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Effect of manufacturing process on the microstructural and rheological properties of milk chocolate

Virginia Glicerina [a,*], Federica Balestra [a], Marco Dalla Rosa [a], Santina Romani [a,b]

[a] Interdepartmental Centre for Agri-Food Industrial Research, Alma Mater Studiorum, University of Bologna, Piazza Goidanich 60, Cesena, FC, Italy
[b] Alma Mater Studiorum, University of Bologna, Department of Agro-Food Science, Campus of Food Science & Technologies, Piazza Goidanich 60, 47521 Cesena, Italy

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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.

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 and Stanley, 1999; 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.

* Corresponding author. Tel.: +39 0547/338120; fax: +39 0547/382348.
E-mail address: virginia.glicerina2@unibo.it (V. Glicerina).

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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 W for 25 min. 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 500× 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).

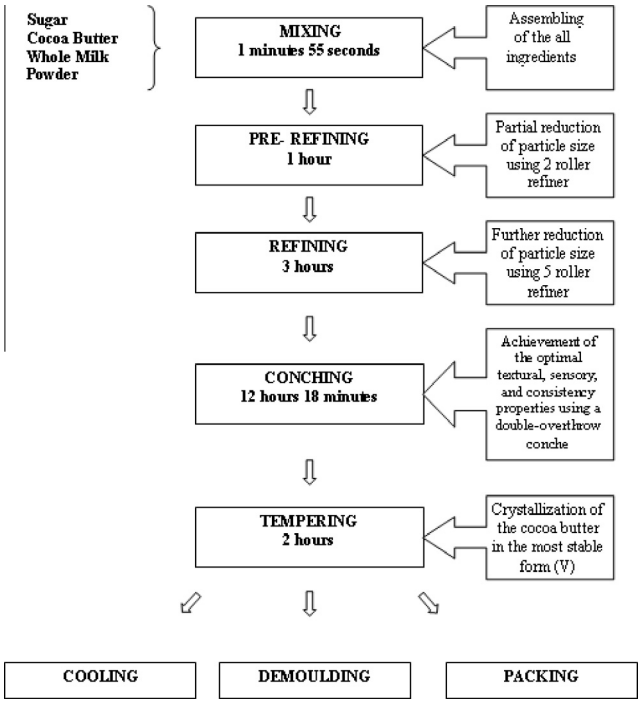

Fig. 1. Scheme of chocolate manufacturing process (adapted from Babin, 2005).

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 viscosity was 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 Eq. (1):

$$\tau^{0.6} = \tau_0^{0.6} + \eta_{PL}\gamma^{0.6} \quad (1)$$

where τ_0 is the yield stress and η_{PL} 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.