Lait 82 (2002) 531–539
© INRA, EDP Sciences, 2002
DOI: 10.1051/lait:2002030

Original article

Effect of milk composition on spray-dried high-fat milk powders and their use in chocolate

Kieran KEOGH[a]*, Myra TWOMEY[a],
Brendan O'KENNEDY[a], Donal MULVIHILL[b]

[a] Dairy Products Research Centre, Teagasc, Moorepark, Fermoy, Co. Cork, Ireland
[b] Food Chemistry, Department of Food Science and Technology, University College Cork, Ireland

Abstract – High-fat and skim-milk powders were produced under constant processing conditions from Spring and Autumn herd milks. The influence of the seasonal variation in protein and solid-fat contents at 10 °C of these milks on some properties of the milk powders was determined. The free-fat content and the median particle size of high-fat milk powders were significantly ($P < 0.05$) affected by the protein content and solid-fat content of the milk. There was a curvilinear relationship between the median powder particle size and solid-fat content at 10 °C of the milks. Milk composition did not significantly affect the properties of the skim-milk powders studied. The results showed that it is possible to predict the free-fat content of high-fat milk powders from the protein and solid-fat content of the milks used. Relationships between the spray-dried high-fat milk powder properties and the rheology of milk chocolate at the end of conching and its final hardness were then determined. Eleven spray-dried high-fat milk powders were blended with skim-milk powders to give a 26% fat ingredient for chocolate manufacture. The Casson viscosity (Pas) of the chocolate at 40 °C at the end of conching decreased as the free-fat content increased ($P < 0.01$) and as the powder median particle size increased ($P < 0.05$). The Casson yield value (Pa) at 40 °C at the end of conching decreased ($P < 0.01$) as the solid fat content at 10 °C increased. The hardness of the tempered milk chocolate was significantly reduced as the free-fat content increased ($P < 0.05$) and as the particle size increased ($P < 0.05$). It was concluded that spray-dried high-fat milk powders with different properties can be used to make chocolates with a range of viscosities and yield values for different end uses.

Milk composition / milk powder composition / chocolate rheology

Résumé – **Effet de la composition du lait sur les poudres de lait à teneur élevée en matière grasse séchées par atomisation et leur utilisation dans la fabrication de chocolat.** Des poudres de lait grasses et maigres ont été produites dans des conditions de fabrication constantes à partir de laits de troupeaux de printemps et d'automne. L'influence de la variation saisonnière des teneurs en protéines

---

Communication at the 1st International Symposium on Spray Drying of Milk Products, Rennes, France, October 16–18, 2001.
* Correspondence and reprints
Tel.: 353 25 42235; fax: 353 25 42340; e-mail: kkeogh@moorepark.teagasc.ie

et matière grasse solide à 10 ºC de ces laits sur quelques propriétés des poudres de lait a été déterminée. La teneur en matière grasse libre et la taille des particules (D (v, 0,5)) de poudres de lait à teneur élevée en matière grasse était affectée significativement (*P* > 0,05) par la teneur en protéines et en matière grasse solide du lait. Il y avait une relation curvilinéaire entre la taille des particules (D (v, 0,5)) de la poudre et la teneur en matière grasse solide à 10 ºC des laits. La composition du lait n'affectait pas significativement les propriétés des poudres de lait écrémé étudiées. Les résultats ont montré qu'il est possible de prédire la teneur en matière grasse libre des poudres de lait à teneur élevée en matière grasse à partir de la teneur en protéines et en matière grasse solide des laits utilisés. La relation entre les propriétés des poudres atomisées de lait à teneur élevée en matière grasse et la rhéologie du chocolat au lait à la fin du conchage et sa fermeté finale ont ensuite été déterminées. 11 de ces poudres ont été mélangées avec des poudres de lait écrémé pour obtenir un ingrédient à 26 % de matière grasse pour la fabrication de chocolat. La viscosité Casson (Pas) du chocolat à 40 ºC à la fin du conchage diminuait lorsque la teneur en matière grasse libre augmentait (*P* < 0,01) et lorsque la taille de particules médianes augmentait (*P* < 0,05). La valeur de rendement Casson (Pa) à 40 ºC à la fin du conchage diminuait (*P* < 0,01) lorsque la teneur en matière grasse solide à 10 ºC augmentait. La fermeté du chocolat au lait à température ambiante était réduite de façon significative lorsque la teneur en matière grasse libre augmentait (*P* < 0,05) et lorsque la taille des particules augmentait (*P* < 0,05). On peut en conclure que les poudres atomisées de lait à teneur élevée en matière grasse avec des propriétés différentes peuvent être utilisées pour fabriquer des chocolats avec une gamme de viscosités et de valeurs de rendement permettant des usages différents.

Composition du lait / composition de la poudre de lait / rhéologie du chocolat

1. INTRODUCTION

Milk powder is an important ingredient in chocolate. Roller-dried milk powder is preferred to the spray-dried product due to its high free-fat content (defined as solvent-extractable fat), large lamellar particles and absence of vacuoles [6], but spray drying has become the standard drying process in the dairy industry, for economic reasons. However, the influence of milk composition on milk powder properties is not well documented. The first objective of the current study was to determine the influence of changes in milk composition of Spring and Autumn milks on the properties of spray-dried high-fat powders produced under constant processing conditions [16].

Milk chocolate is a dispersion of cocoa solids, milk solids and sugar in a continuous fat phase of cocoa butter and milk fat. Each component affects the rheology of chocolate at the end of the conching stage, which in turn influences the end-use of the chocolate. The rheological properties of chocolates made with commercial roller-dried, spray-dried regular (high and low free-fat) and skim-milk powders to which milk fat was added were qualitatively associated with properties of the powders such as free-fat, particle size and vacuole volume but the results were confounded since the properties of the chocolate were not related to the individual powder properties [8–10]. Other workers related the properties of chocolate to the particle size of the chocolate after the refining [2, 15]. It is our view, therefore, that the effects of the individual properties of powders on the rheological properties of chocolate have not been quantified.

In the second part of this work [17], 11 powders (excluding 5 confirmatory trials) were selected from the spray-dried high-fat powders already prepared. The primary objective was to determine the influence of variations in the free-fat content, solid fat content at 10 ºC, median particle size and vacuole volume of the spray-dried high-fat powders on the rheology of milk chocolate at conching and on the hardness of the final chocolate. A secondary objective was to develop a model to predict the rheological properties of milk chocolate from the properties of the spray-dried high-fat milk powder used in its manufacture. Ultimately, the intention was to produce spray-dried milk powders with a functionality as close as possible to roller-dried powder.

2. MATERIALS AND METHODS

2.1. Milks and powders

Milks were obtained throughout lactation from Spring and Autumn herds at this centre. Storage, pasteurisation, separation and analyses of the milks, production and analyses of the powders and statistical analyses of the results were as already reported [16]. The ingredients used, production and analyses of the chocolates have also been reported [17] and only a summary is given here. Batches of milk chocolate were manufactured using spray-dried high-fat milk powders with a range of free-fat contents, solid fat contents, particle sizes and vacuole volumes. These high-fat powders (~ 55% fat) were blended with skim-milk powders to give a full-fat (~ 26% fat) powder for evaluation in milk chocolate. The free-fat content, expressed as a percentage of the powder fat [1], was determined by taking 10 g of powder and shaking gently with CCl_4 for 15 min at ambient temperatures. The statistical software package, ECHIP^{TM}, (Hockessin, DE, USA) was used to create a linear design with centre point consisting of eleven trials and five replicated trials. Five confirmatory trials were also carried out to test the goodness of fit of the model developed to predict the chocolate rheological properties.

2.2. Ingredients for chocolate

Cocoa butter (Cadbury Ireland Ltd, Dublin, Ireland), cocoa liquor (Nestlé Rowntree, Mallow, Co. Cork, Ireland), sucrose (Irish Sugar Company, Mallow, Co. Cork, Ireland), milk powders (prepared as described previously, [17]) and lecithin (Topcithin 300, Lucas Meyer, Hamburg, Germany) were used to make milk chocolate.

2.3. Chocolate making

Cocoa liquor (2.8 kg) and 60% of the cocoa butter (2.7 kg) were melted at 60 °C in a Stephan mixer (Stephan Söhne Gmbh, Hameln, Germany). Sugar (9.7 kg) and milk powder (5.5 kg) were then added to the melted fat and mixed for 5 min. The mixture was refined in two passes, which gave a particle size of the same order as refining through a commercial five-roll refiner (Bühler, New Barnet, Herts., UK). The refined mix was then conched in a Frisse conch (Bühler) for 7 h at 60 °C. Lecithin (0.2%) and the remaining cocoa butter (1.8 kg) were added during the final stages of conching. A sample of melted chocolate was taken for rheological assessment at the end of the conching process and the remaining chocolate was stored in an incubator overnight at 60 °C.

On the following day, two 2 kg portions of the chocolate were tempered in a Hillard Chocolate System (Jimsan Enterprises, Inc., West Bridgewater, MA, USA) which was located in a controlled environment (21 °C and 50% RH). The moulded chocolate was placed in a temperature-controlled room at 15 °C for 30 min before de-moulding and the finished bars were wrapped in aluminium foil and stored at 15 °C until analysed.

2.4. Rheological properties of chocolate

Samples of each chocolate were taken at the end of conching for rheological evaluation in a stress-controlled rheometer (Bohlin CVO, Cirencester, UK) with concentric cylinder geometry. All measurements were taken at 40 °C. Samples were pre-sheared for 2.5 min at 18.5 s^{-1} followed by a shear stress sweep from 300 to 30 Pa in 3.5 min.

2.5. Hardness of moulded chocolate

The maximum force (N) required to penetrate the moulded chocolate at 20 °C for a distance of 5 mm with a 10° stainless steel cone travelling at a velocity of 0.5 mm·s^{-1} was measured using an Instron Universal Testing Machine, (Instron, High Wycombe, UK) [11].

3. RESULTS AND DISCUSSION

3.1. Free-fat of high-fat milk powders

The mean values of the properties of the powders (free fat, median particle size, particle span, particle density, bulk density, vacuole volume and interstitial air) manufactured from the Spring and Autumn milks were not significantly different. The data was therefore re-analysed with the fixed variable "herd milk type" omitted. From this analysis, the protein and solid-fat content at 10 °C of the milks significantly affected the free-fat content and median particle size of the powders. Figure 1 shows the effect of milk protein and solid-fat content on the free-fat content of high-fat milk powders. At a solid-fat content at 10 °C of 44.6%, the free-fat content of the powders increased from 57.9 to 91.2 g·100 g^{-1} fat as the protein content of the milk increased from 3.18 to 3.81 g·100 mL^{-1}. The free-fat content of the milk powders increased by 52.1 g·100 g^{-1} fat, as the milk protein content increased from the lowest to the highest value. The milk protein content significantly influenced the free-fat content of the milk powders but only in a linear fashion. There was a curvilinear relationship between the solid-fat content of the milks at

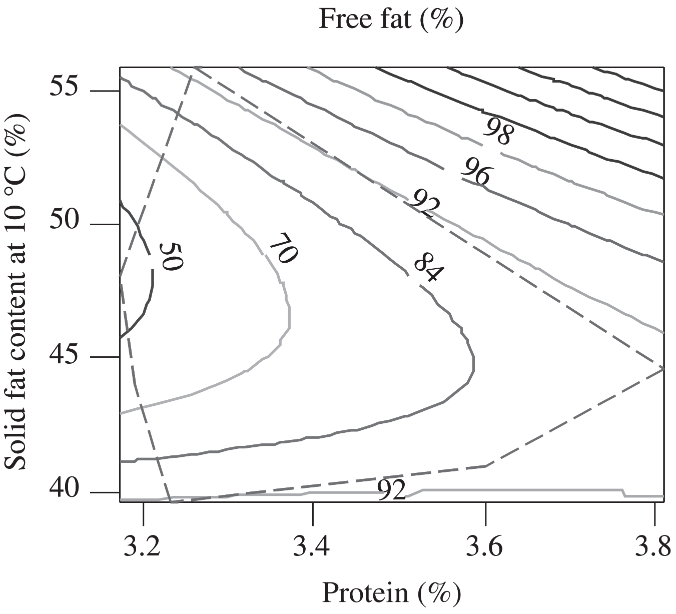

Figure 1. Effect of milk protein content and solid fat content at 10 °C of Spring and Autumn herd milks on the free-fat content of high-fat milk powders. The dotted line represents the design area [16] (with permission).

10 °C and the free-fat content of the powders. The interaction effect (milk protein content × solid-fat content at 10 °C) was not significant. The model equation describing the relationship between the free-fat content of the milk powders and the milk protein and solid fat contents at 10 °C was as follows :

Free-fat content = 2184.3 – 105.9 × MP – 89.6 × SFC + 46.1 × $(MP)^2$ + 0.57 × $(SFC)^2$ + 10.8 × (MP × SFC), F-value = 1.59, $P < 0.05$, P value of SFC < 0.01, P value of $(SFC)^2 < 0.001$, other coefficients not significant.

Using the model, at a constant protein content of 3.25 $g \cdot 100\ g^{-1}$, the free-fat content of the powders decreased from 93.6 $g \cdot 100\ g^{-1}$ fat at a low solid-fat content of 39.6% to 55.6 $g \cdot 100\ g^{-1}$ fat at a solid-fat content of 47.8% and increased again to 94.6 $g \cdot 100\ g^{-1}$ fat at the high solid-fat content 55.9%. The lowest powder free-fat level occurred at a mean solid-fat content of 47.8%. The solid-fat content of the milk affected the free-fat content of the powder to a greater degree than the protein content of the milk.

3.2. Powder particle size of high-fat milk powders

The linear and interaction effects of milk protein content and solid fat content at 10 °C on the powder particle size were not significant. There was a curvilinear relationship between the solid-fat content at 10 °C and the median particle size. The model equation describing the relationship between the median particle size of the milk powders and the milk protein and solid fat contents at 10 °C was as follows:

Median particle size = –166.9 – 157.3 × MP + 22.9 × SFC + 11.0 $(MP)^2$ – 0.26 × $(SFC)^2$ + 0.84 × (MP x SFC), F-value = 1.05, $P < 0.01$, P values of SFC < 0.05, other coefficients not significant.

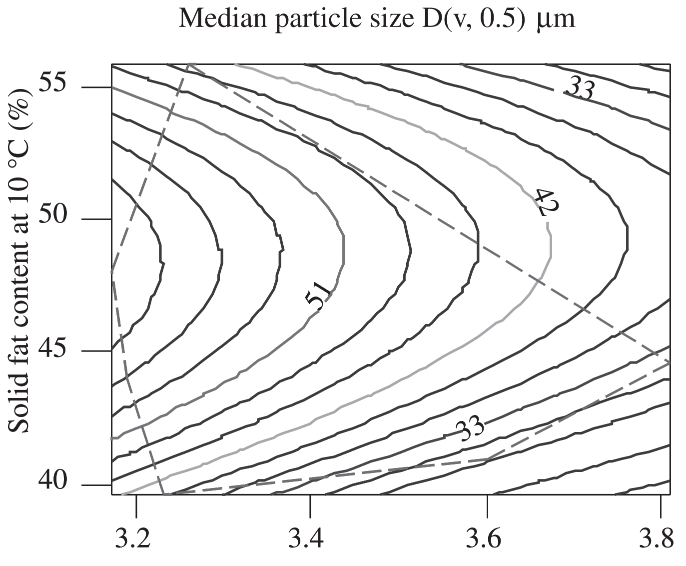

Figure 2. Effect of milk protein content and solid fat content at 10 °C of Spring and Autumn herd milks on the median particle size D(v, 0.5) of high-fat milk powders. The dotted line represents the design area [16] (with permission).

For example, at a constant protein content of 3.25%, the median particle size increased from 38.4 μm at the low solid-fat content of 39.6% to a maximum of 59.0 μm at a solid-fat content of 47.8% and decreased again to 44.3 μm at the high solid-fat content of 55.9% (Fig. 2). The effect of the solid-fat content on the powder particle size was opposite to its effect on the free-fat content of powders. An inverse relationship was found between the free-fat content of the powders and powder particle size ($r = –0.70$, $n = 23$, $P < 0.001$). Buma [5] attributed this relationship to the surface to volume ratio, which is large for small particles, resulting in more exposure of the surfaces to the solvent, than in the case of larger particles.

3.3. Casson viscosity of chocolate

The Casson viscosity of the milk chocolates ($n = 16$) at the end of conching ranged from 1.420 to 2.560 Pas. The Casson viscosity decreased significantly as the free-fat content of the high-fat powders and median particle size [D(v, 0.5)] of the high-fat powders increased (Tab. I). The Casson viscosity of the chocolate decreased ($P < 0.01$) from 2.140 Pas to 1.290 Pas as the free-fat content of the high-fat powders increased from 40 to 96 g·100 g^{-1} fat at the mean values of the other powder variables (solid fat content, 47.5%; particle size, 50 μm; vacuole volume, 2 mL·100 g^{-1} powder). As the level of free-fat in the powder increased, the ratio of disperse phase to continuous phase in the chocolate decreased, which resulted in a decrease in chocolate viscosity [12, 18].

The Casson viscosity of the chocolate also decreased ($P < 0.05$) from 2.350 Pas to 1.080 Pas as the particle size of the high-fat powders increased from 28 to 66 μm at the mean values of the other powder variables (free-fat content, 68 g·100 g^{-1} fat; solid fat content at 10 °C, 47.5%; vacuole volume, 2 mL·100 g^{-1}). As the particle size of a powder increases, the specific surface area decreases, and, therefore, less fat from the continuous phase is required to coat the

Table I. Effects of free-fat content, solid fat content, median particle size and vacuole volume of spray-dried high-fat milk powder on the Casson viscosity, yield value and hardness of chocolate.

	Casson viscosity (Pas)		Casson yield value (Pa)		Hardness (N)	
Term	Effect	Significance[a]	Effect	Significance[a]	Effect	Significance[a]
Constant	1.72		18.1		2.65	
Free fat content (g·100 g^{-1} fat)	–0.84	**	1.1	NS	–0.38	*
Solid fat content at 10 °C (%)	–0.18	NS	–9.8	**	–0.26	NS
Median particle size [D(v, 0.5)] μm	–1.20	*	1.9	NS	–0.61	*
Vacuole volume (mL·100 g^{-1} powder)	–0.47	NS	8.1	*	0.25	NS
Residual standard deviation	0.27		3.6		0.14	
Replicate standard deviation	0.25		2.5		0.13	

[a] Significance: NS = Not significant, * = $P < 0.05$; ** = $P < 0.01$.

powder particles. As a result, the viscosity of the chocolate decreases. It has been shown that suspension Casson viscosity decreases as the particle size of the refined mix increases [1, 15]. In the former work, the increased particle size of the refined mix was due to increased lactose content of the special milk powder used, while in the latter case, it was attributed to the increased particle size of the sugar used. Thus, the effect of the particle size of spray-dried powders is not fully defined in the literature. This is probably because of the difficulty of preparing sufficiently large amounts of high free-fat milk powders of different particle sizes.

3.4. Casson yield value of chocolate

The Casson yield value of the chocolates ($n = 16$) at the end of conching made using the high-fat powders ranged from 12.4 to 25.9 Pa. The Casson yield value of the chocolate decreased as the solid fat content at 10 °C of the high-fat powders increased ($P < 0.01$) and increased as the vacuole volume increased ($P < 0.05$). The free-fat content and the median particle size of the powders did not significantly affect the Casson yield value (Tab. I). The Casson yield value of the chocolate decreased from 23.1 Pa to 13.4 Pa as the solid fat content of the high-fat powders increased from 39.0% to 55.9% at the mean values of the other powder variables (free-fat, 68 $g \cdot 100\ g^{-1}$ fat; particle size, 50 µm; vacuole volume, 2 $mL \cdot 100\ g^{-1}$). The lowest Casson yield values of 12.4–16.0 Pa were obtained in chocolate trials 4, 5, 6 and 10 made using powders 1, 12, 9 and 7 produced in the indoor feeding period, when the solid fat content at 10 °C was high. At a measurement temperature of 40 °C, all the milk fat is liquid, so an associative effect rather than a direct effect of solid fat content at 10 °C of the high-fat milk powders on Casson yield value is probable. This suggests that some other, as yet unknown, component of the milk powder which varies in a similar way to the solid fat content is actually responsible for the changes in the Casson yield value.

As the vacuole volume of the high-fat powders increased from 0 to 3.9 $mL \cdot 100\ g^{-1}$ at the mean values of the other powder variables (free-fat content, 68 $g \cdot 100\ g^{-1}$ fat; solid fat content, 47.5%; particle size, 50 µm), the Casson yield value of the chocolate increased from 14.1 Pa to 22.2 Pa (Tab. I). Powder particles with high vacuole volumes have a greater tendency to shatter during the refining step. The small powder particles, especially fines produced result in a larger surface area for coating by the continuous phase; this probably contributed to the increase in Casson yield value.

3.5. Hardness of finished chocolate

The hardness of the finished chocolate decreased significantly as the free-fat content of the high-fat powder increased ($P < 0.05$) and as the powder particle size increased ($P < 0.05$). Solid fat content at 10 °C and vacuole volume did not significantly affect the hardness of the chocolate (Tab. I). Chocolate is tempered during a carefully controlled thermal regime after conching to ensure that stable fat crystals are formed in the final product [14]. The hardness of chocolate is a useful indicator of good tempering, that is, the degree to which a stable fat crystal network has been formed [13]. In this study, chocolate hardness decreased from 2.84 to 2.46 N as the free-fat content of the high-fat powder increased from 40 to 96 $g \cdot 100\ g^{-1}$ fat at the mean values of the other powder variables (solid fat content, 47.5%; particle size, 50 µm and vacuole volume 2 $mL \cdot 100\ g^{-1}$ powder). As the powder free-fat increased, more of the milk fat contributed to the continuous phase of the chocolate, thus, decreasing the final hardness of the chocolate. Barna et al. [3] also reported the same effect by direct addition of more than 10% milk fat to cocoa butter. The hardness of the chocolate also decreased from 2.98 to 2.32 N as the particle size of the high-fat powder increased at the mean value of the other powder variables (free-fat content, 68 g·100 g^{-1} fat; solid fat content, 47.5%; and vacuole volume 2 mL·100 g^{-1} powder). This decrease may be associated with a greater amount of milk fat released during refining of the larger powder particles [7], thus more milk fat is available to the continuous phase and thus the final chocolate is softer.

4. CONCLUSIONS

The free-fat content and median powder particle size of high-fat powders prepared from Spring and Autumn milks at various times throughout lactation were related to the protein content and solid-fat content at 10 ^{o}C of the milks. As the protein content of the milks increased, the free-fat content of the powders increased linearly. There was a curvilinear relationship between the median powder particle size and the solid-fat content at 10 ^{o}C of the milks. The effect of the solid-fat content of the milks on the free-fat content of the high-fat powders was curvilinear. This curvilinearity may be due to factors associated with late lactation such as the level of protease activity, and an increase in the soluble casein content of milk [4]. In the case of the Autumn milk powders it may be due to a combination of low milk protein content and high solid-fat content in the November-December period when cows were in mid-lactation and indoors. This study also showed [16] that it is possible to predict the free-fat content of high-fat milk powders, made under the same processing conditions, based on the protein and solid-fat content of the milks used. This should make it possible to produce milk powders with specific properties required for use in milk chocolate and other food products.

ACKNOWLEDGMENTS

This project was part-funded by EU Structural funds as part of the Non-Commissioned Food Research Programme. The technical assistance of R. Kennedy and K. Enright is greatly appreciated.

REFERENCES

[1] A/S Niro Atomizer. Determination of free-fat on the surface of milk powder particles, in: Haugaard Sorensen I., Krag J., Pisecky J., Westergaard V. (Eds.), Analytical methods for dry milk products, 4th edition, De Forenede Trykkerier A/S, Copenhagen, Denmark, 1978, p. 46.

[2] Aguilar C.A., Ziegler G.R., Viscosity of molten milk chocolate with lactose from spray-dried whole-milk powders, J. Food Sci. 60 (1995) 120–124.

[3] Barna C.M., Hartel R.W., Metin S., Incorporation of milkfat fractions into milk chocolate, Manuf. Confect. 72 (1992) 107–116.

[4] Barry J.G., Donnelly W.J., Casein compositional studies. 1. The composition of casein from Friesian herd milks, J. Dairy Res. 47 (1980) 71–81.

[5] Buma T.J., Free fat in spray dried whole milk, 1. General introduction and brief review of the literature, Neth. Milk Dairy J. 25 (1971) 33–41.

[6] Campbell L.B., Pavlasek S.J., Dairy products as ingredients in chocolate and confections, Food Technol. 41 (1987) 78–85.

[7] Chevalley J., Chocolate flow properties, in: Beckett S.T. (Ed.), Industrial chocolate manufacture and use, 2nd edition, Blackie Academic and Professional, London, UK, 1994, pp. 139–155.

[8] De Koster P., Funktionalität von Milchprodukten in Schokolade (I). Zucker- und Susswaren Wirtscaft 9 (1989) 278–282.

[9] De Moor H., Huyghebaert A., The use of dried milk products in milk chocolate, Instituut tot Aanmoediging van het Wetenschappelijk Onderzoek in Nijverheid en Landbouw (I.W.O.N.L.), De Craeyerstraat 6, 1050 Brussels, Belgium, 1989.

[10] Dewettinck K., De Moor H., Huyghebaert A., The free-fat content of dried milk products and flow properties of milk chocolate, Milchwissenschaft 51 (1996) 25–28.

[11] Full N.A., Yella Reddy S., Dimick P.S., Ziegler G.R., Physical and sensory properties of milk chocolate formulated with anhydrous milk fat fractions, J. Food Sci. 61 (1996) 1068–1084.

[12] Haylock S.J., Fayerman A.M., Properties of milk chocolate and the effect of milk solids, in: Food Ingredients Asia Conference Proceedings, Expoconsult-Publishers, Maarssen, The Netherlands, 1991, pp. 157–161.

[13] Jeffery M.S., The effect of cocoa butter origin, milk fat and lecithin levels on the temperability of cocoa butter systems, Eur. Food Drink Rev. (1993) 65–71.

[14] Jewell G.G., Bradford L., Considerations in chocolate formulations, Manuf. Confect. 61 (1981) 26–30.

[15] Mongia G., Ziegler G.R., The role of particle size distribution of suspended solids in defining the flow properties of milk chocolate, Int. J. Food Prop. 3 (2000) 137–147.

[16] Twomey M., Keogh M.K., O'Kennedy B.T., Auty M., Mulvihill D.M., Effect of milk composition on selected properties of spray-dried high-fat and skim-milk powders, Irish J. Agric. Food Res. 39 (2000) 79–94.

[17] Twomey M., Keogh M.K., O'Kennedy B.T., Mulvihill D.M., Seasonal effects of some milk powder characteristics on the rheology of milk chocolate, Irish J. Agric. Food Res. 40 (2002) accepted.

[18] Verhey J.G., Physical properties of dried milk in relation to chocolate manufacture, Neth. Milk Dairy J. 40 (1986) 261–268.

To access this journal online:
www.edpsciences.org