small number of factors, as the others can be effectively considered constant. And thus this factor can be used as a proxy measurement for monitoring and controlling a complex quality variable that can otherwise not be measured in real-time.

The key considerations when choosing and testing a proxy measurement depend on the industrial context. Not all quality variables have the same requirements. For some characteristics, both upper and lower specification limits are important, as well as the actual level of the result. An example would be bulk density, where the exact result determines the volume of powder that needs to be packed and shipped, and thus the transportation cost. For other tests there may only be an upper limit, and the exact result may not be important so long as it does not breach this limit. Many of the dissolution properties of milk powder fall in this context, for example wettability, dispersibility, solubility and the heat stability tests. In these cases, so long as the specification limits are met, there is no direct economic benefit in having a specific result.

If the test level result is considered of secondary value over knowledge, or if the product is meeting the specification, then the next important factors to consider are the false-positive and false-negative rates. Because a proxy test is not the actual test, it should be expected that there will be some error associated with using it as a predictor. However, the false-positive and false-negative rates cannot be too high in order for the test to be useful for decision making. This is especially important when the quality failure events are rare, but none-the-less very costly economically.

Table 1: A table showing the proportion of poor dispersibility results with a high sifter and packer bulk density during industrial spray drying of instant whole milk powder (IWMP).

<b>Dispersibility Failure Rate</b>			
		<b>Packer Bulk Density</b>	
		<b>Low</b>	<b>High</b>
<b>Sifter Bulk Density</b>	<b>Low</b>	0.08 %	0.5 %
	<b>High</b>	0.6 %	3.8 %

For example, Table 1 shows the dispersibility (a dissolution functional property, discussed in Section 3.1) failure rate as a function of bulk density measured at two different points of the spray drying process. The Table shows that when both the packer and sifter bulk densities are high, the relative probability of failure increases by approximately 50 times compared to when both the sifter and packer bulk densities are low. However, as the absolute failure rate is still remarkably low, having a high and sifter and packer bulk density cannot be used as proxy, as the majority of the time,  $\approx 96\%$ , the powder will still meet specification, even if it is at a higher risk.

Another challenge for a proxy measurement is that it must be repeatable and have good resolution to pick up the differences within the same type of product, as the powder being manufactured at any one industrial plant tends to be fairly consistent. If the proxy test lacks resolution and repeatability it may completely miss the fine differences in the powder that drive the quality from being in, to being out-of-specification.

Therefore a proxy measurement is an alternate measurement of a complex quality variable, such as a functional or sensory characteristic. It has be easy to measure and directly related or proportional to the quality variable of interest, whilst minimising spurious correlations.

### 3 A Case Study of IWMP Dispersibility

The ‘instant’ in IWMP is designed to reconstitute rapidly in cold water and is achieved using two additional processing steps over the traditional WMP manufacturing process: agglomeration and the addition of lecithin. The agglomerated powder is obtained by recycling fines to the top of the dryer in order to incorporate them into larger particles, which are more easily penetrable by water. As WMP contains a large hydrophobic fat fraction, a small quantity of the emulsifier lecithin is added in order to make the surface of particles hydrophilic [5]. This improves the dissolution properties.

One of the instant dissolution properties of IWMP is termed dispersibility which is a measure of how quickly the powder particles break up and dissolve without leaving a residue. This case study considers an industrial plant manufacturing IWMP with premium dispersibility performance. However, this plant failed to consistently meet the specification, and due to the lack of immediate feedback discussed in Section 1, it was not obvious what caused the problem or how to rectify it. Consequently a proxy measurement of the dispersibility of IWMP using particle size measurements will be used as a case study in order to infer a complex quality variable that cannot be measured at the plant at the time of manufacture.

#### 3.1 The Dispersibility Measurement Method

The IDF standard for measuring dispersibility is the ISO 17758|IDF 087:2014, “Instant dried milk – Determination of the dispersibility and wettability”. It involves stirring a specific quantity of milk powder into water at 25 °C for 20 s and then passing it through a 150  $\mu\text{m}$  mesh to retain any un-dispersed solids. The solids content in the reconstituted milk is then calculated to determine the percentage of the powder that has been dispersed successfully, and the higher the percentage of solids in the final liquid the better the dispersibility. However it is not the only method available as noted in [8].

For example the New Zealand Dairy Board method used a different mesh size, of 300  $\mu\text{m}$ , with a different stirring speed and dispersion time, and looks at the residue content retained by the mesh directly by comparison with a chart [5]. Thus the precise analysis method can vary, and can also be product specific. In all cases the analysis is manually intensive, time consuming, and cannot be automated nor set up on-line easily.

This work used process data obtained directly from an industrial plant, and the actual method used to determine the dispersibility of powder is confidential owing to customer-specific requirements for this production. Notwithstanding, the test is similar to those described above and was classified into three categories: superior dispersing powder, which disperses completely within the test time; off-specification powder, which does not meet customer requirements and; good dispersibility, which is powder that still meets specification but not by a large margin.

### 3.2 Using Particle Size as a Proxy Measurement for Dispersion

Different types of dairy powders show varying dispersibility performance due to their disparate chemistry. For example, protein powders normally show poor re-hydration and dispersibility characteristics compared with skim and whole milk powders [9, 10, 11]. However, when a single type of product is considered, the next dominating factor driving dispersibility performance is the particle size [5, 11, 7, 8].

The recommendations governing the extent of the tails of the particle size distribution for best instant properties of IWMP hypothesized by [5] are:

1. No more than 10–20 % of particles  $<125 \mu\text{m}$  in size, (known as the fine fraction).
2. No more than 10 % of particles,  $>500 \mu\text{m}$  in size (known as the coarse fraction).

and it is generally agreed that as the percentage of fine particles in the powder increases, the dispersibility worsens [7, 5]. Following these recommendations, it makes sense to use

the particle size as a potential proxy measurement for the dispersibility of IWMP during production. However it should be noted that these criteria for good performance are fairly loose and it was not clear if this could be transferred to an industrial plant to classify the performance of IWMP i.e. as a real-time proxy measurement.

A laboratory investigation was launched and the particle size was first measured at five different IWMP plants in order to see if those used for manufacturing premium grade IWMP had different distributions compared with those used for manufacturing standard powder. If the particle size could not be used for differentiating the powder from the different plants, then it would be unlikely that it could be used to differentiate powder produced from the same plant due to the reduced variation.

### 3.3 Method and Materials

The particle size measurement depends on the instrument used [12], therefore two instruments were tested in order to see if it affected the accuracy of the proxy measurement. Samples of varying dispersibility were collected from an industrial plant and measured using two instruments: 1) the Malvern Mastersizer 2000, a laser diffraction instrument and, 2) sieving to assess the particle size.

When using laser diffraction, approximately 30 g of milk powder was fed into the feed hopper for the dry dispersion system. The metal ball bearings in the inlet sump were removed, as they caused attrition of the sample. The feed pressure was adjusted to 0.1 bar and a background reading was taken for 10 s before the powder feed flow rate was adjusted to the required laser obscuration of 1–5%. The sample measurement time was set to 20 s. The instrument was further set up with a refractive index of 1.46, and an absorption of 0.

For sieving, only two sieves were used, giving three size fractions and hence lower resolution, in order to minimise potential powder breakdown during measurement. 30 g of powder was weighed out and mixed with 1 wt% Syloid 244 FP flow agent from Grace



Davison, as IWMP has poor flowability characteristics. The powder was then fractionated using 125 and 355  $\mu\text{m}$  aperture sieves, 200 mm in diameter, with a vibratory Retsch AS200 sieve shaker for 30 minutes at an amplitude of 1.8 mm. The powder remaining on each sieve was weighed and the sieves were then cleaned with detergent and dried before the next analysis.

### 3.4 Results and Discussion

Initially, the particle size was measured across five different plants, and it was found that all of the plants manufactured powder within different particle size ranges. The plant used for manufacturing premium product and achieving the specification consistently manufactured powder with the lowest fines content (particles  $<125\ \mu\text{m}$ ), varying between 5–13 wt%, meanwhile the plant of interest for implementation of the proxy measurement had 15–24 wt% fines. Yet another plant, that could not be used to manufacture premium grade product, had a fines content of approximately 30 wt%. Since only the operating conditions at the plants can really be modified, (as opposed to the mechanical design of the dryers once built for example) therefore it is unlikely to be possible to shift the average particle size produced by one plant into the range produced by another, it can only be shifted within a more limited range. This means that the measurement has to be sufficiently sensitive, and repeatable, to pick up meaningful changes in the particle size within powder produced at a single plant.

The particle size of samples taken at the plant of interest measured using sieving and laser diffraction are shown in Figures 1a and 1b respectively. Hatched lines have been overlaid to isolate areas where the off-specification product is most concentrated, and these limits are different depending on the measurement. For sieving, in order to isolate off-specification results it is best to have both an upper fines limit, of 21 wt%, and a lower coarse particle limit, of 16.5 wt%. When using laser diffraction, only one limit was necessary, of maximum 18 vol% fine particles. The recommendations around the

Table 2: Estimated false-positive and false-negative rates for using a) sieving or b) laser diffraction as a proxy for dispersibility test results. (See also Fig. 1.)

(a) Sieving

<b>Dispersibility</b>	<b>&lt;21 wt% fines AND &gt;16.5 wt% coarse</b>	<b>≥21,wt% fines OR ≤16.5,wt% coarse</b>
In-specification	positive-positive 97 %	false-positive 42 %
Out-of-specification	false-negative 3 %	negative-negative 58 %

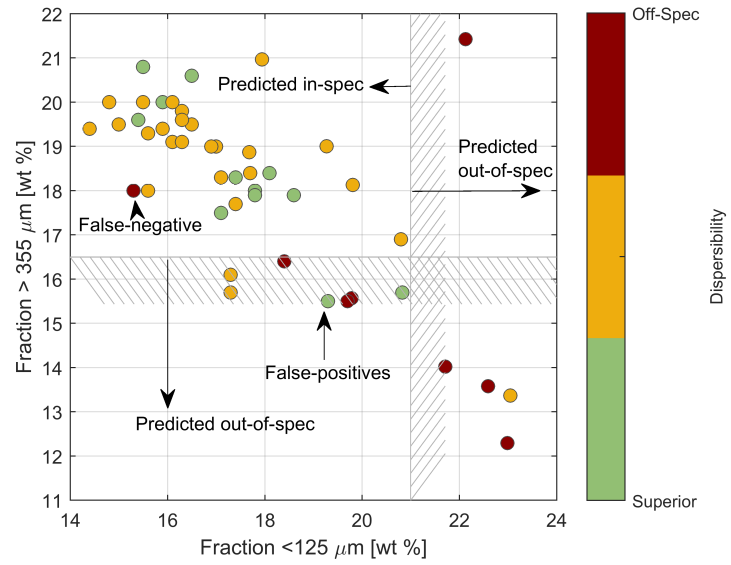
(b) Laser Diffraction

<b>Dispersibility</b>	<b>&lt;18 wt% fines</b>	<b>≥18 wt% fines</b>
In-specification	positive-positive 97 %	false-positive 56 %
Out-of-specification	false-negative 3 %	negative-negative 44 %

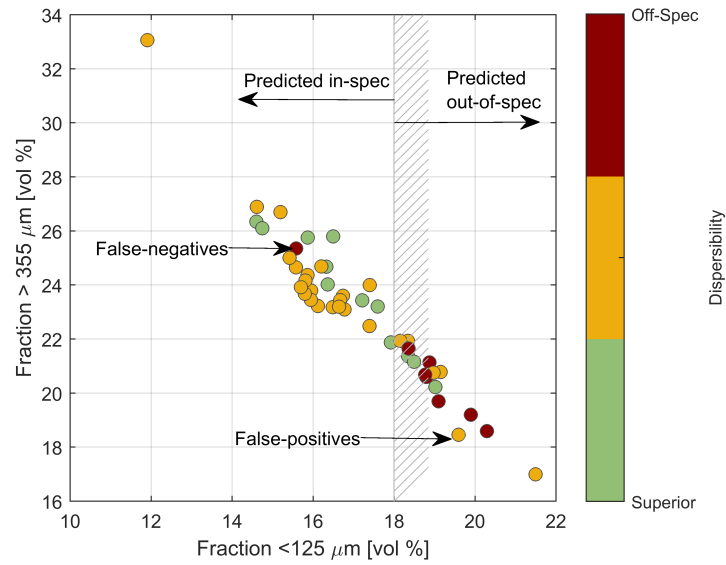
maximum amount of fine particles to meet specification are similar to those discussed in Section 3.2 for powder having good instant properties. However the exact value vary based on the measurement.

Table 2 shows the false-positive and false-negative rates of using particle size as a proxy for the dispersibility. It can be seen that if the powder is within the particle size recommendations, then it is almost guaranteed to be within specification for dispersibility. There is only a small chance of it not meeting specification, given the samples analysed.

If the powder is predicted to be out-of-specification using the particle size criteria, then



(a)



(b)

Figure 1: Suggested criteria for using particle size information as a proxy for dispersibility at the specific industrial plant investigated using a) sieving b) laser diffraction. The overlaid hatched lines isolate most of the off-specification product. Note that the exact limits differ from those given in [5] in Section 3.2.

there is still roughly a 50 % chance of it being within specification. It would be advantageous to reduce this false-positive rate i.e. reducing the results that are expected to fail the specification, when they will likely pass the actual test. This is important when considering making changes to the operational conditions, because they may not be necessary if the powder still has a large chance of being within specification. Furthermore, this may divert attention away from other more pertinent plant issues.

Out-of-specification powder occurs infrequently, which means that the proxy test is expected to be accurate 87 % of the time with sieving and 78 % of the time with laser diffraction. Again the fact that there is a 10 % difference shows that the exact instrument type is important for the measurement, and thus should be chosen carefully.

The accuracy of the investigated proxy measurement is sufficiently high to provide useful information regarding the dispersibility of IWMP: if the proxy indicates that the powder being produced is within specification, then there can be confidence in the dryer operating conditions for producing well dispersing powder. Therefore this approach offers a route for inferring a complex quality variable in real-time that otherwise cannot be measured at the time of production.

Furthermore, these proxy measurements, when done in real-time can then be combined with process data using multi-variate techniques to both increase the accuracy of the method, and understand how the quality is affected by the operating conditions. Measurement is only one of the first steps in working towards real-time quality, as one first needs to know if one is achieving the product specifications before knowing whether corrective action needs to be taken.

## 4 Conclusions

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in real-time. Instant whole milk powder (IWMP) dispersibility, a manual and laborious test, was used as a case-study. It is normally carried out post-production at industrial dryers, resulting in no immediate feedback regarding the manufactured powder, and thus difficulty in linking the quality to the processing conditions and an inability to control the quality in real-time. Particle sizing was tested as a proxy measurement for inferring the dispersibility of IWMP at an industrial plant. It was found that it could be applied in industry to predict the quality of the powder with high confidence, of 97 % given the data, for predicting in-specification product. Although the predictability of out-of-specification product was not as high,  $\approx 50$  %, the low frequency of such events meant that the proxy still had a relatively high accuracy overall. The ability to implement a measurement during production allows for feedback regarding the process to occur in real-time, and enables the development of understanding of what processing conditions, and how, affect the quality of the product. ————— .r33 This work shows how a proxy measurement, an alternative simpler technique, can be used to infer a complex product quality parameter for application in industry in real-time. Instant whole milk powder (IWMP) dispersibility, a manual and laborious test, was used as a case-study. It is normally carried out post production at industrial dryers, resulting in no immediate feedback regarding the manufactured powder, and thus difficulty in linking the quality to the processing conditions and an inability to control the quality in real-time. Particle sizing was tested as a proxy measurement for inferring the dispersibility of IWMP at an industrial plant. It was found that it could be applied in industry to predict the quality of the powder with high confidence, of 97 % given the data, for predicting in-specification product. Although the predictability of out-of-specification product was not as high,  $\approx 50$  %, the low frequency of such events meant that the proxy still had a relatively high accuracy overall. The ability to implement a measurement during production allows for feedback regarding the process to occur in real-time, and enables the development of understanding of what processing conditions, and how, affect the quality of the product. ===== This work shows how a proxy measurement (an alternative simpler measurement) can be used to infer a complex product quality variable

for application in industry in real-time. The measurement of instant whole milk powder (IWMP) dispersibility is only measurable via a manual and laborious test, was used as a case-study. Dispersibility measurement is normally carried out post-production at industrial dryers, resulting in no immediate feedback regarding the manufactured powder, and thus difficulty in linking the powder quality to the processing conditions and an inability to control the powder quality in real-time. Particle size was tested as a proxy measurement for inferring the dispersibility of IWMP at an industrial plant. It was found that it could be applied in industry to predict the quality of the powder with high confidence, of 97% given the data, for predicting in-specification product. Although the predictability of out-of-specification product was not as high,  $\approx 50\%$ , the low frequency of such events meant that the proxy measurement still had a relatively high accuracy overall. The ability to implement a measurement during production allows for feedback regarding the process to occur in real-time, and enables the development of understanding of what processing conditions affect the quality of the product. ~~~~~  
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