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Relative humidity of outlet air: the key parameter to optimize moisture content and water activity of dairy powders

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Abstract – The most widely used technique for dehydration of dairy products is spray drying. This is an effective method for preserving biological products as it does not involve severe heat treatment and allows storage of powders at an ambient temperature. The maximum moisture content of a dairy powder (max 4% for skim milk powder) is defined in the product specification in relation to the water activity, and this must be close to 0.2 at 25 °C for optimum preservation. From an economic point of view, it is very important to operate as closely as possible to this limit. Many dairy manufacturers and researchers have demonstrated and reported that powder moisture is related to the outlet air temperature, but this is not always true. The aims of this study were to evaluate the direct and indirect relationships between outlet air temperature and moisture content of skim milk powder in relation to the spray-drying parameters (concentrate mass flow rate, absolute humidity of inlet air and inlet air temperature) using a thermodynamic approach. Our experiments showed that moisture content of skim milk powder can be close to $5.1 \pm 0.0\%$ with variations in outlet air temperature of 77 to 87 °C. Other experiments showed that the powder moisture content can vary from $4.6 \pm 0.0\%$ to $5.2 \pm 0.0\%$ even when the outlet air temperature remains close to 86 ± 1 °C. These results indicate that there is no direct relationship between outlet air temperature and powder moisture content. It is preferable to use the Enthalpic Mollier-Ramzine diagram of wet air and certain transfer equations related to the Fick and Fourier laws to demonstrate that the powder moisture content is directly related to the relative humidity (RH) of the outlet air. The moisture content and water activity of skim milk powder were close to $5.1 \pm 0.0\%$ and 0.27 ± 0.01 for outlet air RH close to $7.0 \pm 0.1\%$, respectively, whatever the other drying parameter values. We demonstrated in this study that control of the RH of the outlet air is at least as important as control of the outlet air temperature to optimize the moisture content of a dairy powder, regardless of the absolute humidity of the inlet air, concentrate mass flow rate or inlet air temperature.

spray drying / dairy powder / thermohygrography / water activity

摘要 – 出口空气的相对湿度对乳粉水分含量和水分活度的影响。喷雾干燥是广泛使用的乳品脱水技术。由于喷雾干燥过程不采用较强的热处理,干燥产品可以在室温下贮藏,因此,喷雾干燥是保持生物活性产品最有效的方法。利用水分活度原理控制乳粉的最大水分含量(脱脂乳粉水分在4%左右)可以提高乳粉的质量,实验证明在25 °C、水分活度为0.2时是乳粉的最佳保存条件。基于经济上的考虑,乳粉的水分活度要尽可能接近这个限度值。许多研究和实践已经证明乳粉的水分含量与出口空气温度直接相关,但是也有例外。本研究目的是采用热力学方程评价出口空气温度和乳粉水分含量与喷雾干燥过程参数之间(浓缩物料流速、入口空气绝对湿度、入口空气温度)的直接和间接关系。研究证明出口空气温度在

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77 ~ 87 °C 范围内变化时, 脱脂乳粉的水分含量接近 $5.1\% \pm 0.0\%$; 而当出口空气温度保持在 86 ± 1 °C 时, 乳粉中水分含量在 $4.6 \pm 0.0\%$ 至 $5.2 \pm 0.0\%$ 内变化。上述结果表明出口空气温度与乳粉水分含量之间没有直接的关系。根据湿空气的 Mollier-Ramzine 焓图和基于费克和傅立叶定律的传递方程证明乳粉的水分含量与出口空气相对湿度直接相关。在出口空气相对湿度接近 $7.0 \pm 0.1\%$ 时, 无论其它干燥参数如何变化脱脂乳粉的水分含量和水分活度分别在 $5.1 \pm 0.0\%$ 和 0.27 ± 0.01 。本研究证明, 在不考虑进口空气绝对湿度、浓缩物料的流速或进口空气温度的情况下, 若要获得最佳的乳粉水分含量, 控制出口空气相对湿度与控制出口空气温度同样重要。

喷雾干燥 / 乳粉 / 温湿度测定 / 水分活度

Résumé – L’humidité relative de l’air de sortie : un paramètre-clé pour optimiser la teneur en eau et l’activité de l’eau des poudres laitières. Le séchage par atomisation est la technique de déshydratation la plus couramment employée en industrie laitière. De par son traitement thermique réduit, c’est une technique très efficace permettant de préserver des produits biologiques sous forme de poudre et de les stocker à température ambiante. La teneur en eau ou humidité maximale pour une poudre laitière (4 % max pour une poudre de lait écrémé) est déterminée en fonction de l’activité de l’eau qui doit être proche de 0,2 à 25 °C pour une conservation optimale. D’un point de vue économique, il est vraiment très important de travailler aussi proche que possible de ces limites. Beaucoup d’industriels laitiers et de chercheurs ont démontré que la teneur en eau d’une poudre était en relation avec la température de l’air de sortie, mais ce n’est pas toujours vrai. L’objectif de cette étude était d’évaluer les relations directes et indirectes entre la température de l’air de sortie et la teneur en eau d’une poudre de lait écrémé en relation avec les paramètres de séchage (débit de concentré, température et humidité absolue de l’air d’entrée) par une approche thermodynamique. Nos expériences ont montré que la teneur en eau d’une poudre de lait écrémé pouvait être proche de $5,1 \pm 0,0\%$ avec des variations de la température d’air de sortie comprises entre 77 et 87 °C. D’autres expériences ont montré que la teneur en eau d’une poudre de lait écrémé pouvait varier de $4,6 \pm 0,0\%$ et de $5,2 \pm 0,0\%$ avec une même température d’air de sortie proche de 86 ± 1 °C. Ces résultats indiquent qu’il n’y a pas de relation directe entre la température d’air de sortie et la teneur en eau d’une poudre. Il est préférable d’utiliser le diagramme enthalpique de l’air humide de Mollier-Ramzine et quelques équations de transferts suivant les lois de Fick et de Fourier pour démontrer que la teneur en eau d’une poudre est directement en relation avec l’humidité relative (HR) de l’air de sortie. La teneur en eau et l’activité de l’eau d’une poudre de lait écrémé étaient proches respectivement de $5,1 \pm 0,0\%$ et de $0,27 \pm 0,01$ pour une HR proche de $7,0 \pm 0,1\%$ quelles que soient les valeurs des autres paramètres de séchage. Nous avons démontré dans cette étude que le contrôle de l’HR de l’air de sortie est un paramètre au moins aussi essentiel que la température de l’air de sortie pour optimiser la teneur en eau d’une poudre laitière, sans se soucier des valeurs du débit de concentré, de l’humidité absolue et de la température de l’air d’entrée.

séchage par atomisation / poudre laitière / thermohygrométrie / activité de l’eau

1. INTRODUCTION

Drying (consisting of lowering water activity by water elimination) is an effective method for preserving biological products, since it does not involve severe heat treatment and allows storage at an ambient temperature. Large amounts of liquid dairy products (skim and whole milk, whey, and various fractions resulting from membrane filtration and chromatographic separation) are dried in order to produce feed, food and ingredients. Most of these powders are

spray-dried. This process consists of spraying the concentrated liquid in droplets of about 50 µm into a large drying chamber containing air heated at around 200 °C. The temperature of the product itself lies between the wet bulb temperature and the temperature of the outlet air, i.e. it remains below 100 °C. Since drying occurs within a few seconds, thermal damage is limited. Classical spray-dryers are combined with a fluid bed in the wet zone, which usually agglomerates the fine powder coming from the drying chamber, completes the drying

process and cools the powder. In recent 3-stage installations, another fluid bed with agglomeration and additional drying functions is included at the bottom of the drying chamber [2, 8].

There have been few scientific or technical studies on the powder quality obtained from spray drying related to the process parameters, physico-chemical composition or microbiology of the concentrates. Manufacturers have acquired expertise in milk and whey drying processes through an empirical approach. However, due to the variety and complexity of the mixes to be dried, more rigorous methods based on physico-chemical and thermodynamic properties have now become necessary. Indeed, the lack of technical and thermodynamic data and of scientific methods has prevented manufacturers from optimizing equipment in terms of energy costs and powder quality [5].

Masters [2] and Pisecky [3] showed that the moisture content of a dairy powder is related to the outlet air temperature, the moisture content decreasing when the outlet air temperature increases. However, the knowledge of moisture content is not sufficient in itself to evaluate food stability during storage. The study of water activity (a_w) has been developed to take into account any interactions of water with the other food components. Water activity reflects water availability, i.e. the greater the water availability, the higher the a_w value, which involves certain biological (growth of moulds, yeast or bacteria), biochemical (lipid oxidation, enzymatic or non-enzymatic reactions) or physical changes (stickiness, caking, collapse or lactose crystallization). The water activity of dried milk products is correlated with moisture content and temperature. At low moisture content (corresponding to $a_w < 0.2$) the casein is the main water absorber. Within the intermediate range of $0.2 < a_w < 0.6$, sorption is dominated by the transformation of the physical state of lactose, and salts have

a marked influence above this level [3]. The a_w of milk powders consisting of the non-fat milk solids and milk fat is mainly controlled by the moisture content of non-fat solids, since the fat has no influence. The main interest in these relationships is the control of the shelf life of foods [4]. The a_w should be close to 0.2 at 25 °C for optimum preservation [1]. All these studies have shown that there is a strong correlation between a_w and moisture content, but without describing relationships with the spray-drying parameters (temperature, and relative (RH) and absolute (AH) humidity of the inlet and outlet air).

The aim of this study was to demonstrate the relationships between the relative humidity of the outlet air, moisture content and a_w of a dairy powder.

2. MATERIALS AND METHODS

2.1. Skim milk powder

The skim milk used in this study was obtained from a local dairy plant (Préval, Montauban, France) and concentrated by two-stage falling film vacuum evaporation (GEA, Niro Atomizer, St Quentin-en-Yvelines, France) at Bionov (Rennes, France) at $400 \pm 5 \text{ g}\cdot\text{kg}^{-1}$. The first evaporation stage was carried out at 60 °C, leading to a concentrated milk at 40 °C. The evaporation capacity was $180 \text{ kg}\cdot\text{h}^{-1}$. The spray drying of skim milk concentrates was performed at Bionov (Rennes, France) in a 3-stage pilot-plant spray-dryer (GEA, Niro Atomizer, St Quentin-en-Yvelines, France). The atomizer was equipped with a pressure nozzle (0.73-mm-diameter orifice) and a 4-slot core (0.51 mm nominal width), providing a 60° spray angle. Evaporation capacity was 70 to $120 \text{ kg}\cdot\text{h}^{-1}$ (depending on inlet and outlet air temperature and air flow) according to Schuck et al. [7]. The temperature of the concentrate before

Table I. Spray-drying parameters, moisture content and water activity of skim milk powder.

Test	Concentrate flow rate (kg·h ⁻¹)	Inlet air		Outlet air			Powder	
		Temperature (°C)	Absolute humidity (g·kg ⁻¹ dry air)	Temperature (°C)	Absolute humidity (g·kg ⁻¹ dry air)	Relative humidity (%)	Moisture content (%)	Water activity –
1	132 ± 6	230 ± 1	1.0 ± 0.1	86 ± 2	27.0 ± 0.5	7.0 ± 0.2	5.2 ± 0.0	0.28 ± 0.01
2	97 ± 5	178 ± 1	1.0 ± 0.1	77 ± 2	19.0 ± 0.6	7.0 ± 0.2	5.2 ± 0.0	0.27 ± 0.01
3	98 ± 5	197 ± 1	1.0 ± 0.1	87 ± 3	20.0 ± 0.8	5.0 ± 0.3	4.5 ± 0.0	0.23 ± 0.01
4	109 ± 6	197 ± 1	1.0 ± 0.1	82 ± 2	22.0 ± 0.5	7.0 ± 0.2	5.1 ± 0.0	0.27 ± 0.01
5	109 ± 4	197 ± 1	7.0 ± 0.1	81 ± 2	27.0 ± 0.5	9.0 ± 0.2	5.6 ± 0.0	0.33 ± 0.01
6	110 ± 5	210 ± 1	7.0 ± 0.1	87 ± 3	27.0 ± 0.6	7.0 ± 0.2	5.0 ± 0.1	0.27 ± 0.01
7	104 ± 3	197 ± 1	7.0 ± 0.1	85 ± 3	26.0 ± 0.5	7.0 ± 0.3	4.9 ± 0.0	0.27 ± 0.01
8	94 ± 2	185 ± 1	7.0 ± 0.1	82 ± 2	24.0 ± 0.6	7.0 ± 0.2	5.1 ± 0.0	0.27 ± 0.01

drying was close to 40 ± 2 °C. Inlet air humidity was controlled and adjusted by a dehumidifier (Munters, Sollentuna, Sweden).

2.2. Chemical and physical analysis

Solid concentration and free moisture content were calculated by weight loss after drying 1.5 g of the sample mixed with sand in a forced air oven at 105 °C for 7 h (concentrate) or 5 h (powder). Water activity (a_w) was measured in a water activity meter (a_w -meter; Novasina RTD 200/0 and RTD 33, Pfäffikon, Switzerland) at 25 °C.

2.3. Thermodynamic measurements

Concentrate mass flow rate was measured with a flowmeter (Danfoss, Nordborg, Denmark). Temperature (°C), absolute humidity (AH; kg water·kg⁻¹ dry air) and relative humidity (RH; %) were measured using a Hygropalm thermohygrometer (Rotronic, Bassersdorf, Switzerland) for the inlet air and a Hygroflex thermohygrometer (Rotronic, Bassersdorf, Switzerland) for the outlet air [6].

2.4. Statistical analysis

For each powder from the same batch, average and standard deviations of

concentrate mass flow rate, temperature, and absolute and relative humidity were deduced from measurements every 5 s for 30 min during each experiment after 30 min of stabilization.

For each powder from the same batch, three replications of solid concentration, free moisture and water activity determination were performed. The Student's *t* test was used as a guide for pair comparisons of the trial means. Differences among trials that are described subsequently as being significant were determined at $P < 0.05$.

3. RESULTS AND DISCUSSION

The spray-drying parameters (absolute humidity of inlet air, inlet and outlet air temperatures, and concentrate mass flow rate) were adjusted in order to vary the relative humidity of the outlet air as well as the a_w and moisture content of the skim milk powders over 8 experiments. All measurements and calculations are summarized in Table I, according to the experimental conditions. According to Masters [2] and Pisecky [3], decreasing the outlet air temperature increased the moisture content and a_w , shown in the comparison of the results of tests 3 and 4 (Tab. I). However, other results showed that modification of the outlet air temperature by varying the inlet air temperature and concentrate flow rate air (comparison

of the results of tests 1 and 2, and those of tests 6, 7 and 8) did not affect the moisture content or a_w . Other results in Table I also show that keeping the outlet air temperature constant (tests 3 and 6, and tests 4 and 5) significantly affected the moisture content of the skim milk powder after modification of concentrate flow rate, absolute humidity and temperature of the inlet air. These results showed that the direct relationship reported by Masters [2] and Pisecky [3] was not always observed with regard to the spray-drying parameters.

The only constant relationship was that between the relative humidity (RH) of the outlet air and the moisture content or a_w of the skim milk powders. Indeed, when the RH increased, the moisture content and a_w also increased. For example, the outlet air RH was close to $7.0 \pm 0.1\%$, and the moisture content and water activity of skim milk powder were close to $5.1 \pm 0.1\%$ and 0.27 ± 0.01 , respectively, whatever the values of the other drying parameters. When the outlet air RH increased from 7.0 to 9.0% (test 5, Tab. I), the moisture content and a_w increased from 5.1% to 5.6 and 0.28 to 0.33, respectively.

The enthalpic diagram (enthalpy, H , and temperature, T , versus absolute, AH , and relative humidity, RH) can be used to explain these results. All the thermodynamical characteristics of the air from the 8 experiments were placed in this enthalpic diagram (Fig. 1) according to the absolute humidity and the temperature of the outlet air. This figure shows that the points of tests 1, 2, 4, 6, 7 and 8 were on the same RH curve ($\approx 7\%$) and the corresponding moisture contents were close (5.1%). These figures confirmed that the moisture content of the skim milk powder was related to the RH of the outlet air. The relationship between the RH of the outlet air, the moisture content and the a_w of the dairy powders depends on the biochemical composition or the type of the spray-dryer, combined with the residence time

distribution (RTD). Indeed, the value of RH (7%) is only usable for skim milk powder in our pilot plant. Experimental data (not obtained in this study) summarized in Table II showed that increasing the RTD from the pilot-plant spray-dryer to the multi-stage spray-dryer also increased the RH of the outlet air (e.g. from 8 to 20% for skim milk) to obtain consistently a dairy powder at 0.2 of a_w . Increasing the amorphous lactose content decreased the RH of the outlet air (e.g. from 20 to 15% for a multi-stage spray-dryer). Increasing protein content increased the RH of the outlet air (e.g. from 15 to 30% for a compact spray-dryer), explained by the changes in moisture content according to the changes in a_w and biochemical composition.

Figure 2 explains the effects of drying air parameters on outlet air RH and powder moisture content. Using the enthalpic diagram in combination with our results and the findings of Masters [2] and Pisecky [3] showed that the moisture content of dairy powders depends on the a_w , which is related to the RH of the outlet air (RH_2). This RH depends on the temperature (T_2) and absolute humidity (AH_2) of the outlet air. If the AH_2 and T_2 do not change, or increase or decrease simultaneously, the RH_2 , moisture content and the a_w of the dairy powder do not change. If the AH_2 or T_2 do not change simultaneously, the moisture content, RH_2 and a_w of the dairy powder may be modified. As shown in Figure 2, AH_2 depends on the concentrate flow rate and AH of the inlet air after heating (AH_1), which depends on the AH of the inlet air before heating (AH_0) and on the type of heating (direct or indirect). T_2 depends on the concentrate flow rate and the temperature of the inlet air after heating (T_1), which depends on the heat power and the inlet temperature before heating (AH_0).

Figure 3 shows the influence of various factors such as the RH of the outlet air on powder moisture, in agreement with the results of Pisecky [3]. As shown

Figure 1. Enthalpic diagram.
H = enthalpy; T = temperature; AH = absolute humidity; RH = relative humidity.

Table II. Relative humidity (%) for water activity at 0.2 in relation to the type of the spray-dryer and the biochemical composition of the dairy powders.

Powders	Pilot plant spray-dryer	Compact spray-dryer	Multi stage spray-dryer
Skim milk	8 ± 2	15 ± 2	20 ± 2
MPC/I-WPC/I	20 ± 2	30 ± 2	40 ± 2
Whey	4 ± 2	10 ± 2	15 ± 2

MPC/I = milk protein concentrate/isolate; WPC/I = whey protein concentrate/isolate.

in this figure, moisture content depends on the drying air parameters, on the spray-dryer and on the preceding process. Moisture content is influenced by the a_w , which according to this study depends on the RH of the outlet air. The RH is influenced by the AH and temperature of the outlet air, which depend on the concentrate flow

rate, the inlet air temperature and AH before and after heating (Fig. 2). The moisture content is also influenced by the size of the droplet, depending on the type of sprayer and viscosity. The viscosity of the concentrate depends on the heat treatment, temperature, residence time, homogenization, total solid content and biochemical

		ΔAH_0 Type of heating $\Delta [C]$ Flow ΔAH_1		
		$\nearrow AH_2$	$\searrow AH_2$	$= AH_2$
Δ Heat Power Δ Air T_0 $\Delta [C]$ Flow	$\nearrow T_2$	NO CHANGE	DECREASE	DECREASE
	$\searrow T_2$	INCREASE	NO CHANGE	INCREASE
	$= T_2$	INCREASE	DECREASE	NO CHANGE

Figure 2. Effects of drying air parameters on relative humidity of outlet air and powder moisture content.

Δ = variation; AH = absolute humidity; [C] = concentrate; T = temperature; 0 = air before heating; 1 = air after heating; 2 = air after drying.

Figure 3. Influence of various factors on powder moisture content.

RH = relative humidity; H_2O = moisture content; η = viscosity; T = temperature; T_g = glass transition temperature; TS = total solids; $\varnothing$ = size; [C] = concentrate; a_w = water activity.

composition. The Temperature, T , of the droplet and thus the powder are influenced by the temperature and AH of the inlet air and by the RH, AH and temperature of the outlet air. The glass transition temperature (T_g) depends on biochemical composition, a_w and moisture content. The knowledge of T and T_g makes it possible to control agglomeration, stickiness, cakiness and re-hydration.

4. CONCLUSIONS

In conclusion, the experiments reported here show that the outlet air temperature is not always the optimum parameter to affect the moisture content of a dairy powder. The RH of the outlet air is the key parameter to optimize the moisture content and water activity of dairy powders. However, the conclusions are based on a relatively small number of unreplicated trials with a limited range of conditions, and therefore further work is required to confirm the conclusions.

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