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Nota Técnica

# PARTICLE SIZE DISTRIBUTION APPLIED TO MILK POWDER REHYDRATION

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Besides increasing shelf life of fluid milk, milk powder has physical and functional properties that allow it to be used as ingredient. The rehydration process is complex and happens in four steps: wettability, sinkability, dispersibility and solubility. Works have been conducted aiming at the development of an easier, more convenient and reproducible method to evaluate the dissolution properties of dairy powders. Therefore, the aim of this work was to evaluate dispersibility and solubility through particle size distribution in order to establish a new rehydration index for whole milk powder. The particle size distribution and morphological characteristics of seven samples of milk powder (from A to G) were analyzed. Samples F and C differed from the others which formed a similar group. Principal component analysis divided the samples into three different groups, allowing the indication of an efficient rehydration index to determine the powders dispersability.

Keywords: dairy products; laser diffraction; microscopy; dispersibility; solubility.

## INTRODUCTION

Milk is very important for diet because of its nutritional value, which may build up immune protection and aid in the prevention of some illnesses. However, the nutrients and high humidity make it very perishable. Hence, dairy products as milk powder became an alternative to increase shelf life.[1] Besides long storage periods, milk powder displays physical and functional properties that allow it to be used as ingredient in dairy industries (e.g. fermented milk, processed cheese, and ice cream) as well as in other food industries such bakery, confectionery and meat. When used as ingredient, milk powder improves nutritional value of final product while contributes to properties as color, texture, flavor, humidity, total solids, yields, emulsification, gelation, solubility, viscosity, foaming, and whipping.[2-4]

The rehydration process in water is complex and can be described in four stages or inherent properties of the powder. The first is wettability, the powder ability to absorb water on its surface and to penetrate the surface of water; second comes the sinkability, which is based on the powder capacity to immerse in water after getting wet; dispersibility comes next and represents the aggregates separation with release of individual particles; in the end comes solubility, a characteristic related to the speed of dissolution and total solubility which is also related to the quantity of material dissolved in a saturated solution.[4-6]

Brazilian law sets dispersibility for instant milk powder (%w/w) as 85% for the whole product and 90% for the skimmed and the partially skimmed milk powder.[7] The International Dairy Federation (IDF) sets the ISO 17758 | IDF 087 methodology as reference for dispersibility determination, which is by definition the percentage by mass of the dry matter of the samples that can be dispersed in a defined amount of water. Water content of the sample must be previously analyzed for this determination. However, tests to determine rehydration properties are usually highly dependent on the measurement technique and the classification scale, which in turn force industries to develop their own methods, adapted to specific applications or to meet clients demands.[8] Works have been conducted with the aim of developing easier, more convenient and reproducible methods to evaluate the dissolution properties of dairy powders.[8,9]

It is well known that these properties are influenced by composition and chemistry of particles surface, as well as particle size distribution and density of the particles.[8] Ergo, analysis of particle size distribution has been frequently used to characterize rehydrated milk powder.[10,11]

In this way, this work intended to evaluate dispersibility and solubility through particle size distribution in order to establish a new rehydration index for whole milk powder.

## EXPERIMENTAL

Seven samples of whole milk powder from different brands were analyzed (A, B, C D, E, F, G codification).The powders morphological analyses were conduct through images of scanning electron microscopy - SEM (x500) (Hitachi TM 3000, Hitachi Ltd., Tokyo, Japan). Particle size distribution was analyzed by laser diffraction (Beckman Coulter LS 13 320, Miami, FL, EUA) coupled to the aqueous liquid module (Beckman Coulter, Miami, FL, EUA).

Powders form samples, without rehydration, were slowly added to the analysis module, filled with room temperature water at 25 °C, until reading signal levels of 50% + or – 5% were achieved on the photodetectors PIDS (Polarization Intensity Differential Scattering System). Data were collected on the region of 0.04 to 2.000 µm every 90 seconds over 5 different times (1.5; 3.0; 4.5; 6.0; and 7.5 minutes).

Results were calculated with refractive indices of 1.332 for the dispersant (water) and 1.57 for the casein micelles, and 1.47 for the fat globules aiming at the observation of total solubility.[12,13] Data were represented by the percentage of occupied volume by the particles as a function of their size. Statistical analyses were conducted with the equipment software.

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## RESULTS AND DISCUSSION

Figure 1 shows the morphological analysis images for the different brands of powders. According to the morphological results, it is possible to observe that sample F presented lower amounts of fine particles, big individual particles and probably the occlusion of air was bigger during spray drying, resulting in different particle structure and agglomeration. In contrast, sample C presented a pattern of higher amount of broken particles. The remaining samples showed similar characteristics of agglomeration and presence of fine particles.

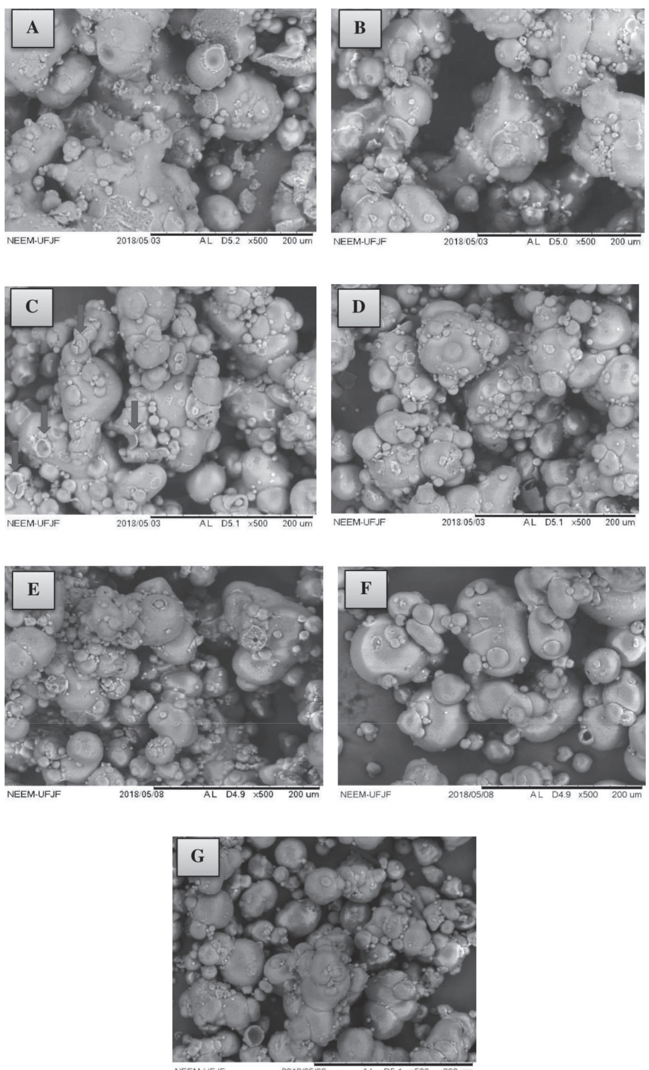


***Figure 1.** SEM images (x500) of samples*

The properties of the powders can be determined by the type of agglomeration which is affected by the process used to return the fines to the system.[14] If the fines are added close to the atomizer, the high humidity leads to more compacted agglomerates with a structure that resembles an onion. On the contrary, if the distance from the fines introduction to the atomizer is increased, the agglomerates are less compacted and called raspberry or grapes structures in descending order of compaction.

Particle size distribution was analyzed to understand the rehydration process and the results are shown in Figure 2.

Each sample showed a particular pattern for particle size distribution but, once again, samples F and C presented a distinguished behavior among the other samples. There was also some similarity of samples A and B with sample F, while samples D, E and G had a pattern more like sample C.

The proposed methodology considers that the ideal condition for particles distribution is in the region under 1 µm, the nanometric region of caseins. This could be observed with sample F, which also displayed an increase in particles at this region with the course of analysis time. On the contrary, sample C particles stayed above 1 µm, which can be considered the region in which fat and other non-hydrated particles can be found. The caseins may occupy the region above 1 µm when participating in the formation of fat globules after homogenization for milk powder production.[15]

Therefore, statistical analyses of particle size distribution were conducted to confirm the differences among the analyzed samples, as shown in Table 1.

Statistical analysis confirmed the differences for samples F and C while the others showed more similar pattern and values among each other. Chemometric analysis was performed with the goal of classifying the samples according to their properties and the results can be seen in Figure 3.

Figure 3.a shows an 66.7% of accumulation of responses in PC1 with a small range of variation inversely to the accumulation of responses in PC2 (8.54%), in which there is a bigger range of variation. Because of this bigger variation and to avoid the agglomeration of results, Figure 3.b shows the samples division with their respective rehydration indexes and possible classification in low, intermediate and high properties of dispersion and solubility.

Ergo, the behavioral profile of the samples was achieved through principal component analysis. Three groups were obtained with the division based on the quantity of particles under 1 µm. This value was chosen based on the characteristics of the curve obtained by the particle size distribution analysis. The curve is classified as bimodal, showing two modes with interstice close to $<1$ µm and $>1$ µm. The analysis of this result shows a mix of two distinctive population of particles, one being the caseins and the other being fat and other non-hydrated particles.

All in all, it was possible to create a rehydration index in which powders with $RI < 5$ are the ones with less condition to recover their original conformation after being rehydrated (low dispersion and solubility); $RI \geq 5$ and $\leq 20$ represents powders with intermediate condition to recover their original conformation after being rehydrated (medium dispersion and solubility); and $RI > 20$ belongs to the ones with higher condition to recover their original conformation after being rehydrated (high dispersion and solubility). It is worth mentioning that parameters as mixing equipment design, conditions of operation such as agitation and temperature, and chemical composition of the powder affect the rehydration of dairy powders.[6]

Thus, the suggestion of a rehydration index may help the choice of the best powder according to the application. For example, products with $RI > 20$ should be chosen for direct consumption because the original structure can be achieved more efficiently. On the other hand, powders with $RI < 5$ or $RI \geq 5$ and $\leq 20$ could be used as ingredients for the production of other foods (e.g. dairy products, bakery, confectionery, and meat products) without compromising the final characteristics because of its direct application with no rehydration. Therefore, this work stipulates an efficient rehydration index to determine powders dispersion.

At last, Figure 3.c shows the contribution of each resolution channel to the rehydration index intensity. Powders with higher amount of small particles (close to 0.195 µm) presented an increase in RI intensity, while powders with bigger particles (close to 52.6 µm) had a decrease in RI intensity.

The particle size analysis investigated on this work is the result of the combination of 80 resolution channels. Hence, this technique is more accurate and faster, from the analytical point of view, than the official dispersion analysis which comes from a single result. Thereby, the determination of RI through particle size may replace or complement the original analysis of powders dispersion although it is necessary to use an analytical device. The standard dispersibility analysis is manually intense, time consuming and cannot be automated or easily online configured. Thus, alternative techniques have been studied using laser diffraction, sifting,[8] particle size distribution[6] and even the development of a device[9] to facilitate the determination of dispersibility.

## CONCLUSIONS

According to morphology and particle size analysis, samples

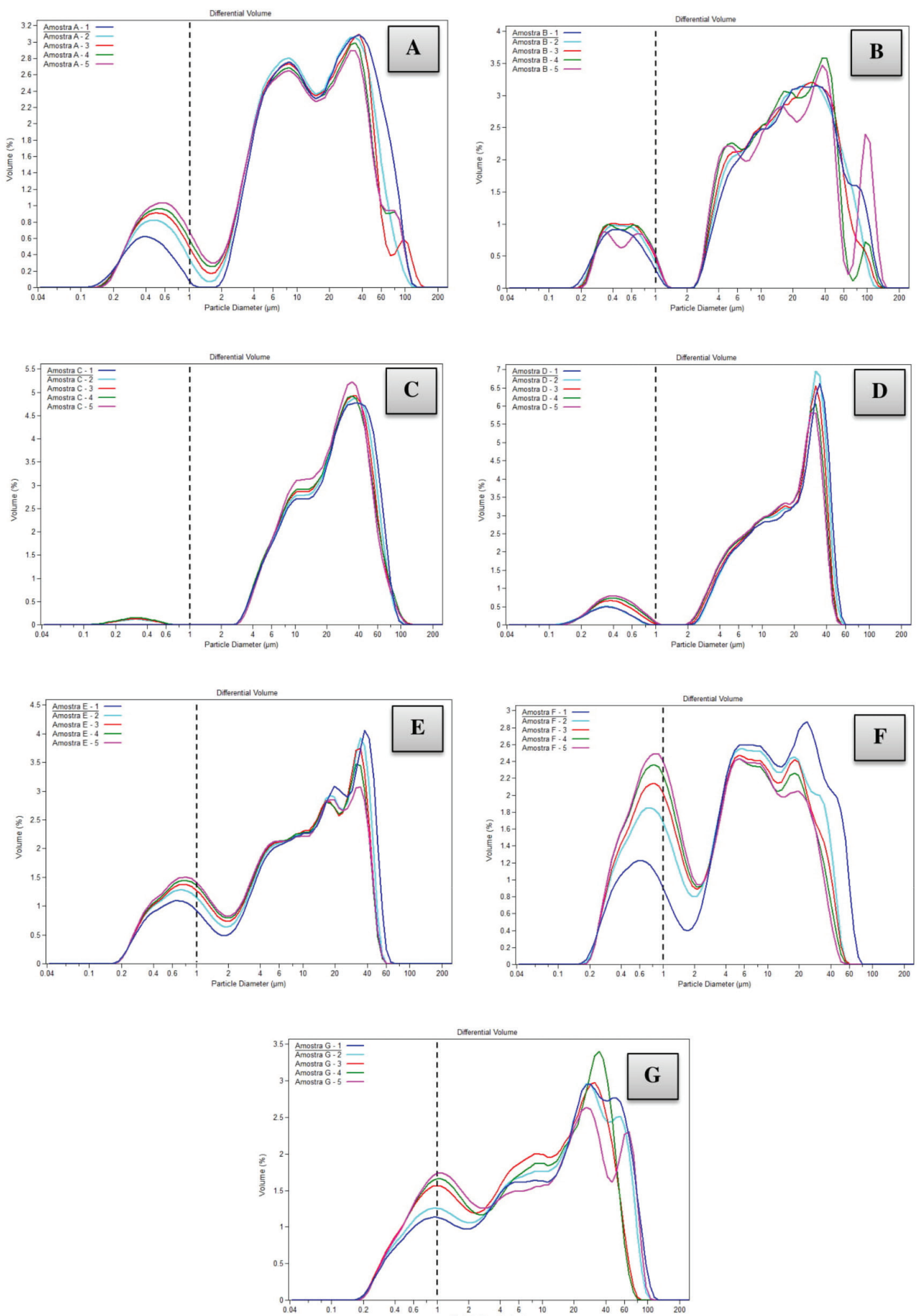


***Figure 2.*** *Particle size distribution over time (1.5; 3.0; 4.5; 6.0 e 7.5 minutes)*

**Table 1.** Particle size distribution of milk powder samples on the last reading (7.5 minutes)

| Sample | $d_{10}$ (µm) | $d_{90}$ (µm) | $d_{100}$ (µm) | < 1 µm (% volume) |
|---|---|---|---|---|
| A | 0.66 | 44.64 | 133.70 | 13.90 |
| B | 0.86 | 71.09 | 161.20 | 11.10 |
| C | 7.02 | 50.84 | 133.70 | 0.00 |
| D | 2.25 | 33.59 | 57.77 | 9.81 |
| E | 0.59 | 33.17 | 69.61 | 18.20 |
| F | 0.49 | 21.61 | 57.77 | 27.70 |
| G | 0.69 | 54.66 | 121.80 | 16.40 |
| Means | 1.79 | 44.23 | 105.08 | 13.87 |

C and F presented contrasting results while the other samples had similar results among each other. The measurement of immediate dispersibility is performed through a manual and laborious test. Therefore, the particle size analysis seems to be a simple and efficient method to determine dispersibility and solubility properties. This work established a rehydration index that makes it possible for the classification of whole milk powder according to dissolution properties for dairy powders.

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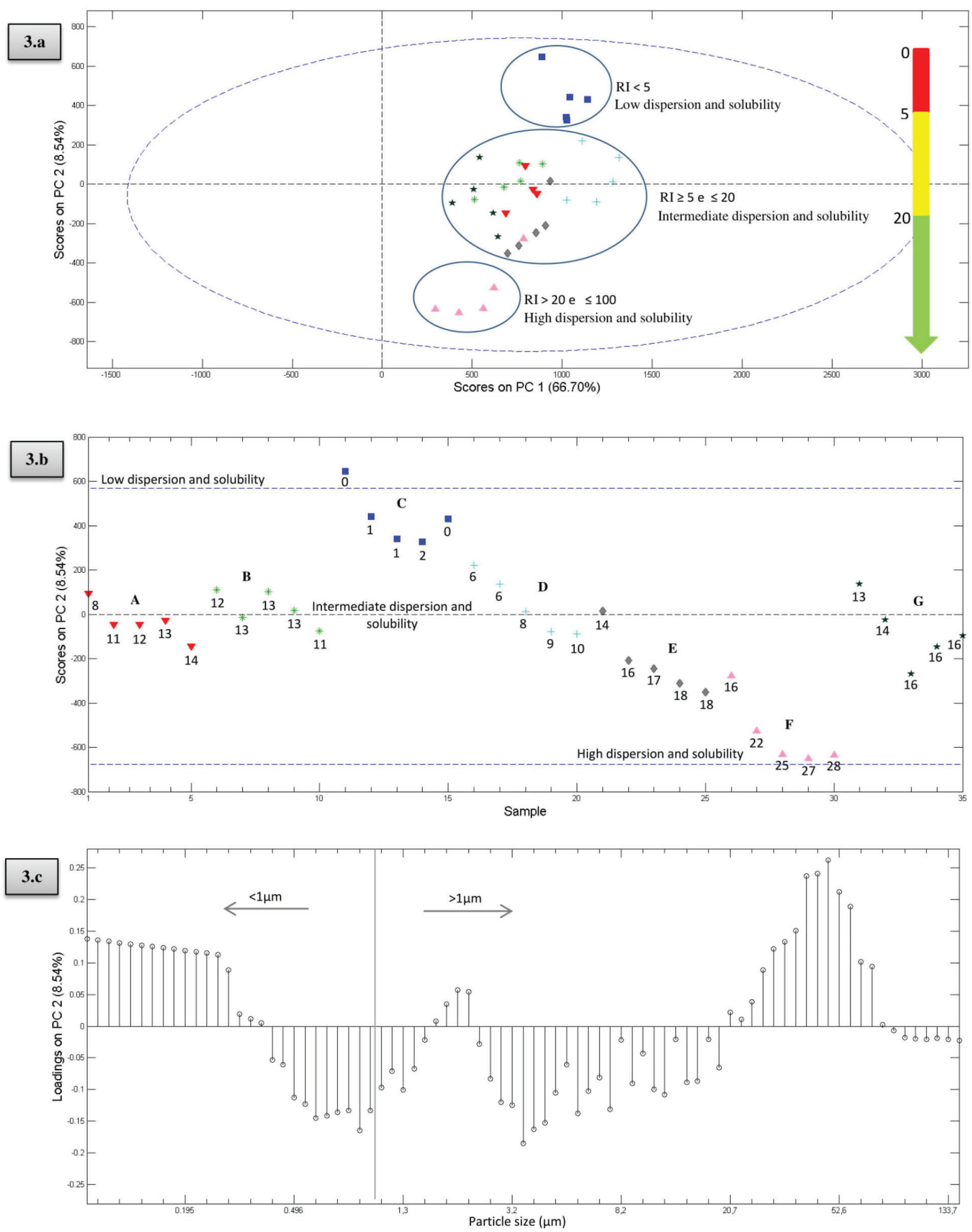


***Figure 3.*** *(a) Graphic of scores PC1 versus PC2; (b) graphic of samples versus scores PC2 (the numbers displayed in the graphic are referent to the rehydration indexes); and (c) graphic of loading PC2. Graphics represent the particle size analysis of seven milk powder samples A (▼), B (✳), C (■), D (+), E (◆), F (▲) and G (★)*

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