

Flow properties of industrial dairy powders

Jean Luc ILARI

Département de Génie des Procédés Alimentaires, Laboratoire de Génie Alimentaire et Particulaire,
ENITIAA, BP 82225, 44322 Nantes Cedex 3, France

Abstract – This paper is a review of the flowability of dairy powders and of the existing methods for the measurement of flow of powders. Their application to dairy powders is discussed and followed by some recommendations. The physical properties tester from Hosokawa is today the best for dairy powder flowability measurements. Results for 53 dairy industry powders are presented and discussed.

Dairy powder / flowability / physical property / method / behaviour

Résumé – L'écoulement des poudres de produits laitiers. L'article étudie et classe les méthodes de mesure de l'écoulement des poudres, et s'intéresse à leurs possibilités d'application aux poudres laitières. Il analyse et discute les difficultés, risques d'évolution de produits et hétérogénéité des pratiques, et insiste sur les diverses précautions à prendre. De l'analyse comparative des méthodes se dégagent quelques recommandations relatives à l'usage et l'exploitation des résultats. Cette comparaison conduit à recommander la méthode de l'analyseur de propriétés physiques Hosokawa. Les mesures de propriétés comportementales, indices d'écoulement et de foisonnement (propriétés fusantes) de 53 poudres d'industries laitières sont présentées avec d'autres éléments de caractérisation et discutées.

Écoulement / poudre laitière / propriété physique / méthode / comportement

1. INTRODUCTION

The common sense idea of flow is understood well when you are emptying a bottle of water (or wine), and can be extended to a similar situation of a bottle full of solid

powder. This extension works for semolina, but not at all for icing sugar or flour as examples. The main reason is the cohesion of the small particle powders, and eventually arching. The flow of powders is an important industrial problem. Historically, the

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Correspondence and reprints

Tel.: (33) 2 51 78 54 62; fax: (33) 2 51 78 54 67; e-mail: ilari@enitiaa-nantes.fr

pharmaceutical industries, making many industrial products from basic powders, met the “flow problem”. Research developed early to improve the flow and get practical results, reported by Neumann [24]. The initial work led practically reducing each powder (with a certain distribution of particles) to a “quasi-model powder”. This simplification is based on size control and the use of sieving techniques. In a way, this industrial solution stopped the research on understanding the flow behaviour of real powders. Then an intensive and legal use of glidants (silica and stearates) became the second solution [9].

Today we are still in this situation with dairy industrial powders, also called milk powders. They can neither be considered as model powders, because they often have large scale granulometries coupled with structural changes, nor as a “homogeneous” family as they are so diverse in chemical content.

Generally speaking, flow (which means difficulty or ability in flowing, and also behaviour all along the flow) is an averaged behavioural property of the heterogeneous particle population of a given powder. Each powder itself presents a very significant number of acting and influencing behaviour parameters, characterising the single and the bulk materials. For each parameter, a dispersion does exist and is very often forgotten. This dispersion, a “supposed minor parameter” has consequences on all the other properties of each powder, including flow behaviour and measurement! So the system is complicated, and it is difficult to go from intuitive qualitative relations to quantitative physical ones. One of the main consequences is that there are very few international publications about the flow of dairy powders, and with limited objectives. Flow and flow measurement methodology are difficult to compare or to present together.

In dairy powder processing plants, for research purposes as well as for laboratory

everyday work, people try to characterise all the powder properties to reach the concept of functionality or “end use properties”. Flowability is just one among all the most interesting properties included in “technological functionality”, a general concept with many application fields. And anyone involved practically in big-bag filling and storage, bin filling and emptying, silo management, air transportation or even dosage, mixing and conditioning of powders is concerned. They experience every day why the flow properties of dairy powders are so important for good industrial activity.

2. THE GREAT DIVERSITY OF DAIRY POWDERS AND THE INFLUENCE OF THE NEIGHBOURING FACTORS

2.1. Dairy powders

Dairy powders present a very large diversity. Some of the powders derive directly from the milk, just modified eventually by a centrifugation treatment for standardisation. Milk, skimmed milk and full fat milk are just a diversification of the same raw material. They differ only by the fat content, but the other components of these three powders are globally the same. The greater difficulty comes from the fact that the fat can be situated inside the particles, or in other cases outside. If the fat is on the surface, the powder becomes sticky when the temperature rises. Indeed, when fat melts, it gives an adhesive, rubby and viscous liquid that breaks any particle motion. Consequently, some of these powders do not flow in certain temperature conditions. Their fat can be extracted by successive melting and crystallisations with temperature changes.

But dairy powders also include hundreds of derived bioproducts, developed within the whole dairy industry, and following the idea of avoiding any loss and

sparing any biochemical materials. All these products, issuing from biotechnology and from the cracking of milk and whey, and also from the cheese industry by-products, present a very large panoply of components, structures, physical states, degrees of purity, distributions of particle shapes and sizes. This incredible diversity makes the idea of measuring their flow properties with the same machinery a dream rather than reality. The versatility of components and structures also makes the conception of a polyvalent measurement system very difficult. The dry powder form also covers recent development of ingredients and biologically active additives, dairy powders of which the components are the fruit of an extraction, pure or fermented materials, eventually encapsulated. The value of these new products is 10, 20 or 100 times more than that of a classical milk powder. It becomes very important to avoid losses and to correctly manage the process, and so flowability becomes an important property.

2.2. Role of neighbouring parameter

When you compare dairy powders and a model powder of glass particles, you understand a great difference: glass beads keep their properties on a large scale of physical parameter change, but dairy powders are continuously in evolution, from the beginning of the drying process to storage and end use. All the physical parameters act on powder evolution, but with special kinetics depending on the neighbouring parameters. These evolutions and their kinetics are specific to any dairy powder and include all the previous technological steps as major parameters. Two similar samples immediately after drying, even if they are close together and with the same physical properties (a_w , specific weight, size and shape of particles, etc.) cannot present the same flow if they are differently influenced by the interparticle behaviour, depending on temperatures, relative humidity, compression

levels, and even geographical situation in the whole population.

As a behavioural property, the flow of a powder is an integration of all the particle interactions. Everyone is able to understand that the “flow function” is not at all a simple one and that as much as possible, physical parameters have to be fixed. In many advanced countries laboratories are also regulated in moisture and temperature. In all other situations too many changing parameters influence the measurements. So it should be accepted that we must remain very prudent in the comparison of different measurements because the drying kinetics, handling and neighbouring parameters are never the same. Two sister particles can also present different thermodynamical histories.

3. PHYSICAL PREREQUISITES AND INTERACTIONS WITH THE MEASUREMENT METHODS

The representative value of the sample is fundamental, and the powder is in continuous evolution. Practically, the majority of the laboratory rooms depend on the daily situation and climate. Theoretically, only a physically controlled and regulated room should be devoted to the flow measurements.

Very important for the definition of the best storage conditions, the equilibrated water activity (a_w) is not known exactly. It differs by whether the powder is in desorption or in adsorption, because of a marked hysteresis on many sorption isotherms of dairy powders. As a consequence, when one receives a powder in one's laboratory, one could dry it or moisten it in function of both the relative humidity and the temperature. The time itself induces changes. Practically all storage conditions present an effect on the powder. Everyone knows that cement bags can

solidify. After consolidation with time (and/or moisture) “caking” does also exist for all dairy powders, and obviously can stop any continuous powder processing. The general tendency in the laboratories is to keep the powder inside its original bag until the beginning of the measurements, and to work quickly when the powders stay in the open atmosphere to avoid moisture sorption (or minimise influence). It is very important to remember the problem of sampling. A measurement is valuable only if the sample is representative of the whole powder. For one dairy powder we have done a full-scale examination of segregation in bags, very important even for this low density ($\sim 300 \text{ kg}\cdot\text{m}^{-3}$) powder [16]. Segregation makes sampling very difficult.

Evaluation of the flow capacity of powders is a very important topic for many industries, not specific to dairy ones. It must be understood that the dairy powder properties could be difficult to discover if we try to determine their flowability with the same laboratory machines as those used for metallic or glass powders (which present a much higher density and potential energy). The rheological behaviour of a powder is complex as it is linked to all cohesion, densification and avalanche phenomena.

Very often cohesion and densification develop forces against flowing, which also supposes a free trajectory for each particle of the bed. The physical state of a powder is not well defined, as powder behaves as a multiphasic product. All the possible exchanges between phases are already initiated during drying, and linked to molecular and particular interactions throughout the time. The very great diversity in methods is multiplied by the great diversity in classes of particulate products, demonstrated by the Geldart classification [6]. The flow of particulate materials under gravity does exist in natural phenomena, and the simple observation of nature enables the development to imitation or physically similar methods using the gravity field. Many

scientists want to separate the influence of the powder properties and of the external factors and parameters to try to explain the behaviour. But flowing induces so many numerous particle interactions inside a given powder that it seems hard to get easy conclusions.

In some well-known and fixed conditions the external factors and parameters stay constant (constant neighbouring temperature and moisture, for example). But this could induce errors if one forgets that in many real situations these external parameters change themselves, and consequently modify both the powder properties and their resulting flowability, different from those appreciated or measured in fixed conditions.

The methodology of comparison leads to using the same method for close powders, with a good chance of success. But initially developed for mine dusts, career stones and fine, pure chemicals and their mixes, flours and ground dry materials, metals and crystallised materials, the more classical methods seem at the beginning to have been created and developed each for a very specific product. So these methods are difficult to correlate in the majority of concrete situations.

Through our experience, we suggest understanding that the concept of flow is at the centre of a cobweb from the origins, having connections with civil engineering, laboratory characterisation, rheology of solids, mechanics of fluids, process engineering, sensory analysis, behaviour studies, physics, mathematics and statistics.

4. APPLICABILITY OF METHODS TO DAIRY POWDERS

Our laboratory practised all of the methods on a large quantity of diverse powders, with different success and pleasure. We concentrated on methods which appeared to be more discriminative, or rich in both

quantitative and qualitative results and information. Each measurement is worth while when one understands what has been done and then discusses the results. Big differences do exist between these methods: the preparation of the powder to be measured is not the same, and follows different requirements that induce specific structural states, and so specific flow behaviour. Storage and consolidation, vibration segregation and cohesion development, all have a very large impact on “interparticle” phenomena.

All these methods concerning powders originate from many different industries and apply different principles. It is actually difficult to compare them but a mechanical engineering professor, J. Schwedes, three years ago realised a general inventory for a private international company (I.F.P.R.I., International Fine Particle Research Institute, Wilmington, USA). This work is not completely accessible to public, but only partly [25]. Some years before, a good presentation and discussion, with practical experience from pharmaceutical powders was also done by Svarowsky [26]. The purpose and the growing number of these methods indicate that in every field specialists want to predict the flow behaviour of powders and extend it to all technological situations by the use of simulation methods applied to model powders. This problem is still in full discussion in the working party “Mechanics of Particulate Solids” of the European Federation of Chemical Engineering. More recently, Guigon [10] also discussed the main methods practised in chemical engineering and applied to chemical powders.

The historical use of these diverse methods for dairy and even food powders is very short compared to its use for minerals and geological materials, metals and oxides, sands and glass, chemicals, salts and cements and even complex compounds and new taylorised materials. But it is important to understand that changes in drying tech-

nologies involve a new need for milk and dairy powder characterisation. The flow measurement methods have been applied first to very simple powders (which behave as physical models), then extended to more complicated powders. They worked well with special milk powders in which, for example, the size distribution is very reduced (a simple situation because of a simplified product). The crisis of energy has led to a step by step change in drying installations with significant consequences for the products. Fluidisation first permits better heat and mass transfer coefficients together with more regularity in the size and structure of dried particles. Agglomeration develops inside the fluidised bed, together with a better productivity. Then fluidisation combines with agglomeration and coating by using additional nozzles and often spraying additional components, such as liquid emulsifiers or ligands. This combination of drying with particle texturisation permits the production of a new kind of dairy powder [15]. These agglomerated dairy powders, presenting new structures, also possess new physical properties, new particle shapes and new particle interactions which are obligatorily involved in the flow phenomena. From this time we know that the flow properties depend on the drying technology. The four generations of spray dryers each give a special kind of powder, and with a good reproducibility so long as machinery and parameters are run equally. We proved [7] some years ago that for the recent multistage dryer MSD, if you change the technological parameters, it should be possible to “taylorise” some characteristics of the produced powder and consequently its flow properties. The study and interpretation of concrete flow behaviour measurements introduce more and more parameters. If they are independent and not linked together we could discuss their precise influence. Flow properties are strongly linked to “security of factories” and “functional properties of products”. Faced with the difficulties reported by industrial partners, we have

decided to apply the behavioural experimentation of Carr [4] to specific milk powders. As a consequence, the special Hosokawa Tester [11, 12] has run in our laboratory for more than twenty years. It has helped to solve some practical and difficult problems, inside and outside industry. We use it every day, when needed, instead of other machinery we developed in parallel, such as the mechanical shear cells and the consolidation bench. We developed and studied them at the beginning, and finally we have stopped using them because they are too “time consuming”.

5. FLOW MEASUREMENT METHODS APPLIED TO DAIRY POWDERS

5.1. The mechanical methods (using shear testers)

They were developed from soil mechanics and physical theories [23], and have been applied to some dairy powders: their main problem concerns their price, their precision mechanically speaking, the number of shear cells needed for reliable results and the time needed to prepare, realise and treat the results. A lot of work is devoted to these shear test devices, the last one being circular, such as the Pesch cell or the Walker device [27] and the others, the first ones, proceeding from the Jenike [21] shear cell (USA) or from the French “boîte de cisaillement de Cassagrande”.

These measurements were developed historically on products other than dairy ones. They determine cohesion and give a value for the angle of internal friction of the powder itself. They can be adapted to measure the angle of friction on a surface. These methods are physically acceptable and give good and reliable results. But they depend on the consolidation pressure that milk powder normally accepts without rupture, on the powder bed geometrical structure, and they are really too time consuming. Reliable results have been obtained by Loisel [23], summarised in the following table, and concerning an agglomerated skimmed milk powder ($D_{50} = 60 \mu\text{m}$) from a second generation dryer (Tab. I).

The angles of internal friction values are difficult to determine as they depend on so many manipulations and historical parameters. The results often depend on the laboratory. For skimmed milk powders, the scientific literature oscillates between 36 and 44 degrees, depending on the dryer, the analyst, the shearing device and laboratory conditions.

5.2. The practical use testers

They are commercial systems derived from the previous ones, but faster than the above shear testers. These are Hosokawa Cohetester from Japan (Hosokawa Nauta, Doetinchem, Holland) and Johansson Hang-Up Indicizer (JR Johanson inc., The Solids Flow Consultants, San Luis Obispo,

Table I. Cohesion (kPa) and angles in degrees of internal friction (standard deviation) for normal skimmed milk powder.

Shearing device	Filing method	Angle of internal friction	Cohesion (kPa)
Cassagrande	Many couch	39.6 (2.9)	3.7 (1.3)
Shear cell	Uniaxial comp.	39.8 (1.2)	4.8 (1.8)
Triaxial cell	–	41.7 (2)	6 (2)
Triaxial cell	Consolidated	43.5 (2)	1 (2)

California, USA) which are sold at a high price. But they are not so physically well defined as the previous ones, and cannot be considered as pure research tools. They can be considered as routine tools to check or follow the same industrial production, because they give fast and acceptable results.

5.3. The easy methods derived from fluid viscosity evaluation methods

They are based on the action of gravity. These methods combine the principle of the “viscosity measurement” of Newtonian fluids following Engler, with the measurement of the time needed for a given quantity of powder to flow through a controlled diameter funnel. This method takes care of the bridges, which can appear inside the powder and stop the flow. The results are expressed in time of the flow for a given quantity, or in the critical diameter value for the hole. The commercial equipment is used in pharmaceutical industries mostly for dense powders and crystalline powders. It was initially developed and discussed by Gioia [8] and one commercial device is called Flodex (Fischer Bioblock Scientific, matériel de mesure de l’indice de fluidité Flodex, Catalogue 2001, p. 299, Illkirch, France).

5.4. The combined time consolidation and wall friction measurements

They derive from the first category ones and again need more equipment and more time to be applied, as they suppose a solid consolidation in time. As a consequence, their cost and financial investment are rather significant, and no more acceptable today. A good description has been given by Loisel [23].

5.5. The sensory evaluation methods and close processes

The first method is the determination of the angle of repose. Too many practical

fluctuations exist in the way one produces the slope as well as in the way one measures the angle (see Svarovsky [26]). Rapid comparison of results from different laboratories can be meaningless. That is a problem and there is a need for normalisation [1, 19, 20]. But everyone can imagine and realise many other sensory methods with some creativity, using some specific tools, such as a glass pipe [13]. I found them interesting to develop and to practise because they helped me to ask and answer a lot of questions about the powders. They can be applied well to dairy products. For example, some users keep the powder closed inside a transparent glass pipe, without the influence of external moisture. It is easy to observe the powder’s behaviour, to compare two powders, even very close in properties, and to try to discriminate them. And it works quite well for a specialist who understands exactly why he is manipulating the powder in this way. But it is difficult to recommend them for general use because some physicists are not convinced of their value. As for the angle of repose determination, so many differences could be in the methodology, the neighbouring parameters, the practice and the subjective interpretation: you must study beforehand, be careful and become a specialist. These methods, that can also be considered as screening ones, are cheap, very worthwhile and easy to apply to dairy powders.

5.6. The latest created methods

They are still in development and evolution, and so no references in the scientific literature are available, only commercial ones.

5.6.1. The automatic device from TSI

The device recently developed by TSI, a turning cylinder, permits the determination of the turning angle of repose of a given quantity of powder placed inside the cylinder. By using electronic cells inside, and an

outside light, it is possible to determine the value of the angle of repose. It is also possible to study avalanches. This method is applied, particularly in the United States, to lactose powder used in pharmaceuticals (TSI Aeroflow, automated powder flowability analyser, TSI, St. Paul, MN, USA).

5.6.2. The avalanche direct determination

The method of measurement of the mass (or the volume) of avalanches is still presently integrated in many research programmes. It was introduced by the Australian scientist Brian Kaye [22]. The stability of the methodology applied to achieve these measurements is very important, specific to any researcher, and needs to be standardised before general use could occur. Through the avalanche methodology, it is possible to get a fractal representation of each powder, which permits interesting comparisons following the evolution of powder properties in time by an easy comparison of the phase space attractor [22]. In fact, the only machinery developed commercially by TSI is “Aero-flow”, an automated powder flowability analyser, which also permits the study of the progress of the powder avalanche. It gives a flowability index (scale between 0 and 20) and a cohesivity index, comparative behaviour items (TSI Aeroflow, automated powder flowability analyser, TSI, St. Paul, MN, USA).

5.6.3. The powder rheometer

The new Manumit method, just developed by “Stable Micro Systems” is an adaptation of the well known TAX2 Texture Analyser, with a rotational torque to press (compaction) or swell (aeration) the powder before you slice it up and down successively. Measurements are known for microcrystalline cellulose only, and the measurement system needs to be standardised on simple powders before being applied to dairy ones (Stable Micro Systems, The new ManUmit Powder Rheometer, Godalming, Surrey, UK).

5.7. The Hosokawa powder tester

The “multisequential” manipulation and evaluation of powder properties through the behavioural tester, Hosokawa, gives the flowability index and the floodability index. This is the tester that is mainly used and recommended for food powders, including dairy ones. The Hosokawa powder tester [11, 12] constitutes a multiparameter analytical tool to characterise many physical powder properties, including the flow ones. It evaluates four physical properties, measuring the angle of repose, angle of spatula, compressibility and cohesion by special behavioural manipulations, as first described by Carr [4]. These four physical properties are assessed in arriving at a flowability index having a theoretical value from 1 to 100 (in practice never under 10). A floodability index, also ranging between 0 and 100, is obtained from the flowability index together with other values. These ones derive from the measured angle of fall, angle of difference and evaluation of dispersibility. Floodability corresponds to the fluidisation of fine particles in the air, difficult to control and spattering. In fact seven parameters characterising the powder are measured and converted into global behaviour values, the flowability and floodability indexes. The algorithm building the relation between the measured values and the index has been experimentally obtained (with more than 300 powders) by the first experimental work of Carr [4]. Indexes by themselves permit classification of powders, with a standard deviation of one unit on the scale 0–100. It is also possible to compare together some of the internal values obtained through this methodology, such as, for example, those of the angle of repose, or the angle of difference. The angle of repose is classically considered as an evaluation of flowability [24]. But one must be very clever in the way the powder is put down on a horizontal surface. Various prototypes of this measurement system exist, which differ in the powder processing and

deposition. As another example, the angle of difference is a good evaluation of the instability of a powder cone under a vibration or a shock, and so is linked to the avalanching phenomenon: the higher the angle of difference, the larger the avalanches of the particles. From the Hosokawa measurements it is also possible to calculate directly the valuable Hausner ratio (tapped specific weight divided by loose specific weight), a compressibility index classically used in pharmaceutical industries. The Hausner ratio is correlated with flowability: the powders with a high Hausner ratio generally present significant cohesion, and consequently bad flowability.

The Hosokawa tester includes, in fact, real manipulation of the powder [4]. If one's senses are really working, one realises at the same time a kind of "sensory analysis" of the powder, a virtual comparison, as well as a real one for many powders. Then it is possible to integrate this evaluation in the report written about the powder. Many years ago, I developed a jury and pushed forward the capacities of sensory analysis for powder evaluation [13]. Today I recommend the Hosokawa methodology, and consider it to be better and more reliable than the sensory analysis because of its larger application field and its reduced risks of error. In fact, there is more complementarity than opposition between the two methods, but the methodology for the Hosokawa tester is easier, and now better known and well accepted even by people with initial criticism.

6. ANALYTICAL RESULTS AND FLOW PROPERTIES OF DAIRY POWDERS

6.1. Additional determinations for maximal analytical control

To avoid the influence of both relative humidity and temperature, we decided to work all the time inside a dedicated closed

laboratory (temperature: 20 ± 2 °C, relative moisture between 50 and 65%) that we considered as "stable" during one flowability determination, and from one to another. In order to study the role of moisture through time, the powder was first completely dried, then prepared in reduced volume tanks at different a_w [23] and for a sufficient time (> some hours) to get the initial equilibrium state (checked by a constant weight of the sample).

Water activity (a_w) of powders was measured with a Novasina capacity sensor, which was calibrated with specific salts before every measurement series. For the particle size measurements, we used both the normative sieve methods and the methodology of Malvern laser diffraction with the size of particles through the volume of equivalent spheres, and the three ratios of 10%, 50% and 90% of the whole distribution curve. Diameters D_v 0.1, D_v 0.5 and D_v 0.9 are present in our results. Finally, to check and understand the flowability of a given powder is not very easy. There is a need for many other determinations which help to characterise precisely the product and the conditions of the measurements.

The following Table II gives a list of the measured characteristics for some well-characterised dairy powders, and gives the code for the reading of the results. Table II gives the code, with the explanation: percent moisture (1) is obtained with an oven at 102 °C (constant weight), equilibrium a_w (2) at a temperature of (20 ± 2 °C) with a Novasina Capacity probe, and tapped and loose specific weights (4 and 5) are in $\text{g}\cdot\text{cm}^{-3}$ or in $\text{t}\cdot\text{m}^{-3}$. They are measured by the Hosokawa tester methodology. All the angles (8, 9, 10, 11) are in degrees, and the indexes of flowability and of floodability (12 and 13) are calculated from Hosokawa [4, 11] and in the scale 0–100. The codes for reading all the following tables are the same as in Table II.

Table II. Code of measured characteristics.

Code	Measured characteristics	Units
1	Moisture	%
2	Equilibrium water activity	–
3	Granulometry D_v 0.1, D_v 0.5, D_v 0.9	μm
4	Loose specific weight	$\text{g}\cdot\text{cm}^{-3}$
5	Tapped specific weight	$\text{g}\cdot\text{cm}^{-3}$
6	Calculated compressibility	%
7	Hausner ratio = (5) / (4)	–
8	Angle of repose	°
9	Angle of fall	°
10	Angle of difference = (8) – (9)	°
11	Angle of spatula	°
12	Flowability index Hosokawa [0–100]	–
13	Floodability index Hosokawa [0–100]	–

6.2. Results on flowability and floodability of dairy powders

6.2.1. Flowability of dairy ingredient powders and feeds for calves

The first analytical work was done because we had been asked to answer the question: why there was so a great diversity in the flowability of the formulations of the Univor[®]. We controlled the flowability of the mix and tried to find an explanation by also controlling the flowability of the ingredients included in the mix. The chemical formulations of the calf replacers Univor A, B, B', C and D, chemically and nutritionally equivalent, all present bad but different flowability indexes; respectively, 37, 37, 51, 31 and 39. But the flowability of their main raw materials are not so bad, and have better flowability indexes, respectively, 60 (skimmed milk), 53 (whey proteins), 46.5 (milk 35% tallow). The size measurements indicate that granulometric repartition plays a large role in flowability, as a multimodal powder presents the best index

(51), due mostly to its large particles (> 160 μm). It appears from this that the change in drying technologies for saving energy could have a good impact on some end use properties of powders, such as flowability.

Specific results on feed for calves, Tetilac and its ingredients

Tetilac is the trademark of a recent feed for calves. As the formulation becomes more and more complicated for economical reasons, many additives have been introduced to improve the formula and theoretically to avoid practical difficulties. Tetilac is a good example of the bad flowability of a mix, after economical modifications including much more components, and also a flowability agent, the glidant "tixosil". The ingredients also have very bad flowability, such as the protein Deltavo, with a very low index value of 17; and the glidants, when alone, also get bad values of 38 (silica) or 35 (tixosil). This means that it is not easy to introduce them into the powder system and to mix them. Spray

Table III. Analytical results on feeds, milk replacer, dairy powders and ingredients.

Powder / Code	1	4	5	6	7	8	9	10	11	12	13
Skimmed milk powder	5.0			3.3		44.0	24.0	20.0	65.0	60.0	76.0
Sprayed whey	3.3			34.1		39.0	18.0	22.0	57.0	53.0	87.0
Tallow sprayed 35% fat	2.0			27.6		40.0	20.0	19.0	62.0	46.5	68.0
Emulsifying agent (2%)				24.3		38.0	15.0	24.0	44.0	59.0	84.0
Silica (glidant)						47.0	26.0	22.0	62.0	38.0	73.0
Calf replacer:											
Univor A (mix)	3.4		Monomod. 26 % [63.8]	32.5		44.0	21.0	23.0	75.0	37.0	66.0
Univor B (mix)	<5		Bimod. 32 % [>160]	34.2		50.0	41.0	9.0	56.0	37.0	47.0
Univor C (mix)	<5		Monomod. 31%[80,100]	37.6		48.0	33.0	15.0	71.0	31.0	51.0
Univor B' (mix)	3.9		Trimod. [40.5]63.8[>160]	26.3		40.0	19.0	21.0	65.0	51.0	79.0
Univor D (mix)	<5		Trimod. agglom. 39% [>160]	31.2		45.0	24.0	21.0	64.0	39.0	66.0
Product TETILAC. A mix feed for calves and its raw ingredients		0.43	0.65	33.8	1.51	45.0	23.0	22.0	68.0	36.0	63.0
Skimmed milk spray 0%		0.46	0.65	29.2	1.41	42.0	23.0	19.0	76.5	44.5	74.0
Minerals agglomerated		0.68	1.02	33.3	1.50	40.0	24.0	16.0	69.0	43.5	68.5
“Cristal” whey turbine		0.43	0.63	31.7	1.47	39.5	23.5	16.0	74.5	46.5	71.0
Dairy proteins Deltavo		0.31	0.58	46.5	1.87	55.5	42.5	13.0	77.5	17.0	27.0
Full fat milk spray 35%		0.37	0.53	30.2	1.43	47.0	29.0	18.0	69.0	38.0	59.5
Delectosed whey MSD nozzle + fat		0.43	0.56	23.2	1.30	39.5	23.5	16.0	57.5	50.0	70.0
MSD nozzle whey + fat		0.38	0.54	29.6	1.42	43.0	28.0	15.0	62.0	42.0	58.5
Dairy proteins Dievet		0.45	0.60	25.0	1.33	37.0	27.5	9.5	72.5	47.0	57.5
Dairy proteins Almospray		0.47	0.75	37.3	1.60	41.0	23.0	18.0	51.0	45.0	72.0
Dairy proteins Protarmor		0.42	0.66	36.4	1.57	47.0	22.0	25.0	60.5	45.5	77.0
Glidant “Tixosil” alone		0.11	0.20	45.0	1.82	43.0	29.0	14.0	60.5	35.0	65.5
Whey		0.49	0.78	37.2	1.59	46.0	29.0	17.0	62.5	38.5	64.5

dried products (including many technologies of drying) can integrate formulation powders, such as multicomponents Univor or Tetilac, but they also have flowability problems, and their addition could not improve the flowability of the mix.

6.2.2. Comments on the different dairy feed ingredients

Crystalline powders: the first idea is that in the majority of the situations, we are not facing a sort of model powder with an average size, but a real, complicated size distribution, even sometimes with different structures. For the MSD powders, the large sized particles are agglomerates [15], and in the same sample the little sized ones are particles, not agglomerated, or which separate after being agglomerated.

The angle of spatula is interesting, very low for the sugar "sol 5", large sugar particles having excellent flowability. Lactose flowability is in-between this and icing sugar. That flowability value of 60 is just the lower limit to avoid processing problems [11]. Hygroscopicity is a factor influencing the flowability of lactose, with eventual recrystallisation.

The new commercial sugar "sol 5" is a good flowing product and has a low compressibility, instead of a large distribution granulometry. The four dextroses are market competitors. They have poor flowabilities and a more significant difference in floodability. These powders could be improved through granulation. They will present problems in use.

The raw materials for formulation of calf, sheep and animal feeds all have bad or very bad flowability, and present a large scale of a_w and of other characteristics. They are used in heterogeneous powders, very difficult to process. The mineral integrated mix for feed industries should be difficult to prepare with only one unit operation (mixing), from such a large diversity

of component properties and physico-chemical behaviour.

6.2.3. Discussion on milk products for human beings

For all these spray dried dairy powders, particularly among the first ten where particle size is well known, a great variety appears in the values of the physical characteristics as well as in the flowability and floodability values. The limited number of results makes it difficult to understand which parameters are influencing the flowability of a given powder. But the fat content, the particles and the powder structures play a role.

From the five results for skimmed milk it is possible to see that the generation of the dryer influences many primary parameters, which are included in the flowability evaluation. These five powders are close together, with the same chemistry. In practice, it is necessary to reach the technologically acceptable compromise for powder properties when drying the powder.

There is no parallelism between specific weight and flowability. Each powder has a primary (dispersion) and a secondary (agglomeration) structure, which both integrate together in the results of the measurement of flowability. The lecithin-enriched instant milk MSD certainly has good solubility, but its flowability number is too low. The dispersion of the values shows that it is possible to tailorise the flowability of MSD, as already known [7], and also that of other processes and products [17]. It can be concluded from experience that the convexity of agglomerates and their relative homogeneity in size develop a better flowability than that presented by some previous kinds of spray dried powders with a large sized dispersion. But if one uses all the capacities of formulation in a MSD process [17] the new formulated powder that is created could have bad flowability again. It could result from as many different parameters as bad shapes,

Table IV. Flowability of lactose, substitutes, ingredients or dairy mix additives.

Products / Code	1	2	3	4	5	6	7	8	9	10	11	12	13
Lactose		0.10	15.5 - 96.9 - 228	0.64	1.00	35.8	1.55	42.0	19.5	22.5	63.3	60.0	75.5
Sugar sol 5		0.25	223 - 635 - 1131	0.88	0.97	2.6	1.11	41.0	25.2	15.8	42.5	80.5	72.5
Iced sugar			5.7 - 25.8 - 140.7	0.50	0.97	48.2	1.93	43.7	19.6	24.1	70.7	40.0	79.5
Dextrose "amylum"		0.30	48.8 - 135 - 315	0.56	0.76	26.3	1.38	42.8	31.2	11.6	61.5	51.5	59.5
Dextrose "avebe"		0.32	63 - 168 - 371	0.62	0.81	23.4	1.30	43.0	24.0	19.0	58.5	51.0	71.5
Dextrose "roferose"		0.27	29 - 106 - 268	0.55	0.83	33.7	1.50	46.0	27.0	19.0	66.0	40.5	59.5
Dextrose "cerestar"		0.36	41 - 110 - 227	0.58	0.75	22.6	1.29	40.0	25.0	15.0	55.8	53.0	68.0
Vitamin B2	0.3	0.32		0.15	0.35	57.1	2.33	48.1	26.9	21.2	74.5	24.0	48.5
NaHCO ₃	3.0	0.30	18% < 50 5% > 315	1.07	2.83	62.2	2.64	48.2	31.0	17.2	60.2	34.0	61.0
Ca ₂ H ₂ (PO ₄) ₂	3.5	0.73	90% [50-100]	0.77	1.13	31.8	1.46	49.3	26.5	22.8	68.6	41.0	67.0
Ethoxyquine	3.6	0.12		0.16	0.17	6.5	1.06	44.8	32.2	12.6	60.6	50.0	61.5
Cu SO ₄	31.0	0.39	Trimodale D _v 0.5 = 125	1.13	1.38	18.2	1.22	43.1	27.7	15.4	58.4	57.0	73.0
Chloride of choline	1.5	0.11	Trimodale D _v 0.5 = 160	0.64	0.73	12.3	1.14	39.4	21.8	17.6	71.3	56.0	74.0
MnO	0.4	0.41	D _v 0.5 = 160	0.94	1.90	50.3	2.01	49.2	25.6	23.6	65.0	29.0	67.5

Table V. Flowability of human dairy powders.

Spray dried products / Code	1	2	3	4	5	6	7	8	9	10	11	12	13
Milk A 26% fat			51.7 - 144.1 - 365.9	0.36	0.56	35.4	1.55	50.5	36.0	14.5	56.5	51	59.5
Milk B 26% fat		0.16	48.2 - 137.0 - 326.0	0.36	0.57	36.9	1.59	51.8	33.5	18.3	73.3	31	44.0
Dried milk normal				0.36	0.47	23.5	1.31	53.0	35.0	18.0	73.0	42	74.5
Agglomerated "instant" dried MSD + lecithin			$D_v 0.5 = 150.7$	0.33	0.49	32.6	1.48	57.0	46.0	11.0	67.5	26	39.0
Milk MSD "Canalac"			$D_v 0.5 = 243.8$	0.48	0.52	36.5	1.08	41.0	28.0	13.0	48.5	38	56.0
Acid whey			6.5 - 35.2 - 221.4	0.50	0.78	36.7	1.55	43.0	36.5	6.5	54.7	60	61.0
Coffee whitener 50% fat MSD			$D_v 0.5 = 206..3$	0.41	0.55	25.5	1.34	39.0	12.0	27.0	65.1	55	81.0
Skimmed milk 0% fat		0.53	31.1 - 81.3 - 172.0	0.50	0.63	20.9	1.26	34.0	16.0	18.0	53.8	66	84.0
Skimmed milk:													
Spray dried 1st gen.	< 5			0.62	0.89	30.0	1.44	48.0	31.0	17.0	77.0	52	72.0
Spray dried 2nd gen. with nozzle	3.92	0.24		0.55	0.78	29.0	1.42	42.0	29.0	13.0	64.0	42	62.0
Spray dried 2nd gen. with turbine	3.97	0.24		0.55	0.80	30.0	1.45	46.0	23.0	23.0	67.0	39	68.0
Spray dried MSD	4.70			0.39	0.53	26.0	1.36	49.0	38.0	11.0	55.0	47	60.0
Spray dried MSD (other factory)	< 5	0.24		0.54	0.72	25.0	1.33	45.0	26.0	19.0	66.0	59	77.0

puzzle effects (giving cohesion), reactive surface states and electrical charge considerations of the constituent primary particles. Also, the use of “glidants”, small particulate additives, develops on spray-dryers. Ten years ago in our laboratory an in-depth study of the mechanisms involved was achieved by Graindorge [9]. Glidants act, but their place of introduction and their quantity ratio have to be optimised (the legal maximum is 2% mass). The tendency is to add too much, with finally a worse result and consecutive pollution. Today, industrial people should have a certain, and “confidential”, know-how to follow and control the variations of powder properties [2, 3] due to the different generations of the dryers themselves. But each practical optimisation seems to be only a partial one, because only one parameter or factor of the powder is optimised, and very often others are lost.

7. CONCLUSION

The Carr study [4, 5] permits the building of a good classification system for predicting on a large scale the flowability and the floodability scores of particle populations, called an index. This classification is based first on size, with subdivisions in function of shape (micaceous, film, chips, flakes, fibrillar), and also on size uniformity (the uniformity coefficient is the ratio of the sieve aperture passing 60% (weight) divided by that passing 10%: it is a dispersion index of the population). When we care about dairy powders and their flowability, we have to study flowability with the aim of getting better control of the process, by a kind of preventive measure for a preventive management of the powder all through its life. But flowability is actually known to depend on two phenomena: the initial flow of some individual particles in an unbalanced physical situation (that is the initiation of flow), and continuity and regularity during flowing. These two main

steps do not appear with evidence in the Hosokawa methodology. Some of these phenomena have been extensively studied these last few years through avalanching behaviour and the theory of the attractor by Kaye et al. [22]. The cooperative behaviour of particles in powders includes both initial potential energy (linked to their density) and the particle interactions. So, for low-density powders such as the dairy and food ones, the initial flowing energy is low, and the interparticle forces are sufficient to brake the flow. The interparticle forces are themselves dependent on many factors, such as chemical components, physical state, size and distribution or repartition, shape and repartition, and even moisture and repartition, to stay limited to the more important ones. As a consequence the prediction, or the explanation, of the generic flowing properties cannot be reached today by a general formula, because too many factors are incorporated.

But in reduced fields of investigation, it should be possible to model flowability with only a limited number of specific factors. That is the reason why the Geldart classification of powders [6] in homogeneous behaviour classes through fluidisation is useful, and similar ideas are good to apply to the dairy powders.

We also have to think about this classification in function of the technological factors, which drive the creation of powders, with the aim of building the network of relations between technology parameters and characteristics of the products [18].

On the other hand, the quality of dairy powders also depends on many other end use properties, including solubility, wettability, mechanical resistance and even the impermeability of particles. We understand immediately that the quality requirements of dairy powders are diverse, and could be contradictory to the concept of flowability alone. The management of technology is, as a consequence, a compromise in function of many requirements. This

compromise may be reached for simple powders, close to the concept of "model" by their relative homogeneity in properties (such as being about the same size, same shape, same pure material and with the same surface properties). Today the great diversity of dairy powders obtained from many drying processes make the situation much harder, and suggest new characterisation efforts. That concerns formulated powders, obtained from a dairy base, such as calf feeds, sheep feeds, and also many formulations of baby foods. Cooperative phenomena, depending on many factors which integrate all through the powder technology, give to the ready-to-use powder a behaviour difficult to explain. Our thinking is in favour of integrated global concepts such as the Hosokawa gives. We can compare the Carr methodology [4] with the Geldart one. Geldart et al. [6], through their classification of behaviour in fluidisation (4 classes of integrated or global behaviour), have in fact divided the powders into "homogeneous behaviour" classes. The flowability index is doing the same. From the flowability index, you can fix the limits of homogeneous behaviour in powders, the limit of acceptable value for flowability inside your process, and so it appears really usable both for industrial people and for scientists.

There are many good, long and short ways of evaluating the flow of a dairy powder. The most simple and natural, sensory ones are not at all the worst, but they are often considered as subjective, and any dairy powder is a very complicated system.

After analysing many difficulties, we may conclude that even faced with a very large and versatile need for characterisation of physical and use properties, we must trust in the multidimensional behavioural approach, like the one developed through the work of Carr and commercially developed by Hosokawa. The large quantity of information that is obtained through the Hosokawa methodology is suitable for

flowability determination and permits a great number of other interesting comparisons. The behaviour is correctly evaluated, and some differences in the internal measurements can help interpretation. It should be easy today to establish a database to keep this information, as suggested previously [14]. And it is also possible, in the case of a global characterisation of product and process, to use the statistical tools to develop fruitful data treatment. That is a good solution for industrial problems such as the management of flowability, and of course, a technical and market advantage.

REFERENCES

- [1] AFNOR, Mesurage de l'angle de talus d'éboulement - Agents de surface, poudres et granulés, Norme NF T 73-008, Tour Europe, Cedex 7, Paris la Défense, France, 1978.
- [2] Borios C., Lancelot J., Davenas P., Ilari J.L., Vérification industrielle de l'influence de certains paramètres technologiques de fabrication sur les propriétés physiques de la poudre de lait écrémé, *Ind. Alim. Agric.* 101 (1984) 1211-1219.
- [3] Borios C., Lancelot J., Davenas P., Ilari J.L., Influence des technologies sur les propriétés physiques du lait en poudre, *Tech. Lait* 32 (1985) 10-21.
- [4] Carr R.G.Jr., Evaluating flow properties of solids, *Chem. Eng.* 72 (1965) 163-168.
- [5] Carr R.G.Jr., Classifying flow properties of solids, *Chem. Eng.* 72 (1965) 69-73.
- [6] Geldart D., Harnby N., Wong A.C., Fluidization of cohesive powders, *Powder Technol.* 37 (1984) 25-37.
- [7] Gharemann F., Ilari J.L., Cantoni P., Boudier J.F., Caractérisation des poudres de concentrés protéiques laitiers, *Rev. ENIL* 176 (1994) 25-31.
- [8] Gioia A., Intrinsic flowability: a new technology for powder flowability classification, *Pharmaceut. Technol.* (1980) 1-4.
- [9] Graindorge P., Contribution à l'étude par analyse des données des modes d'action des additifs d'écoulement pulvérulents en mélange avec des poudres alimentaires, Thèse, Université de Nantes, 1992, 248 p.
- [10] Guigon P., Khashayar S., Comment déterminer la coulabilité d'une poudre, in: Euroforum (Ed.), Caractérisation et propriétés d'usage des poudres, Symposium, 27 mars 2001, Paris, France, 2001.

- [11] Hosokawa Micromeritics Laboratory, Manual for the use of Hosokawa powder tester, Hosokawa Micron Corp., Osaka, Japan, 1981.
- [12] Hosokawa Micron B.V., Handbook Mechanical and thermal processing technology, Doetinchem, Holland, 1994, pp. 217–224.
- [13] Ilari J.L., Contribution de l'analyse sensorielle à l'évaluation des milieux pulvérulents, *Sci. Aliments* 4 (HS III) (1984) 89–97.
- [14] Ilari J.L., Une banque de données sur les propriétés physiques et comportementales des poudres alimentaires, *Récents Prog. Génie Procédés* 9 (1989) 90–100.
- [15] Ilari J.L., Conséquences de l'évolution des techniques et procédés de fabrication sur les propriétés rhéologiques des poudres, *Récents Prog. Génie Procédés*, 11 (1991) 250–258.
- [16] Ilari J.L., Multistage spray dryer study with multidimensional statistical methods, in: 1st International Symposium on Spray Drying of Dairy products, Oct 2001, INRA Rennes, France.
- [17] Ilari J.L., Loisel C., La maîtrise de la fonctionnalité des poudres, *Process* 1063 (1991) 39–43.
- [18] Ilari J.L., Qannari E.M., Analyse et gestion de technologies et de produits complexes à l'aide des techniques statistiques multidimensionnelles, in: *Congrès International franco-québécois, Génie Industriel, Albi, France, 1997.*
- [19] ISO, Poudres métalliques : Détermination de l'aptitude à l'écoulement au moyen d'un entonnoir calibré (appareil de Hall) Norme ISO 4490, 1978.
- [20] ISO, Engrais solides : Mesurage de l'angle de talus d'éboulement, Norme ISO 8398, 1989.
- [21] Jenicke A.W., Storage and flow of solids, Bull. N°123, University of Utah, USA, 1964.
- [22] Kaye B.H., Gratton-Liimatainen J., Lloyd J., The effect of flow agents on the rheology of a plastic powder, *Particle part. Syst. Charact.* 3 (1995) 194–197.
- [23] Loisel C., Contribution à l'étude des propriétés rhéologiques de quelques poudres alimentaires, Thèse Université de Nantes, 1988, 250 p.
- [24] Neumann B.S., The flow properties of powders, *Adv. Pharmaceut. Sci.* 2 (1967) 181–227.
- [25] Schwedes J., Testers for measuring flow properties of particulate solids, in: De Silva (Ed.), *Reliable flow of particulate solids*, Proc. Int. Symp., Porsgrunn, Norway, 2000, pp. 3–40.
- [26] Svarovsky L., Angle of repose and other handling angles, in: Svarovsky L. (Ed.), *Powder testing guide: methods of measuring the physical properties of bulk powders*, Elsevier Science Publishers, London, UK, 1987, pp. 71–78.
- [27] Walker D.M., A basis for bunker design, *Powder Technol.* 1 (1967) 228–236.