Use of a Turbidity Sensor to Characterize Micellar Casein Powder Rehydration: Influence of Some Technological Effects

C. Gaiani,¹ S. Banon,¹ J. Scher,¹ P. Schuck,² and J. Hardy¹

¹Laboratoire de Science et Génie Alimentaires, 2 Avenue de la Forêt de Haye, B.P. 172, 54505 Vandœuvre Les Nancy Cedex, France

²INRA, UMR Sciences et Technologies du lait et de l'œuf, 65, Rue de Saint-Brieuc, 35042 Rennes, France

ABSTRACT

A simplified method to study rehydration of dairy powders was developed for native phosphocaseinate powder. The method involved dispersing powder in a stirred vessel equipped with a turbidity sensor under standardized conditions. The changes of turbidity occurring during powder rehydration highlighted several stages. These stages include particles wetting, and then swelling as the water penetrates into the powder bed, followed by a slow dispersion of the particles. With this tool, some technological effects on powder rehydration were analyzed. Ultrafiltrate incorporation to the casein concentrate before spray drying was found to greatly improve the rehydration, whereas mixing ultrafiltrate powder with casein powder after spray drying did not change the rehydration properties. The effect of granulation on powder rehydration stages was also investigated.

(Key words: turbidity, native phosphocaseinate, rehydration, drying)

Abbreviation key: NPC = native phosphocaseinate.

INTRODUCTION

Rehydration is an essential quality attribute of a dairy powder because most powders are dissolved before use (King, 1966). Some common problems are associated with different stages of the rehydration process: wettability, which is the ability to absorb water; sinkability, which is the ability to sink into the water; dispersibility, which is the ability to disperse in single particles throughout the water; and solubility, which is the ability to dissolve in water (Freudig et al., 1999).

The use of a turbidity sensor to continuously follow a physicochemical process is common especially in crystallography (Crawley et al., 1996; Herri et al., 1999; Moscosa-Santillan et al., 2000) and in environmental studies

(Huang and Cheng, 1996; Spicer et al., 1998). In dairy research, turbidity sensors are also used for monitoring enzymatic milk clotting and acidification (Hardy and Fanni, 1981; Hardy and Scher, 1986; Banon and Hardy, 1991). Except for the report of de Wit and Klarenbeek (1986), the use of a turbidity sensor to study rehydration stages of a dairy powder has not been reported. However, these authors did not take into account the stirring effect or the different stages occurring during rehydration.

In the present work, we used native phosphocaseinate (NPC), which is obtained by tangential membrane microfiltration of milk followed by spray drying of the diafiltrated retentate (Schuck et al., 1994). This powder is an attractive material due to its high protein content and can be used as a relevant model of milk micelles (Famelart et al., 1999). However, the low water transfer during NPC rehydration makes rehydration a time consuming step. By improving its rehydration properties, NPC would then become an attractive material for the dairy and food industry.

Rehydration properties of dairy powders have been investigated often (King, 1966; Baldwin and Sanderson, 1973; Schubert, 1993) but the rehydration process of NPC is less studied (Davenel et al., 1997; Schuck et al., 2002). The objectives of the present study were (i) to validate a turbidity sensor under standardized conditions to continuously follow NPC rehydration, (ii) to use the turbidity profiles obtained to study its rehydration, and (iii) to point out some technological factors (ultrafiltrate incorporation mode and granulation) improving its rehydration.

MATERIALS AND METHODS

NPC and Ultrafiltrate Preparation

The concentrates were supplied by Unité Mixte de Recherche: Sciences et Technologies du lait et de l'œuf (INRA Rennes, France). Native phosphocaseinate was separated from skimmed milk by tangential membrane microfiltration followed by purification through water diafiltration according to Pierre et al. (1992) and Schuck et al. (1994). Ultrafiltrate was obtained by membrane

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Corresponding author: Sylvie Banon; e-mail: Sylvie.Banon@ensaia.inpl-nancy.fr.

Figure 1. Diagrams of the 2 incorporation modes for co-drying (A) and dry-mixing (B) powders. For each method, 2 types of powders were prepared: granulate (G) and nongranulate (NG) powder. CD = Co-drying, DM = dry-mixing, NPC = native phosphocaseinate, UF = ultrafiltrate.

tangential UF of microfiltrate collected during NPC production.

Vacuum Evaporation

Ultrafiltrate was concentrated at Bionov (Rennes, France) in a 2-stage pilot plant falling film vacuum evaporator (GEA, Niro Atomizer, St-Quentin en Yvelines, France) at 530 g/kg of total solid. Evaporation capacity was close to 300 kg/h. The temperature of the first effect was $75 \pm 1^\circ\text{C}$ and the outlet temperature of the concentrate was $50 \pm 1^\circ\text{C}$.

Powder Preparation

Ultrafiltrate was added to NPC according to 2 methods: co-drying and dry-mixing (Figure 1). The co-drying powder was produced by adding the UF solution to NPC concentrate before spray drying. The dry-mixing powder was produced by mixing UF powder with NPC powder after spray drying.

The spray drying of concentrates was performed at Bionov (Rennes, France) in a 3-stage pilot-plant spray dryer (GEA, Niro Atomizer) according to Schuck et al. (1998). The temperature of the concentrate before drying was at 20°C for the UF concentrate (after 4 h of crystallization), and up to $40 \pm 2^\circ\text{C}$ for the other dairy products. 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 kg/h (depending on inlet and outlet

air temperature and airflow). The pressure at the nozzle was 16 MPa. Inlet temperature was between $185 \pm 5^\circ\text{C}$ for UF concentrate and $215 \pm 5^\circ\text{C}$ for other concentrates, integrated fluid bed air temperature was $90^\circ\text{C} \pm 1^\circ\text{C}$ for UF concentrate and $70 \pm 1^\circ\text{C}$ for other concentrates, and outlet temperature was $92 \pm 1^\circ\text{C}$ for UF concentrate and $70 \pm 1^\circ\text{C}$ for other concentrates. Inlet air humidity was controlled and adjusted by a dehumidifier (Munters, Solentuna, Sweden). For each powder, 2 granulations were obtained: nongranulate and granulate powders (Figure 1) by reintroduction of the fine particles after the cyclones at the head of the spray-dryer.

Chemical Analysis

The chemical composition of the powders is reported in Table 1. The amount of total solids was determined by weight loss after drying 1-g samples of powder at 105°C for 5 h. Total protein was determined by Kjeldahl method with a 6.38 conversion factor. Lactose was determined by an enzymatic method using a Enzytec lactose/D-galactose kit (SCIL Diagnostics GmbH, Martinsried, Germany) and fat according to the Röse-Gottlieb method by the FIL 9C:1997 (FIL-IDF, 1997). Ash were measured after incineration at 550°C during 5 h.

NPC Rehydration Study

Experimental set-up. The experiments were carried out in a 2-L vessel equipped with a 4-blade 45° impeller (R 100 impeller: 8 cm diameter) rotating at 400 rpm (Lightnin LabMaster Mixer, Axflow, France). The temperature was kept constant at 24°C using a double-wall jacket vessel. The turbidity sensor was placed 3 cm under the water surface and positioned through the vessel wall to avoid disturbances during stirring.

Turbidity changes accompanying powder rehydration were followed using a turbidity meter (Analite NEP 160, McVan Instruments, Mulgrave, Australia). This apparatus used light in the near-infrared region (860 nm); the incident beam is reflected back at 180° by any particle in suspension in the fluid to a sensitive electronic receptor. The turbidity meter was connected to a measurement system for continuous monitoring (Almemo 8990-8 V₅, Ahlborn, Holzkirchen, Germany). This data logger was coupled with a PC equipped with software (AMR WinControl for Almemo). Data were collected automatically every second for 1000 s then every 5 s. All runs were carried out at least in triplicate.

Samples and rehydration media. Rehydration of NPC was studied in 2 media: distilled water and UF solution. Ultrafiltrate solution was prepared from ultrafiltrate powder dissolved in distilled water 24 h before used. Lactose concentration was calculated to be similar

Table 1. Chemical composition of powders.¹

Powder (g/kg)	Incorporation mode ²	Composition				
		Total solids	Total protein	Lactose	Ash	Fat
G NPC	—	957.9	859.6	4.1	75.3	3.3
NG NPC	—	934.9	856.0	4.2	71.1	3.1
G UF	—	979.0	28.6	780.5	172.9	ND ³
NG UF	—	980.1	22.3	776.0	180.3	ND
G (NPC+UF)	CD	953.8	664.2	217.1	65.8	3.9
(G NPC)+(G UF)	DM	963.3	669.0	214.9	65.7	ND
NG (NPC+UF)	CD	935.2	650.9	207.8	68.8	5.9
(NG NPC)+(NG UF)	DM	951.5	661.8	206.5	67.5	ND

¹G = Granulate; NPC = native phosphocaseinate; NG = nongranulate; UF = ultrafiltrate.

²CD = Co-drying; DM = dry-mixing.

³ND = Not determined.

in the UF solution and in the NPC+UF solution. In the experiments, the concentrations of powder were chosen such that total protein concentration was constant at 5% (wt/vol) for the 6 powders studied. The powder was poured into the rehydration media in less than 3 s, 30 s after starting the monitoring.

Static Light Scattering

Particle size distributions were measured from a laser light diffusion apparatus (Mastersizer S, Malvern Instruments Ltd., Malvern, UK) with a 5-mW He-Ne laser operating at a wavelength of 632.8 nm with a 300RF lens.

The dry particle size distribution was determined using a dry powder feeder attachment and the standard optical model presentation for particles dispersed in air was used.

From the stirred vessel, 0.5 mL of casein suspension was introduced into 100 mL of prefiltered distilled water (Millipore, membrane diameter 0.22 μm) to reach a correct obscuration. The Malvern small volume sample cell used allowed the maintenance of a stable suspension during the measurement under stirring at 2000 rpm. The refractive indices used were 1.57 for casein and 1.33 for water (Strawbridge et al., 1995). The results obtained are average diameters calculated from Mie theory. The criterion selected was $d(50)$, which means that 50% of the particles have a diameter lower than this criterion. Results are the average of 3 replicate experiments carried out on different days.

Statistical Analyses

Statistical analysis was carried out by using the software KyPlot freeware, version 2.0. For comparisons between NPC powder rehydration in water and other re-

hydrations (i.e., co-drying powder in water, dry-mixing powder in water, and NPC powder in ultrafiltrate), a parametric multiple test (Dunnnett test with NPC powder rehydration in water as control) was performed. The significance levels were $***P < 0.001$, $**P < 0.01$, $*P < 0.05$, and $^{NS}P > 0.05$.

RESULTS

Typical Turbidity Profile During NPC Rehydration

Typical profile. Rehydration of 5% granulate NPC powder at 24°C occurred in different stages as shown in Figure 2. Dispersion of powder in the stirred vessel led to a quick increase of turbidity (peak A). Peak A was followed by a decrease of turbidity to a minimum (B) after 250 s. Subsequent to this minimum, an increase

Figure 2. Turbidity typical profile as a function of time during rehydration of 5% granulate native phosphocaseinate at 24°C for 50,000 s. Inset shows the first 2500 s. A = Peak of turbidity corresponding to powder wetting; B = minimum of turbidity corresponding to powder swelling; C = turbidity increased corresponding to powder disintegration and dispersion stage; and D = stabilization of the turbidity corresponding to the end of rehydration.

Table 2. Particle size diameter [d(50), μm] of native phosphocaseinate samples taken in the liquid vessel during rehydration and analyzed by static light scattering ($n = 3$).

Time, s	d(50), μm	SD
0	286	4
80	373	14
190	387	8
360	338	7
900	257	8
1000	212	11
5000	134	8
10,000	22	6
15,000	15	7
40,000	0.42	0.13
45,000	0.36	0.08
50,000	0.36	0.05
60,000	0.36	0.03

in turbidity occurred (phase C) from 250 to 45,000 s. After 45,000 s, the turbidity values stabilized around 13,000 NTU (nephelometric turbidity unit) in phase (D) when a fluid similar to milk is formed.

Particle size determination by static light scattering. During NPC rehydration, samples were taken into the stirred vessel and the d(50) was determined by static light scattering (Table 2). In a first phase, a swelling of the particles was observed from 286 to 387 μm . Following the swelling, a particle size decrease was noted with a d(50) varying from 387 to 0.36 μm . Above 45,000 s, the d(50) stabilized at 0.36 μm , that is, the casein micellar size found by static light scattering (Regnault et al., 2004) and this was related to the end of rehydration.

Turbidity profile and particle size comparison. Dispersion of powder in the vessel led to a quick increase of turbidity and the time taken to reach the first turbidity peak (peak A) was related to the wetting time (Figure 3). Indeed, as can be observed, the turbidity signal presented a peak or a shoulder when all the particles were wetted.

The time taken to reach the minimum (B) of turbidity was related to the time taken to reach the maximal particle size (around 190 s). This time is defined as the swelling time.

As a consequence of the swelling, a disintegration of the wet particles and their progressive dispersion could explain the d(50) decrease and consequently the turbidity increase. Phase C was related then to the powder dispersion.

The time for the powder to fully rehydrate was determined by the time needed to obtain turbidity stabilization (phase D). Indeed, the turbidity stabilization time around 45,000 s obtained with the turbidity profile is related to the particle size stabilization time obtained by static light scattering.

Figure 3. Turbidity profile as a function of time during rehydration of granulate native phosphocaseinate (■) and nongranulate native phosphocaseinate (▲) powder at 24°C for 300 s. The wetting time (T_w) is defined as the time to reach the turbidity peak and/or shoulder.

Effect of Added Ultrafiltrate on the Rehydration Process

No ultrafiltrate added: NPC powder. The rehydration times of granulate and nongranulate NPC powders are summarized and given as a reference in Tables 3 and 4. The wetting and swelling times were both lengthened for nongranulate NPC powder, whereas the time to rehydrate was shortened. The time to rehydrate was around 45,051 s for granulate and 36,138 s for nongranulate powder.

Ultrafiltrate incorporation by co-drying: (NPC+UF) powder. Ultrafiltrate powder incorporation to NPC concentrate before spray drying accelerated almost all the rehydration stages compared with NPC powders. For granulate powder (Table 3), the wetting time was not modified but the swelling time was reduced from 219 to 89 s. Turbidity stabilization was rapidly reached and the time to rehydrate the powder was improved ($P < 0.001$) from 45,051 for NPC to 3126 s for (NPC+UF).

For nongranulate powder (Table 4), no differences ($P > 0.05$) were noticed for the wetting time; the time to rehydrate the powder was reduced ($P < 0.001$) from 36,138 to 1710 s. No swelling time (minimum of turbidity) was observed. The minimum of turbidity may be masked by the quick rehydration of the powder, the swelling stage being mixed up with the wetting stage. To slow down reactions, the rehydration stages of the powder were repeated at lower temperature (10°C; results not shown). All stages were lengthened and the minimum of turbidity was shown to correspond to particle swelling, confirming our hypothesis.

Table 3. Rehydration times (in seconds) obtained for granulate powders compared with the reference powder (granulate native phosphocaseinate powder, G NPC).

Powder ²	UF Incorporation mode	Media	Rehydration parameters, ¹ s					
			Tw		Ts		Tr	
			Mean	SD	Mean	SD	Mean	SD
G NPC	—	Water	110	8	219	9	45,051	1389
G (NPC+UF)	Co-drying	Water	88 ^{NS}	6	89*	11	3126***	215
(G NPC)+(G UF)	Dry-mixing	Water	115 ^{NS}	10	204 ^{NS}	55	44,220 ^{NS}	3200
G NPC	—	UF	118 ^{NS}	15	244 ^{NS}	33	48,021 ^{NS}	4111

¹Tw = Wetting time; Ts = swelling time; Tr = total rehydration time.

²G = Granulate; NPC = native phosphocaseinate; UF = ultrafiltrate.

*** $P < 0.001$; ** $P < 0.01$; * $P < 0.05$; ^{NS}Nonsignificant.

Ultrafiltrate incorporation by dry-mixing: NPC powder + UF powder. Ultrafiltrate powder incorporation after spray-drying did not improve the rehydration times. For granulate powder (Table 3), no differences ($P > 0.05$) were noticed for the wetting, swelling, and time to rehydrate. For nongranulate powders (Table 4), the swelling time was lengthened ($P < 0.01$) from 1045 to 1702 s. The wetting time and the time to rehydrate were not modified ($P > 0.05$).

Rehydration of NPC powder in ultrafiltrate media. Rehydration of NPC in ultrafiltrate media instead of water did not change the rehydration times. For granulate powder (Table 3), the times obtained were not different ($P > 0.05$) from those in water. For nongranulate powders (Table 4), the swelling time was lengthened from 1045 to 1838 s. The wetting time and the time to rehydrate were not modified ($P > 0.05$).

Rehydration Times Obtained by Comparison with a Standard

Standards for the determination of powder wettability and dispersibility are commonly used, but these are often empirical and difficult to perform (American Dry Product

Institute, 2002, and Fédération Internationale Laitière, 1985). The FIL determination of wettability consists of measuring the time necessary for all particles to absorb water in an unstirred vessel. As shown in Table 5, the wetting times obtained in our experiment correlated well with the standard wetting times. The experimental values found were always shorter than the standard values due to the stirring effect. Moreover, it is often impossible with the standard method to determine the wettability for poor-wettable powder such as nongranulate powder (time > 10,000 s) in this study.

A linear relationship was found between the time to rehydrate the powder and the dispersibility (ADPI standard) as shown in Figure 4. When a powder disperses easily (high percentage of dispersibility), the time to rehydrate is shorter.

Currently, no standards are available to determine powder swelling time or rehydration time.

DISCUSSION

NPC Rehydration

From comparison of static light scattering and turbidity measurements, it appears that NPC rehydration

Table 4. Rehydration times (in seconds) obtained for nongranulate powders compared with the reference powder (nongranulate native phosphocaseinate powder, NG NPC).

Powder	UF Incorporation mode	Media	Rehydration parameters, s					
			Tw		Ts		Tr	
			Mean	SD	Mean	SD	Mean	SD
NG NPC	—	Water	207	15	1045	39	36,138	1078
NG (NPC+UF)	Co-drying	Water	221 ^{NS}	22	NO ^{3,***}	0	1710***	110
(NG NPC)+(NG UF)	Dry-mixing	Water	238 ^{NS}	26	1702**	72	35,645 ^{NS}	3536
NG NPC	—	UF	253 ^{NS}	34	1838**	99	38,182 ^{NS}	1997

¹Tw = Wetting time; Ts = swelling time; Tr = total rehydration time.

²G = Granulate; NPC = native phosphocaseinate; UF = ultrafiltrate.

³NO = Not observed.

*** $P < 0.001$; ** $P < 0.01$; * $P < 0.05$; ^{NS}Nonsignificant.

Table 5. Wetting times obtained in our experiment compared with Fédération Internationale Laitière standard method.

Powder ¹	Wetting time, s		Standard wetting time, s	
	Mean	SD	Mean	SD
G NPC	110	8	454	46
G (NPC+UF)	88	6	271	21
(G NPC)+(G UF)	115	10	507	38
NG NPC	207	15	>10,000	—
NG (NPC+UF)	221	22	>10,000	—
(NG NPC)+(NG UF)	238	26	>10,000	—
Skim milk powder	15	2	66	13
Whey protein isolate	148	11	780	67
UF powder	4	0	15	2

¹G = Granulate; NPC = native phosphocaseinate; UF = ultrafiltrate; NG = nongranulate.

occurred in different stages: wetting and swelling followed by very slow dispersion of the particles to finally reach a fluid similar to milk. By viscosimetry measurements, Gaiani et al. (C. Gaiani, S. Banon, J. Scher, P. Schuck, and J. Hardy, unpublished data, 2005) noted also the wetting, swelling, and dispersion stages during NPC rehydration. By a nuclear magnetic resonance method, Davenel et al. (1997) observed 2 stages: water absorption by powder, and solubilization of particles (i.e., swelling and dispersion stages). They found an instantaneous increase of the particle hydration up to 4 g of water/g of powder in less than 3 min after powder pouring; our results were similar as the maximum of swelling was observed after 4 min. The full rehydration process clearly takes time to achieve a stable dispersion. Depending on granulation, the time to rehydrate NPC powder was between 36,000 and 45,000 s. In the study by Povey et al. (1999), casein dissolution kinetics was monitored for hours and days before complete rehydration using an ultrasound apparatus.

Figure 4. Relation between the dispersibility (ADPI standard) and the time to rehydrate the powder obtained in our experiment ($R^2 > 0.97$). G = Granulate, NG = nongranulate, NPC = native phosphocaseinate, UF = ultrafiltrate.

Effect of Ultrafiltrate Addition on Powder Rehydration

For co-drying powders of NPC and ultrafiltrate, the time to rehydrate was the most influenced parameter during rehydration, 14 and 20 times less for granulate and nongranulate powders, respectively. Turbidity stabilization was rapidly reached, demonstrating that the presence of ultrafiltrate favored a short rehydration. By the hygroscopic nature of UF, addition of ultrafiltrate concentrate before spray drying greatly improved water transfer in dried particles as observed by Schuck et al. (2002) when adding NaCl to NPC by co-drying.

Comparison of dry-mixing powders with NPC showed that the wetting and swelling time as well as the time to rehydrate were not improved. For NPC rehydration in ultrafiltrate media, similar times were obtained. As the global composition of dry-mixing NPC + UF powders in water and NPC powders in UF was the same, we hypothesized that during rehydration of dry-mixing powders, UF powder quickly rehydrated first followed by the slow rehydration of NPC powder. Rehydration of NPC in ultrafiltrate media is similar to rehydration of (NPC + UF) dry-mixing powder in water. In previous studies, Davenel et al. (1997) did not find any improvement of NPC rehydration if the incorporation of additives occurred after spray drying. On the other hand, Schuck et al. (2002) established a minor improvement of the rehydration time by dry-mixing mineral salts and NPC.

Granulation Effect

The wettability time was systematically better for granulate particles. Indeed, fast wetting is favored with large particles forming large pores, high porosity, and small contact angle between powder surface and the penetrating water. Freudig et al. (1999) found an optimal size for wettability of around 400 μm , close to the size of the granulate powders used in this study. The wetting stage is often described as a rate-controlling step (Schubert, 1993; Freudig et al., 1999). Baldwin and Sanderson (1973) demonstrated, for whole milk powders, that the most significant improvement in rehydration properties was obtained by improving the wetting properties. On the contrary, in our powders, the dispersion stage seemed to be the rate-controlling step. Indeed, even with a shorter wetting time, a granulate powder was slower to rehydrate than a nongranulate powder.

The time for the powder to fully rehydrate was largely influenced by the granulation, as granulation was shown to systematically slow down the rehydration process. Granulate particles penetrated the water quickly but then dispersed slowly, whereas nongranulate particles presented poor wettability (powder floated on the water surface) but then dispersed more quickly.

CONCLUSIONS

The use of a stir vessel equipped with a turbidity sensor to continuously follow NPC rehydration was validated in this study. The interpretation of the turbidity profiles coupled with static light scattering seems to be a promising tool to appreciate casein powder rehydration and parameters like wetting time, swelling time, and rehydration time.

The experiments reported showed clearly that ultrafiltrate incorporation to NPC concentrate before spray drying improved the rehydration time and that the incorporation mode and the granulation were of importance. There is an ongoing need to develop innovative dairy-based ingredients including native phosphocaseinate. By improving its rehydration properties, NPC could become an attractive material for the food industry. Co-drying of ultrafiltrate solution and NPC concentrate could be one way to extend its application. Further studies applying this approach to other dairy powders are in progress.

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