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EFFECT OF MANUFACTURING PROCESS ON THE MICROSTRUCTURAL AND RHEOLOGICAL PROPERTIES OF MILK CHOCOLATE

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Abstract

The effect of different process steps on microstructural, rheological and visual properties of milk chocolate was studied. Each process step affects the microstructural characteristics of milk chocolate, involving modifications on its macroscopic properties, such as rheological attributes. Milk chocolate samples were obtained at each phase of the manufacture process: mixing, pre-refining, refining, conching and tempering. Microstructural properties (network structure and particle size) and rheological parameters (yield stress, apparent viscosity, thixotropy, G' and G'') were evaluated by using respectively an environmental scanning electron microscope (ESEM), and a controlled strain–stress rheometer. Colorimetric analyses (L*, h° and C*) were also performed. ESEM analysis revealed important changes in the network structure during process, with a reduction in particle size and an increase in the voids between aggregates, from the mixing to the refining step. Moreover, an increase of all rheological analyzed parameters from mixed sample to the refined one was found. Samples obtained from the conching and tempering steps were characterized by the lowest statistically significantly values of all rheological parameters. This could be related to the changes in the structure aggregation evidenced by ESEM analysis. From colour results, the samples with the finest particles appeared lighter and more saturated than those with coarse particles.

Keywords: Milk Chocolate; Manufacture steps; Microstructure; Rheology; Appearance.

1. Introduction

Milk chocolate is a complex rheological system having solid particles (cocoa, milk powder and sugar) dispersed in cocoa butter, which represent the fat phase (Pajin et al., 2013). Milk powder is one of the main ingredient of milk chocolate (being used at about 20% w/w in the formulation); this ingredient affects the sensory characteristics of the final product, the processing behaviour and the rheological properties of the fluid chocolate mass (Franke and Heilzmann, 2008; Taylor et al., 2009). The determination of the rheological properties of chocolate is important during manufacturing processes in order to obtain high quality products with well-defined characteristics (Servais et al., 2002; Gonçalves and Lannes, 2010). The rheological characteristics of milk chocolate (pseudoplastic flow with yield stress, apparent viscosity, thixotropy and viscoelasticity) are in fact influenced by formulation (amount of fat, amount and type of emulsifiers) as well as by processing steps (mixing, pre-refining, refining, conching and tempering) (Tscheuschner and Wunsche, 1979; Vavreck, 2004; Schantz and Rohm, 2005). The processing of milk chocolate involves, during each single step (mixing, pre-refining, refining, conching and tempering), modifications in its final quality and attributes, influencing in a strong way the microstructure of the product (aggregation, de-aggregation, reduction of particle size, immobilization of cocoa butter, etc.) (Afoakwa et al., 2009a; Aguilera et al., 2000). In particular, milk powder with its own physical characteristics and inner porosity may have a significant impact on the chocolate processing conditions and on the physical and organoleptic properties of the final product (Liang and Hartel, 2004).

To our knowledge no papers are available in literature regarding the influence of the single process step on microstructural, rheological and appearance properties of milk chocolate.

In our opinion, in order to improve the final quality of milk chocolate it would be interesting to study in depth the evolution of these important quality characteristics during the different process phases (mixing, pre- refining, refining, conching and tempering). For this purpose in the present work the influence of each process phase on microstructural, rheological and colorimetric properties of milk chocolate were evaluated during the overall manufacturing process.

2. Materials and methods

2.1. Materials

Milk chocolate samples were produced in an Italian confectionery factory by using an industrial plant (Buhler, Malmo, Sweden) provided of mixer, pre-refiner, refiner, conching and tempering machine, and equipped to produce 6000 kg of chocolate at every production cycle. Milk chocolate production was made up by different steps as shown in Fig. 1. The ingredients used in the chocolate formulation were: sugar (47%), cocoa butter (25%), whole milk powder (21%) and cocoa liquor (18%). The experimental samples were taken after each production phase: mixing (A), pre-refining (B), refining (C), conching (D) and tempering (E). In particular, the refining step was realized by using a five-roll refiner, that consists of a vertical array of four hollow cylinders temperature controlled by internal water flow, held together by hydraulic pressure. The temperatures of the five cylinders used to press particles were:1st and 2nd cylinder 28°C; 3th 44°C, 4th 49°C and 5th 30°C.

Samples were stored in plastic bucket (1 kg capacity) at room temperature until the analytical determinations. Before performing the analysis the samples were melted in a microwave (Stortz and Marangoni, 2013) at 150 watt for 25 minutes. The melting parameters were chosen after preliminary experiments in order to avoid changes in the chocolate properties.

2.2. Methods

2.2.1. Microstructure analysis

Samples microstructure was observed using an environmental scanning electron microscope ESEM (Evo 50 EP, Zeiss, Germany) equipped with a microprobe (EDS Mod. 350, Oxford Instrument, UK). The detector used was a backscatter electron detector (QBSE) that provided good compositional contrast imaging at 20 kV and in low vacuum mode with 100 Pascal at 500x magnification. These parameters were chosen after preliminary trials and according to Dahlenborg et al. (2010), in order to cause minimal damage on the chocolate surface and in order to optimize the images quality. By using this kind of instrument ESEM, samples are not coated and the images are more dependent on sample rather than coating characteristics, in this way the true structure can be analyzed (Rousseau, 2007). Ten micrographs for each chocolate sample were taken. The acquired images were subsequently elaborated using the software Image Pro-plus 6.0 (Media Cybernetics Inc Bethesda, USA).

2.2.2. Fundamental properties

Measurements were carried out at 40°C using a controlled strain-stress rheometer (MCR 300, Physica/ Anton Paar, Ostfildern, Germany) equipped respectively with a bob and cup geometry and with a plate-plate system to perform analysis in steady state conditions and the dynamic tests respectively. In steady state conditions, after a pre-shearing of 500 s at 2 s^{-1}, apparent viscositywas measured as function of increasing shear rate from 2 to 50 s^{-1} (ramp up) within 180 s, then decreasing from 50 to 2 (ramp down), within each ramp 18 measurements were taken (ICA, 2000).

Chocolate rheological flow curves are usually fitted (Afoakwa et.al., 2008, 2009b; Taylor et al., 2009) by using the Casson model, that is a well-known rheological model to describe the non-Newtonian flow behaviour of fluids with a yield stress (Joye, 2003). In particular, some fluid products, like chocolate, are well described by this model because of their non linear yield-stress-pseudoplastic nature. According to Chevalley (1991) curve points represent a case for a better fit to chocolate data, if the exponent is taken as 0.6 rather than 0.5.

For this reason, in this study the obtained flow curves were evaluated and fitted according to the rheological model of Casson, modified by Chevalley (1991), in order to obtain a better fit of the chocolate samples. The model used is represented in the following equation (1):

$$\tau^{0.6} = \tau_0^{0.6} + n_{PL}\ y^{0.6} \qquad (1)$$

where τ_0 is the yield stress and ηPL is the so-called "plastic viscosity". In order to measure the goodness of fit, the determination coefficient (R^2) was determined. The yield stress and the apparent viscosity were obtained according to ICA (2000), Servais et al., (2004) and Afoakwa et al., (2008), evaluating the shear stress respectively at 5 and 40 s^{-1}. In particular, the apparent viscosity evaluated at the shear stress of 40 s^{-1} according to Do et al., (2007), reflects the microstructure of the sample taking into account the presence of aggregates.

The samples thixotropy was evaluated according to Servais et al., (2004), from the difference between apparent viscosity measured at 40 s^{-1} during ramp up and ramp down. The thixotropy values represent in very close way the value of the hysteresis area between the apparent viscosity curves during the ramp up and the ramp down. The loop area designates the energy required to break down the structure not recovered during the experimentation period (Roopa and Bhattacharya, 2009) and represents the rate of the internal breakdown of matrix (Dolz et al., 2000).

In dynamic conditions, oscillatory tests by using a plate-plate geometry were performed in order to investigate the viscoelastic properties of samples and to evaluate the storage (G') and the loss (G") modulus. In order to identify the linear viscoelastic range (LVR), in which the viscoelastic properties are independent from the stress conditions, strain sweep tests were applied. Frequency sweep tests were carried out in the viscoelastic linear region at the constant deformation amplitudes of 0.12%, previously evaluated with the strain sweep test, in the range from 1 to 100 Hz.

2.2.3. Colorimetric measurements

Colour of chocolate samples was measured using a colour spectrophotometer mod. Colorflex (Hunterlab, USA), equipped with a sample holder (diameter 64 mm). Colour was measured in the CIE L*a*b* scale using the D65 illuminant. The instrument was calibrated with a white tile (L* = 98.03, a* = - 0.23, b* = 2.05) and the calibration was also validated with a green standard tile (L* = 53.14, a* = - 26.23, b* = 12.01) before the measurements.

Numerical values of a* and b* were converted into hue angle (h°) and Chroma (C*) that represent the hue and the saturation index: $C^* = [(a^*)^2+(b^*)^2]^{1/2}$, $h° = [\arctan (b^*/a^*)/2 \pi]^* 360$ (Mc Guire, 1992).

2.3. Statistical analyses

All the analysis were carried out in triplicate for each chocolate sample.

Analyses of variance (ANOVA) and the test of mean comparison according to Fisher Least Significant Difference (LSD) were conducted on all obtained data. Level of significance was $P \leq 0.05$.

The statistical software used was STATISTICA, version 8.0. (StatSoft, Tulsa, Oklahom).

3. Results and discussion

3.1. Microstructural properties of milk chocolate

In Fig. 2 (a, b, c, d, e) micrographs of milk chocolate samples obtained by ESEM analysis are shown.

ESEM was employed in order to evaluate the main microstructural modifications occurred on chocolate samples during the different process steps, concerning sugar crystalline networks, particle-particle interactions, presence of voids and particle-fat behaviour (Afoakwa, et al., 2009). In table 1 are reported the size diameters of the largest particles measured on chocolate samples, being those that underwent the main modifications during process. Microstructure examination, highlighted different structures between samples obtained from the manufcturing steps.

ESEM micrographs showed a decrease in the particle size from sample obtained after mixing (A) to the one taken after refining (C) (Table 1), parallel to an increase in the presence of large voids between aggregates (Fig. 2 a, b, c). The reduction of the particles diameter causes an increase in the particles number, parallel to an increase in the contact points between them, due to chemical and mechanical interactions (Afoakwa et al., 2009; Servais et al., 2004). The increase of particle interactions from sample obtained after mixing (A) to the one taken after refining (C), due to the raise of their specific surface area, involves a reduction of the particles mobility, due to their high aggregation (Bayod, 2008a; Bayod et al. 2008b). On the other side, the presence of large voids between aggregates (filled with cocoa butter) involves an immobilization of a part of cocoa butter that can not contribute to the continuous fluid phase flow. According with the studies of Windhab (2000), the effective immobilized fluid fraction ($\varnothing_{eff}$) in the particle aggregates can be considered as an increase of solid volume, as explained in the following equation:

$$\varnothing_{eff} = \varnothing_{s} + \varnothing_{sif} + \varnothing_{vif} + \varnothing_{hifi} \quad (2)$$

Where $\varnothing_{s}$ = is the volume occupied by solid particles, $\varnothing_{sif}$ = is the volume of the fluid immobilized by surface, $\varnothing_{vif}$ = is the volume of fluid immobilized in particle cavities and into inner voids in particle aggregates and $\varnothing_{hifi}$ = is the part of fluid immobilized when particles or aggregates move within the continuos phase such as in rotation.

For this reason in order to know the effective solid content in a dispersion, all the parameters presents in the equation (2) must be taken into account. In particular, the cocoa butter immobilized in large voids can have a significativant impact on the rheological behaviour of the milk chocolate system (Windhab, 2000).

The micrographs of Fig. 2 (d, e), related to the samples after conching and tempering steps, show a further reduction in the particle size coupled to a reduction of the larger voids between aggregates, that leds to a reduction of the fluid immobilization. In the conching step a destruction of the previous obtained agglomerates and a re-distribution of cocoa butter between particles was noted, according to Attaie et al., (2003). Cocoa butter in fact, due to its free-moving lubricating plastic flow, coats particles and reduces forces and aggregation between solid particles (Beckett, 2000), thus improving their mobility (Aguilera et al., 2004).

3.2. Fundamental rheological properties

In Fig. 3 the flow curves of the milk chocolate samples, obtained increasing the shear rate from 2 to 50 s $^{-1}$, are reported.

The apparent viscosity (***η***) against shear rate (*γ*) was used to represent the rheological behaviour of milk chocolate; it is evident that the apparent viscosity decreases increasing the shear rate, which proves the pseudoplastic or shear thinning nature of chocolate.

According to Juszczak, et al., (2004) this behaviour can be attributed to the breakdown of the inner structure dispersions, in fact the increase of shear rate causes the drop in the apparent viscosity of the molecules orientating along the flow lines.

As illustrated in Fig. 3, sample C achieved after the refining step, had the highest apparent viscosity with initial values ranging around 60 Pa s, followed by sample B, taken after the pre-refining step with initial apparent viscosity values between 20 and 30 Pa s and sample A obtained from the first step, with values between 10 and 20 Pa s. D and E samples, obtained from the last two steps of the manufacture process, had the lowest apparent viscosity values, ranging from 0 to 10 Pa s.

In order to better explain the rheological values obtained by the flow curves, the *Casson yield value* and the *Casson plastic viscosity* parameters were calculated applying the Casson model modified by Chevalley (1991), moreover yield stress and apparent viscosity values were obtained according to Afoakwa et al., (2008) and ICA (2000). All these data are reported in Table 2 for each chocolate sample.

All data were well fitted by the *Casson* model, providing high determination coefficients (R^2) that varied from 0.75 to 0.99. A significantly increase in both *Casson* obtained parameters was highlighted from sample (A) obtained after mixing to the one taken after refining (C). This could be attributed to the increase of the contact point between particles, that need of a major amount of stress to initiate the flow, and to the presence of large void spaces that immobilized cocoa butter between aggregates. In this state the fat can not contribute to the flow as lubricant (Franke and Heinzelmann, 2008). Samples after conching (D) and tempering (E) were characterized by the lowest and significantly similar values of both Casson parameters. In particular, the obtained values of plastic apparent viscosity are in agreement with the results of Wichchukit et al., (2004), that showed that *Casson viscosity* of milk chocolate with 20% of cocoa butter, ranged from 7 to 48 Pa s and led to decrease with the adding of lubricant. In the samples studied in this research work the highest value of *Casson* apparent viscosity was lower (25.7 Pa s), than the one obtained in the study of Wichchukit et al., (2004), probably due to a higher amount of cocoa butter used in formulation (25%), that caused a greater lubricating effect and a reduction of particle–particle interactions (Vernier, 1998).

The yield stress and apparent viscosity parameters, exhibited the same trends of the *Casson yield value* and of the *Casson Plastic Viscosity* in milk chocolate samples. According to the studies of Do et al., (2007) in fact an increase in the apparent viscosity, as from sample after mixing (A) to the one after refining (C), also in this case indicates an higher degree of particles aggregation, while a decrease of this parameter, as for samples after conching (D) and after tempering (E), underlines a lower degree of interactions, as confirmed by microstructural analysis results.

Thixotropy results are shown in Fig. 4. It is possible to notice how C and B samples obtained respectively after the refining and pre-refining steps, that had the most aggregate structure, presented also the significantly highest thixotropy values, related to a more damaged structure. This result according to Afoakwa et al., (2008) could be attributed to the high aggregation of the particulate system and to an elevate number of interactions beetween particles. Sample A taken after the mixing was characterized by an intermediate thixotropic value, between B-C and D-E ones, strictly related with the results obtained from microstructural examination, that reflects the presence of coarse particles and a weak solid structure compared to B and C samples obtained from the pre-refining and refining phase. The lowest significantly values of thixotropy were showed by chocolate samples D, after conching and E, after tempering. According with literature (Afoakwa et al., 2008) in fact, a well conched and tempered chocolate should not be thixotropic and hence should not have a very aggregate structure. Anyway, it is very unusual to have not any thixotropy.

The results of frequency sweep test in terms of storage and loss modulus, evaluated respectively at a frequency of 1 Hz, are reported in Table 3. The response of all samples to the imposed deformation is the stored potential energy, characterized by the predominance of the elastic modulus (G') over the viscous one (G'') (Ahmed and Ramaswamy, 2006; Bayod & Tornberg, 2011).

B and C samples, obtained from pre-refing and refining steps, were characterized by a relative more elastic structure compared to that of the other samples (A, D and E, taken after mixing, conching and tempering). As reported in previous studies (Johansson and Bergensthål, 1992; Glicerina et al., 2013) high values of G' are related to a high level of interactive forces between particles; this confirms the high amount of stress necessary to pre-refining (B) and refining (C) samples to start flow.

The significantly lowest values of G' and G" were found for the samples after conching (D) and after tempering (E), constituted by a weakly structure.

3.3. Colorimetric measurements

The lightness (L*) and hue angle (h°) values of A – E milk chocolate samples are shown in Fig. 5.

A similar trend of lightness and hue angle values was observed in all samples. A and B samples, taken from the first two steps and characterised by coarser particles, had the lowest significantly values of both colour parameters. As known (Voltz and Beckett, 1997; Afoakwa et al., 2008), the human eye detects colour according to how the light is reflected from the surface, thus the size of the both non-fat solid and crystalline fat particles affects the colour of chocolate. In particular, in a dense packed medium, light scattering factors are inversely related with particle diameters (Saguy & Graf, 1991; Afoakwa et al., 2008), for this C, D and E samples, (obtained respectively from the refining, conching and tempering steps) having finer particles and a large specific surface area, tended to scatter more light, appearing lighter than A and B samples, that had larger particles. At the same time the highest hue angle values were found in C, D and E samples, that had a more yellonish-brown hue than A and B ones.

4. Conclusions

The modifications in the microstructure of milk chocolate during the different processing steps involve deep changes in the rheological and colorimetric parameters of product.

In particular, the decrease in particle size detected from sample A taken from the mixing step to C obtained from the refining one, simultaneosly to an increase in the void spaces that immobilize cocoa butter, involves an increase in all rheological analyzed parameters. The re-distribution of cocoa butter during the conching step, let to a decrease in all rheological values in D and E samples obtained after the conching and tempering steps, probably because of the reduction in particle-particle interactions due to the cocoa butter that, wrapping particles, reduces forces between them. At the same time, colorimetric characteristics were also affected by the different microstructure of samples.

From results obtained in this work it can be concluded that the knowledge of the influence of process parameters on the milk chocolate microstructure becomes very important in order to modify, improve and/or optimize the rheological and colorimetric properties of final product.

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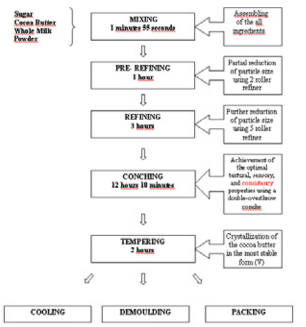
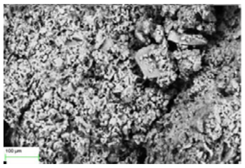
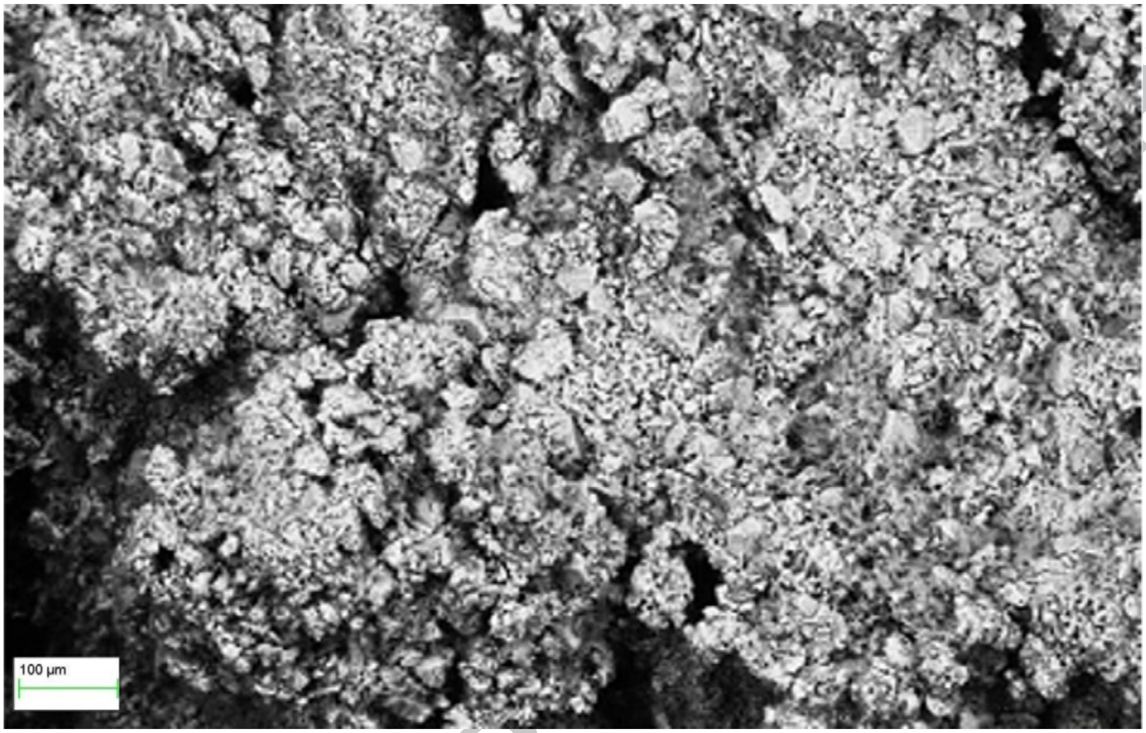
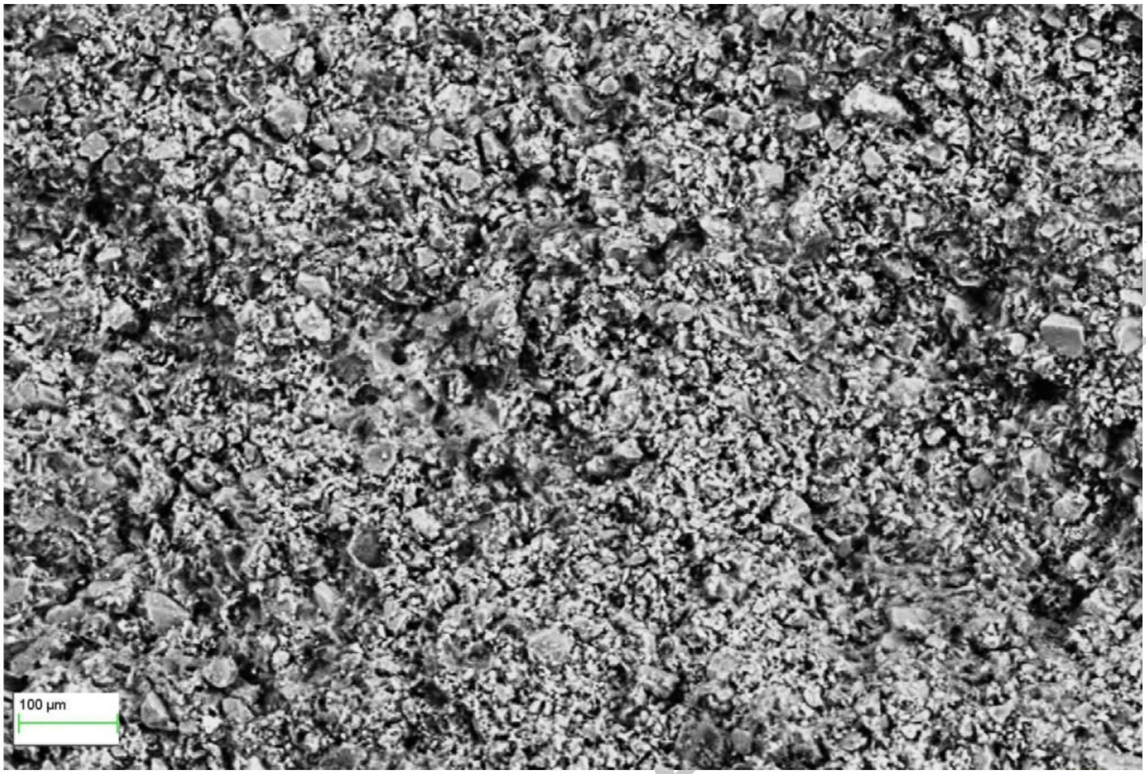
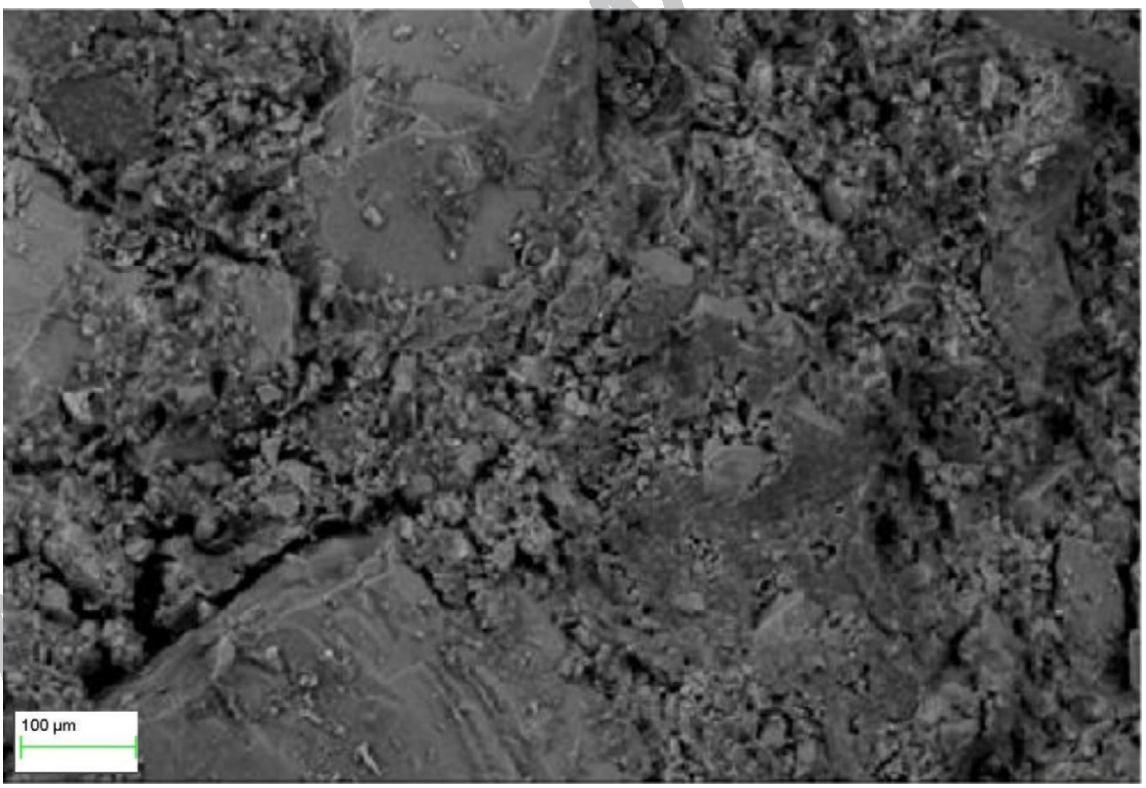

Sugar
Cocoa Butter
Whole Milk
Powder
MIXING
1 minutes 55 seconds
Assembling of the all ingredients
PRE- REFINING
1 hour
Partial reduction of particle size using 2 roller refiner
REFINING
3 hours
Further reduction of particle size using 5 roller refiner
CONCHING
12 hours 10 minutes
Achievement of the optimal textural, sensory, and consistency properties using a double-overthrow conche
TEMPERING
2 hours
Crystallization of the cocoa butter in the most stable form (V)
COOLING
DEMOULDING
PACKING

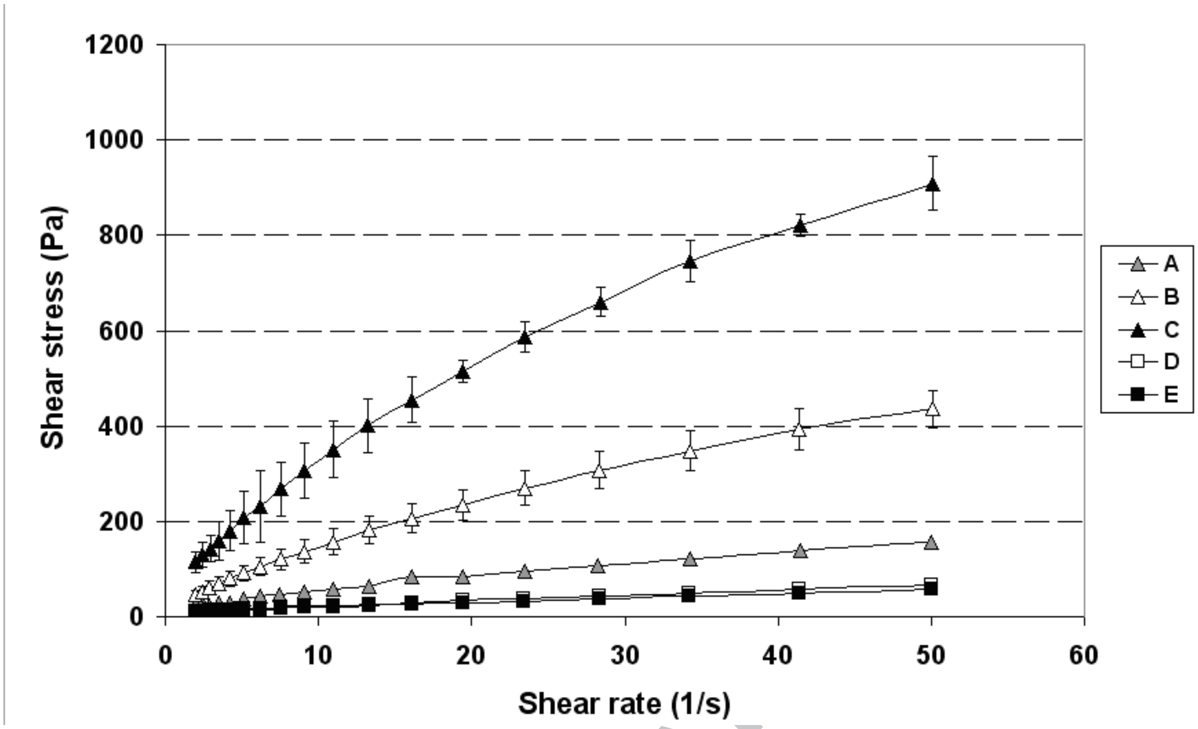
416 

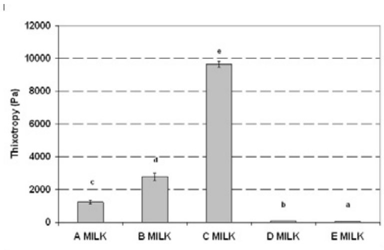
417 

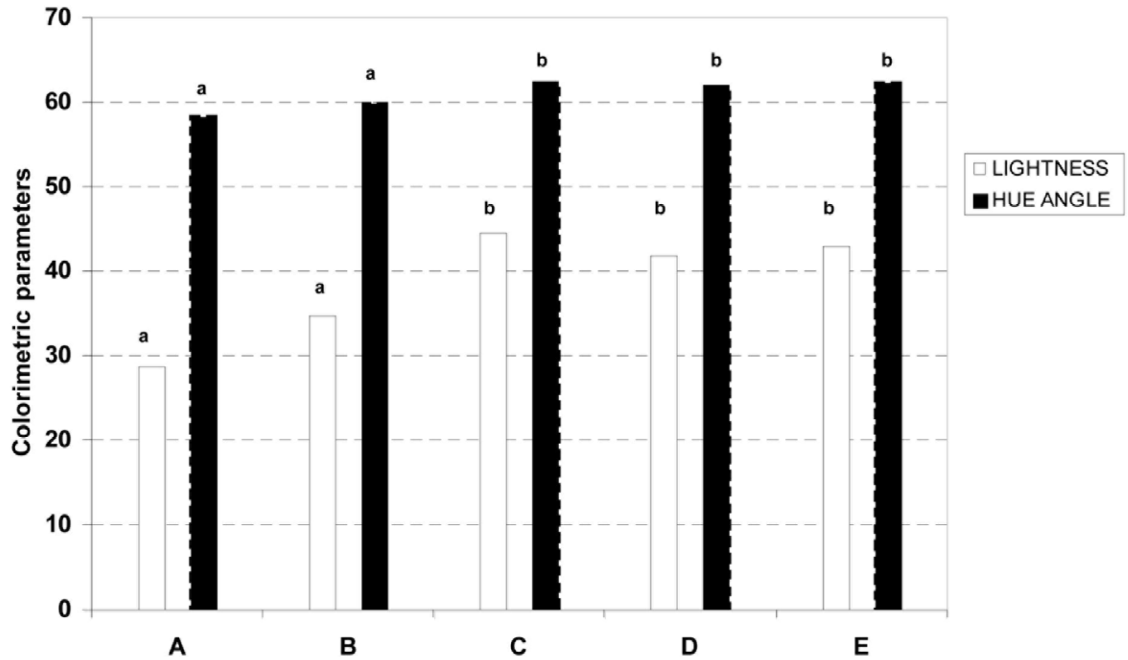
418 


100 µm


100 µm


Shear stress (Pa)
Shear rate (1/s)
1200
1000
800
600
400
200
0
10
20
30
40
50
60
A
B
C
D
E


12000
10000
8000
6000
4000
2000
0
Thixotropy (Pa)
c
a
e
b
a
A MILK
B MILK
C MILK
D MILK
E MILK



Colorimetric parameters
70
60
50
40
30
20
10
0
A
B
C
D
E
LIGHTNESS
HUE ANGLE
a
b

Legends to Figures

Fig. 1. Scheme of chocolate manufacturing process (adapted from Babin, H. 2005).

Fig.2. Microghraphs of milk chocolate after different processing steps: (a) mixing, (b) pre-refining, (c) refining, (d) conching and (e) tempering.

Fig.3. Changes of apparent viscosity (Pa s) of milk chocolate samples, during mixing (A), pre-refining (B), refining (C), conching (D) and tempering (E) steps, evaluated at 40°C.

Fig. 4. Changes of thixotropy of milk chocolate samples during mixing (A), pre-refining (B), refining (C), conching (D) and tempering (E) steps.

Fig. 5. Lightness (L*) and hue angle (h°) colorimetric parameters of milk chocolate samples during mixing (A), pre-refining (B), refining (C), conching (D) and tempering (E) steps.

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Highlights

-Milk chocolate quality is affected by particles caractheristics and from the process.

-Influence of single steps on structural properties are useful, to improve the rheological ones.

-Microstructure and rheology are key parameters to optimize final properties of milk chocolate.

Table 1. Microstuctural analysis of the milk chocolate.

Samples	Particle size (Feret diameter)
A_{mixing}	$103.00^{a} \pm 2.57$
$B_{pre\text{-}refining}$	$67.00^{b} \pm 3.54$
$C_{refining}$	$29.00^{c} \pm 2.37$
$D_{conching}$	$22.00^{c} \pm 2.56$
$E_{tempering}$	$17.91^{c} \pm 3.73$

[a-c] values in the same column followed by different letters differ significantly at $p < 0.05$ level

Table.2. Casson yield values, Casson Plastic Viscosity, Yield stress and Apparent Viscosity of milk Chocolate samples.

Samples	***Casson Yield value* (Pa)**	***Casson Plastic Viscosity* (Pa*s)**	**Yield stress (Pa)**	**Apparent Viscosity (Pa*s)**
A_{mixing}	6.82 ± 0.63^{b}	4.38 ± 0.30^{b}	37.10 ± 3.14^{b}	3.84 ± 0.11^{b}
$B_{pre\text{-}refining}$	11.97 ± 0.58^{c}	7.82 ± 0.83^{c}	91.10 ± 5.95^{c}	10.84 ± 1.39^{c}
$C_{refining}$	35.70 ± 4.70^{d}	15.36 ± 2.30^{d}	209.33 ± 8.14^{d}	23.23 ± 2.15^{d}
$D_{conching}$	2.75 ± 0.23^{a}	1.55 ± 0.35^{a}	16.93 ± 2.17^{a}	1.53 ± 0.13^{a}
$E_{tempering}$	1.95 ± 0.04^{a}	0.21 ± 0.00^{a}	14.56 ± 1.45^{a}	1.32 ± 0.12^{a}

a-d values in the same column followed by different letters differ significantly at $p < 0.05$ level.

Table 3. Storage and loss modulus of milk chocolate samples evaluated at 1 Hz and at 40°C.

Samples	**G' (Pa)**	**G'' (Pa)**
A_{mixing}	8416 ± 125^{b}	1281 ± 32^{b}
$B_{pre\text{-}refining}$	13673 ± 644^{c}	2357 ± 24^{c}
$C_{refining}$	72746 ± 890^{d}	$16873 \pm^{d}$
$D_{conching}$	3983 ± 112^{a}	807 ± 34^{a}
$E_{tempering}$	2873 ± 97^{a}	798 ± 84^{a}

a-d values in the same column followed by different letters differ significantly (***p* < 0.05**).