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Original article

Effect of the particle size of spray-dried milk powder on some properties of chocolate

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Abstract – Spray-dried milk powders have a median particle size of 30–80 µm. Roller-dried powder particles, which are larger (about 150 µm), are preferred for chocolate making. Spray-drying variables were therefore studied to produce larger powder particles for chocolate. The particle size of the spray-dried powders increased with increasing size of the spray nozzle orifice used. The free-fat content, vacuole volume and insolubility index of the powders increased at the above normal air outlet temperatures used to dry the large powder particles. Vacuole volume and moisture contents typical for spray-dried powders were obtainable at air outlet temperatures up to 90 °C. The particle size of the chocolate mix after refining and the Casson yield value of the chocolate after conching reached minimum values using spray-dried powders with median particle size values of 132–162 µm. These minimum values in the chocolates were also correlated with higher contents of free-fat and lower vacuole volumes in the powders. The spray-dried powders produced good quality British-style chocolates, where the particle size after refining is conventionally <26 µm, but not continental chocolates, where the particle size should be <20 µm.

Spray-drying / milk powder / particle size / chocolate rheology

Résumé – Effets de la taille des particules de la poudre de lait sur les propriétés du chocolat. La taille moyenne des particules des poudres de lait séchées par atomisation est, en génerale, comprise entre 30 et 80 µm. En chocolaterie, on préfère l'emploi de poudres de lait séché sur rouleaux (taille des particules de l'ordre de 150 µm). Ainsi la modification des conditions de séchage par atomisation, pour obtenir des particules de taille plus élevée, a été étudiée pour cet usage. La taille des particules des poudres atomisées augmentait avec l'accroissement du diamètre des buses utilisées. La teneur en matière grasse libre, le volume vacuolaire et l'indice d'insoluble des poudres étaient accrus avec l'élévation de la température de l'air de sortie. Le volume vacuolaire ainsi que la teneur en eau recherchés pour l'utilisation en chocolaterie étaient obtenus pour une température d'air de sortie de 90 °C. Les poudres ainsi obtenues, avaient des particules de taille moyenne comprise entre 132 et 162 µm, une teneur élevée en matière grasse libre et un faible volume vacuolaire. Ces poudres satisfaisaient ainsi aux valeurs minimales requises pour le « mix » chocolat après raffinage et pour la valeur limite d'écoulement selon Casson après conchage. Elles permettaient l'obtention de chocolat de type britannique de bonne qualité (où il est recherché des particules de taille <26 µm, après raffinage et conchage), mais elles ne convenaient pas pour l'utilisation en fabrication de chocolats de type européen (où il est recherché des particules de taille <20 µm).

Poudre de lait / séchage / atomisation / taille de particule / rhéologie du chocolat

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1. INTRODUCTION

Roller-dried milk powder containing 26 g fat·100 g^{-1} powder is preferentially used in milk chocolate because of its high content of free-fat (>90 g·100 g^{-1} fat is solvent extractable), large lamellar-shaped particles (median particle size is about 150 μm) and absence of vacuoles [8]. Increasing free-fat in milk powders reduces the ratio of the dispersed phase to the continuous fat phase in chocolate, thus beneficially reducing the values of the Casson viscosity and yield value of the molten chocolate at the end of conching [12]. Conversely, a smaller particle size in chocolate after refining results in higher rheological parameters in the chocolate at the end of conching [1]. This is because smaller particles have larger surface areas, which adsorb larger amounts of the continuous fat phase. Vacuoles in spray-dried milk powder particles increase the volume to mass ratio. The presence of vacuoles also leads to shattering of the powder particles during refining of the chocolate mix, which results in the formation of small particles or fines and higher rheological values in the subsequent molten chocolate [9].

The production of milk powder by spray-drying is now the norm in the milk powder industry, as the process is more efficient in terms of energy and scale. However, spray-dried milk powders containing 26 g fat·100 g^{-1} powder have <10 g free-fat·100 g^{-1} fat, have spherical shaped particles (typically median particle size 30–80 μm) and contain up to 10 mL vacuole volume·100 g^{-1} powder. These properties make them less suitable for chocolate manufacture [8]. Recent work [12] demonstrated the positive effects of using blends of high-fat milk powder and skim milk powder in chocolate. High-fat (~56 g fat·100 g^{-1}) milk powders have higher levels of free-fat which, even when blended with skim milk powder to the standard fat content result in a reduced particle size of the chocolate mix after refining. It seems that chocolate mixes containing high fat powder/skim milk powder blends are more easily refined. Significantly, the smaller particle size of the refined mix was associated with a decrease rather than an increase in the Casson viscosity of the molten chocolate at the end of conching, presumably because the increased free-fat outweighed the effects of the smaller particle size of the chocolate mix. Higher fat powders also had lower vacuole volumes due to the foam depressing effects of fat, which resulted in a lower Casson yield value of the molten chocolates. The free-fat content of the milk powders was also increased by ultrafiltration of the milks to increase the protein content relative to lactose, but the increased protein counteracted the effect of increased free-fat on the Casson viscosity. Increasing the viscosity of the milk concentrate increased the particle size of the milk powders, but the associated increase in moisture levels of the powders counteracted the expected decrease in the Casson yield value of the molten chocolates. A small decrease in the Casson viscosity of the molten chocolates (~0.3 Pas, $P < 0.05$) was however achieved.

The effect of the particle size of milk powders with similar moisture contents and vacuole volumes on the properties of chocolate should therefore be investigated. It was shown [14] that the median particle size of high-fat powders (55 g fat·100 g^{-1}) produced with a 2-fluid nozzle/Anhydro pilot-scale dryer varied seasonally from 28 to 66 μm. Using blends of the high-fat and skim milk powders to give a standard powder (26 g fat·100 g^{-1}), the Casson viscosity decreased by 1.20 Pas ($P < 0.05$) as the particle size increased at the mean value of the other properties of the high-fat powder [15]. It is still necessary to produce standard spray-dried powders with particles as large as roller-dried powder (~150 μm) for evaluation in chocolate. This is the main objective of the current work, but high levels of free-fat and low levels of vacuole volume, typical of roller-dried powders, are not expected. Up to now, the milk powder industry has not provided a spray-dried milk powder with a particle size optimised

Table I. Characteristics of nozzles.

Size (cat. no.)	Orifice size (diameter, mm)	Core size (cat. no.)	Number of nozzles used
74	0.57	20	3
60	1.01	20	2
56	1.19	21	2
50	1.77	21	1
49	1.85	27	1
48	1.93	27	1
44	2.18	27	1

specifically for the chocolate industry. The reason for this was partly that the production of large particle size powders is outside the norm in standard spray-dried powders, but still achievable.

The composition and processing of chocolate also has major effects on its properties, especially the fat content, the refining conditions (gap width and roller pressure), conching time and tempering conditions. In the current work, the chocolate-making recipe was as before [12] but the refining conditions were slightly altered.

2. MATERIALS AND METHODS

2.1. Milk standardisation

Whole milk from a local manufacturing plant supplied from mainly Spring-calving herds was used. Storage, pasteurisation, separation and analyses of the milks were as already reported [14] and a short summary is given here. The whole milk was standardised by separating a portion of skim milk, which was used to adjust the milk to give a milk powder composition equivalent to 26 g fat·100 g^{-1} powder. The standardised milk was then heated at 97.5 °C × 2 min in the pasteuriser section of an evaporator to increase the heat stability of the concentrated milk and subsequently cooled to 45 °C.

2.2. Milk concentration and drying

The heat-treated milk was then concentrated to 39–55 g solids·100 g^{-1} in a pilot-scale Niro 3-effect falling film evaporator with a water evaporation capacity of 800 kg·h^{-1}. The concentrate was pumped by positive displacement pump from the evaporator through a scraped-surface heat exchanger, where the temperature of the concentrate was raised to 65 °C. From here, the concentrate was delivered by means of a high-pressure pump to the atomiser of a Tall-Form dryer, model TDF-20 (Niro A/S, Copenhagen, Denmark). The pilot scale dryer had a nominal water evaporation capacity of 70–100 kg·h^{-1}. The concentrate was spray atomised using nozzles of various orifice size (Tab. I), as supplied by Spraying Systems Ltd., Farnham, UK. Nozzle size numbers 50 to 44 were larger than conventionally used, in order to obtain large spray-dried milk powder particles in the range 100–200 μm for the manufacture of chocolate. The temperature of the inlet air applied to the Tall-Form dryer of 180 °C is typical but the temperatures of the outlet air of 80–115 °C are higher than the conventional range of 68–75 °C for this dryer. The powders were stored in sealed bags at a constant temperature of 15 °C.

2.3. Compositional analysis of milk concentrates and powders

The total solids of the milk concentrates were determined by microwave heating, using a CEM LabWave 9000 (CEM Corp., Matthews, NC, USA). The moisture content of the milk powders was determined by the oven drying method [2]. The free-fat content was determined by the Niro method [3] using CCl_4 as solvent with constant shaking over 15 min. All tests were carried out in duplicate and the mean calculated.

2.4. Viscosity of concentrates

A strain-controlled Bohlin VOR rheometer (Bohlin, Cirencester, UK) with concentric cylinder geometry was used. Samples of milk concentrate were taken from the evaporator and 13.25 g was weighed into a C25 cup. The samples were subjected to a pre-shear at 461 s^{-1} for 300 s at 45 °C, followed by an up and down shear rate sweep from 1.16–461 s^{-1} and the mean values of each sweep taken. The concentrate viscosity profiles obeyed the power law model most closely ($r > 0.998$), that is:

$$\sigma = k\dot{\gamma}^n$$

where σ is the shear stress (Pa), $\dot{\gamma}$ is the rate of shear (s^{-1}), the exponent n is the power law factor and k is the consistency index (Pas^{n-1}) or the viscosity (Pas^n) at $\dot{\gamma} = 1\ s^{-1}$.

2.5. Particle size of milk powders

The size of the particles in the milk powders was measured using a Malvern Mastersizer X (Malvern Instruments, Malvern, UK) fitted with an MSX15 small volume sample presentation unit. The instrument uses an approximation of the Mie-scattering theory to determine particle size, which utilises the refractive index of the dispersed phase and its absorption. A relative refractive index of 1.095 and an absorption value of 0.1 were used in the calculations. A 2 mW He-Ne laser beam (633 nm) and a 300 RF lens (size range 0.05 to 879 μm) were used for the measurements. The powders were suspended in propan-2-ol and sonicated (Sonicator, Hielscherr, Germany, model UP 200H) for 2 min before each determination. The results are expressed as the volume weighted median diameter D(v, 0.5).

2.6. Vacuole volume

The particle density ($g \cdot mL^{-1}$) was first calculated by measuring the air-free volume of a known weight of powder using a pycnometer (AccuPyc 1330, Micromeritics, Norcross, GA, USA). The pycnometer determined the density of milk powder by measuring the pressure change of helium in a calibrated volume. The vacuole volume was calculated from the difference between the solids and particle densities [4].

2.7. Powder insolubility index

The powder insolubility index was measured after reconstituting 13.0 g powder in 100 mL water at 24 °C [10].

2.8. Statistical analysis

The properties of the milk concentrates, powders and chocolates were correlated using simple linear regression with intercept using the statistical functions in Microsoft Excel software. The significance levels were based on the analysis of variance using a 2-tailed Student's t-test [5].

2.9. Chocolate ingredients

The milk chocolate recipe had a 25 $g \cdot 100\ g^{-1}$ incorporation rate of milk powder, mean total fat content of 30.9 $g \cdot 100\ g^{-1}$ and moisture content of 1.2 $g \cdot 100\ g^{-1}$. The following ingredients were used in the manufacture of milk chocolates as previously described [12]:

- Cocoa butter (Cadbury Ireland Ltd, Dublin);
- Cocoa liquor (Nestlé Rowntree, Mallow, Co. Cork);
- Sucrose (Irish Sugar Company, Mallow, Co. Cork);
- Milk powders (prepared as described above);
- Lecithin (Topcithin 300, Lucas Meyer, Hamburg, Germany).

2.10. Chocolate process

The process remained unchanged from before [12], except the gap widths were reduced and pressure increased between the rollers of the three-roll refiner used (Bühler, New Barnet, Herts., UK) as follows: to 280 μm and 3.0 MPa between each pair of rollers in the first pass through the refiner and to 70 μm and 3.0 MPa in the second pass. These settings gave a particle size of 25 μm in the chocolate after refining when a spray-dried milk powder containing 26 g fat·100 g^{-1} with a median particle size of 132 μm was used. For the roller-dried powders, a wider gap width of 140 μm at the same pressure was used in the second pass to give a particle size after refining of <20 μm. The refined mix (7 kg) was then conched in the Lipp conch (Lipp Mischtechnik, Mannheim, Germany) for 7 h at 60 °C. Lecithin (0.2 g·100 g^{-1}) and the remaining cocoa butter were added after 5 h of conching. A sample of molten chocolate was taken for rheological assessment at the end of the conching stage and the remaining chocolate was stored in an incubator overnight at 50 °C.

On the following day, two 2 kg portions of the chocolate were tempered as before. 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.

2.11. Rheological properties of milk chocolate

Samples of the molten chocolate were taken at the end of conching for rheological evaluation in a strain-controlled Bohlin VOR rheometer (Bohlin, Cirencester, UK) with concentric cylinder geometry. All measurements were made at 40 °C. Samples were pre-sheared for 2.5 min at 18.5 s^{-1} followed by a shear rate sweep from 60 to 1 s^{-1} in 3.5 min. The data was then used to calculate the Casson viscosity (η_{CV}) and Casson yield value (σ_{CA}) [13] using the following equation:

$$\sqrt{\sigma} = \sqrt{\sigma_{CA}} + \sqrt{\eta_{CV}}\sqrt{\dot{\gamma}}$$

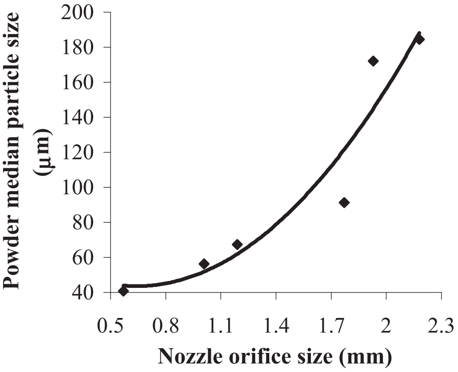

Figure 1. Effect of nozzle orifice size on powder median particle size.

where σ is the shear stress (Pa), σ_{CA} is the Casson yield value (Pa), η_{CV} is the Casson viscosity (Pas) and $\dot{\gamma}$ is the shear rate (s^{-1}).

3. RESULTS AND DISCUSSION

3.1. Effect of nozzle orifice size on milk powder particle size

Powders containing 26 g fat·100 g^{-1} powder (nominal) were first produced with 6 nozzles varying in size as outlined in Table I. Data from milk concentrates with 45.9–47.3 g solids·100 g^{-1} and dryer air outlet temperatures of 74 or 81 °C were used (Tab. II). Figure 1 shows that the median particle size [D(v, 0.5)] of the powders increased curvilinearly from 41 to 185 μm ($r = 0.953$, $n = 6$, $P < 0.01$) as the nozzle orifice size increased. The model indicated that nozzle sizes 50, 48 and 44 (1.77–2.18 mm) produced powder particles in the size range of interest for chocolate (130–185 μm). It has already been established [11] that milk powder particle size increases with the viscosity of the milk concentrate used.

3.2. Effect of air outlet temperature on some milk powder properties

Powders were then produced using air outlet temperatures of 74–115 °C (Tab. II).

Table II. Properties of concentrates and powders.

Nozzle	Air outlet	Milk concentrate		Milk powder				
diameter	temperature	Total solids	Viscosity	Moisture	Free fat	Particle size	Vac. volume	Insolubility index
(mm)	(°C)	($g\cdot 100\ g^{-1}$)	$(mPas)^{n-1}$	($g\cdot 100\ g^{-1}$)	($g\cdot 100\ g^{-1}$ fat)	[D(v, 0.5) µm]	($mL\cdot 100\ g^{-1}$)	($mL\cdot 50 mL^{-1}$)
Figure 1 (no. of powders = 6)								
0.57	74	45.9	153	2.4	12.9	40.8	6.00	0.10
1.01	74	45.9	153	2.9	6.3	56.4	5.50	0.10
1.19	74	45.9	153	3.5	7.3	67.5	4.40	0.10
1.77	74	47.0	ND	3.9	7.1	91.2	5.80	0.40
1.93	81	47.0	536	4.7	2.4	172.1	6.50	0.10
2.18	81	47.3	303	5.1	3.0	184.6	7.90	1.10
Figures 2a, 2b, 2c, 2d (no. of powders = 9)								
1.77	74	47.0	ND	3.9	7.1	91.2	5.75	0.40
1.77	81	47.0	ND	2.3	4.2	64.5	7.88	2.00
2.18	81	52.3	303	5.1	3.0	199.6	7.89	1.10
1.77	91	48.4	501	2.1	20.3	75.4	10.30	2.50
2.18	91	52.3	303	4.1	4.6	203.3	10.78	3.00
1.77	100	48.4	501	1.7	11.9	78.5	15.84	5.50
2.18	100	52.3	303	3.5	6.3	203.9	15.05	5.50
1.77	115	48.4	501	1.2	19.9	96.6	28.12	6.00
2.18	115	52.3	303	3.0	10.2	218.2	25.90	7.00

In a first series, nozzle size 50 (1.77 mm) and milk concentrates containing 47.0 or 48.4 g solids·100 g^{-1} were used. A second series was also produced using nozzle size 44 (2.18 mm) and a milk concentrate containing 52.3 g solids·100 g^{-1}. Figure 2a shows the effects of air outlet temperature on vacuole volume, where the modeled data were coincidental for both nozzles. The vacuole volume increased curvilinearly with air outlet temperature ($r = 0.9964$, $n = 9$, $P < 0.001$), reaching 10.2 mL·100 g^{-1} powder at air outlet temperature 91 °C. Figure 2b shows the effects of air outlet temperature on free-fat, where the data were nozzle-dependant. The free-fat increased linearly with air outlet temperature for nozzle size 50 (1.77 mm) ($r = 0.900$, $n = 5$, $P < 0.05$) and for nozzle size 44 (2.18 mm) ($r = 0.993$, $n = 4$, $P < 0.01$). The free-fat levels were higher for the smaller nozzle size 50, because of the smaller powder particle size. The model indicates that free-fat levels of 11 and 5 g·100 g^{-1} fat were obtained at outlet temperature 91 °C using nozzles 50 and 44, respectively. It is known that free-fat increases with air outlet temperature between 70 and 105 °C [6] and decreases with concentrate total solids [7] using a rotary atomiser.

Figure 2c shows the effects of air outlet temperature on powder moisture, where the data were nozzle-dependant. The moisture content of the powders decreased curvilinearly with air outlet temperature for nozzle size 50 (1.77 mm) ($r = 0.963$, $n = 5$, $P < 0.01$) and for nozzle size 44 (2.18 mm) ($r = 0.9999$, $n = 4$, $P < 0.001$). As expected,

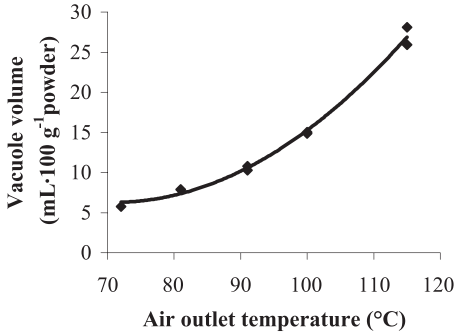

Figure 2a. Effect of air outlet temperature on vacuole volume of powder.

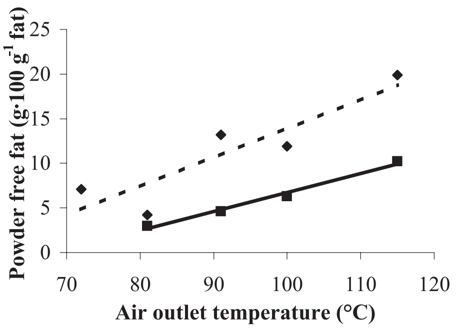

Figure 2b. Effect of air outlet temperature on powder free fat using spray-dryer nozzle size 1.85 mm (---◆---) and nozzle size 2.17 mm (—■—).

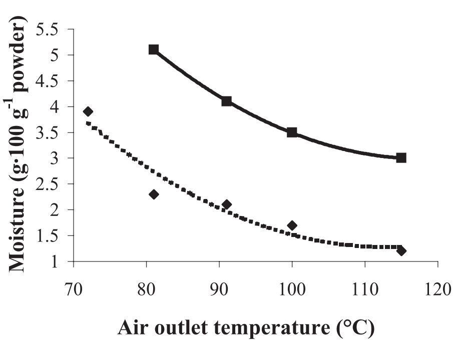

Figure 2c. Effect of air outlet temperature on moisture of powder using spray-dryer nozzle size 1.85 mm (---◆---) and nozzle size 2.17 mm (—■—).

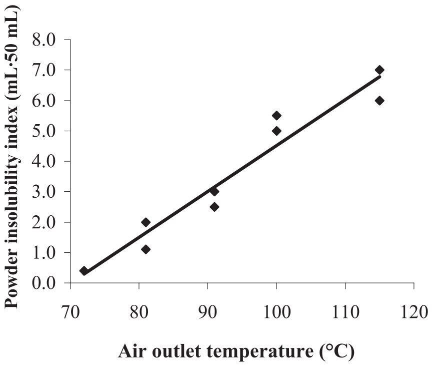

Figure 2d. Effect of air outlet temperature on powder insolubility index.

the moisture levels were higher when the larger nozzle size was used but decreased with air outlet temperature with both nozzles [6]. The model indicates that moisture levels of <3.0 g·100 g^{-1} powder are obtained at outlet temperatures above 76 °C and <2.0 g·100 g^{-1} at outlet temperatures above 87 °C using nozzle size 50.

Figure 2d shows the effects of air outlet temperature on the insolubility index, where the modeled data were coincidental for both nozzles. The insolubility index of the powders increased linearly with air outlet temperature ($r = 0.968$, $n = 9$, $P < 0.001$). The model indicates that an insolubility index of 3.2 mL·50 mL^{-1} reconstituted powder was obtained at an air outlet temperature of 91 °C using either nozzle size.

3.3. Effect of milk powder particle size on some chocolate properties

A series of powders ranging in median particle size from 76.8 to 196.2 μm was used for making chocolates (Tab. III). Differences in the powder particle size were

Table III. Properties of milk powders and chocolates.

Nozzle diameter (mm)	Milk concentrate total solids ($g\cdot100\ g^{-1}$)	Powder Moisture ($g\cdot100\ g^{-1}$)	Powder Free fat ($g\cdot100\ g^{-1}$ fat)	Powder Particle size [D(v, 0.5) μm]	Powder Vac. volume ($mL\cdot100\ g^{-1}$)	Chocolate Particle size after refining (μm)	Chocolate Viscosity (Pas^{n-1})	Chocolate Yield value (Pa)
Pilot-scale spray-dried								
1.77	47.0	2.1	6.1	76.8	12.2	31	1.70	25.2
1.85	41.0	3.1	12.8	109.5	7.6	29	1.70	20.9
1.77	39.2	3.8	14.1	132.0	7.8	25	1.72	20.5
1.85	47.6	3.8	16.4	143.5	7.7	24	1.63	19.3
1.85	47.6	3.8	16.4	143.5	7.7	24	1.54	18.8
1.93	41.1	4.5	9.6	161.8	7.5	25	1.75	22.5
1.85	51.1	3.7	6.6	168.1	11.0	27	1.66	23.0
1.93	53.6	3.8	5.0	196.2	10.7	29	1.63	21.6
Commercial roller-dried								
NA	NA	2.8	74.6	152.9	0.0	18	1.53	22.8
NA	NA	2.9	84.2	166.3	0.0	17	1.65	23.8

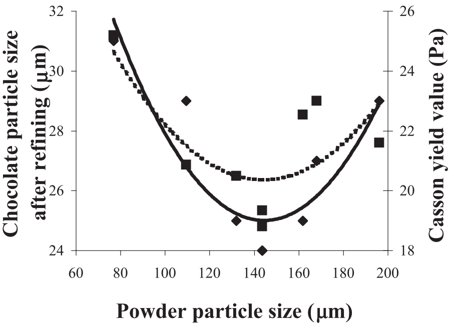

Figure 3. Effect of powder particle size on chocolate particle size after refining (—◆—) and on Casson yield value after conching (--- ■ ---).

achieved using nozzle sizes 50, 49 and 48 and milk concentrates varying in total solids from 39.2 to 54.6 $g\cdot100\ g^{-1}$. An air outlet temperature of 91 °C was used in order to produce powders with vacuole volume and moisture values as close as possible. Nevertheless, values varied slightly because of the effects of nozzle size and concentrate total solids, as shown in Table III. Figure 3 shows the effect of the powder particle size on the particle size after refining the chocolate mix and on the yield value of the molten chocolates after conching. The particle size after refining reached minimum values of 24–25 μm using powders with median particle size values of 132, 143, 143 and 162 μm ($r = 0.904$, $n = 8$, $P < 0.01$). This is commercially important, as grittiness is detectable in chocolates with particle size values >26 μm. This is the standard particle size in British and some US chocolates, though continental European and some US chocolates have a lower particle size of <20 μm. A particle size after refining of <24 μm could not be achieved using these spray-dried powders, as the refining conditions used were maximum. By contrast, two commercial roller-dried powders gave chocolates with lower particle sizes after refining of 17 and 18 μm, but with viscosity and yield values similar to chocolates made with spray-dried powders (Tab. III) using our intermediate refining conditions. The particle size after refining was also inversely related to the free-fat content ($r = -0.737$, $n = 8$, $P < 0.01$), moisture content ($r = -0.766$, $n = 8$, $P < 0.01$) and directly to the vacuole volume of these spray-dried milk powders ($r = 0.741$, $n = 8$, $P < 0.01$). This confirms [12] that milk powders with higher free-fat contents produce chocolates with smaller particle size after refining. There was no evidence from the particle size distribution of an increase in fines due to fracturing of the larger powder particles. For the first time, the results suggest that as the moisture content of the milk powders increased, the chocolate mix was more easily refined, possibly due to increasing softness of the powder particles. The Casson viscosity was not affected by any of the variables. However, the Casson yield value reached a minimum value of 19 Pa using a powder with median particle size 143 µm ($r = 0.737$, $n = 8$, $P < 0.05$). The reason for the increase in Casson yield value above a milk powder particle size of 143 µm could be due to the lower free-fat ($P < 0.01$) and higher vacuole volume of these powders ($P < 0.05$, Tab. III).

4. CONCLUSIONS

The median particle size of spray-dried milk powders containing 26 g fat·100 g^{-1} powder can readily be increased up to 200 µm by increasing the nozzle size of the concentrate used for drying. The air outlet temperature must then be increased to about 90 °C using nozzle size 50 (1.77 mm) to reduce the moisture contents of the powder to 2.0%. Under these conditions, the vacuole volume (10 mL·100 g^{-1} powder) and free-fat content (11 g·100 g^{-1} fat) of the powders does not match those of roller-dried powders. However, the insolubility index increased to 3.2 mL·50 mL^{-1} reconstituted powder, making the spray-dried powder more like roller-dried powder. The particle size of the chocolate mix after refining and the Casson yield value of the chocolate after conching reached minimum values using a powder with median particle size 143 µm. This was also associated with the higher free-fat content and lower vacuole volume of these powders.

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