Influence of Refining and Conching Systems on Rheological and Sensory Properties of Chocolate

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Abstract

There are different refining systems on the market and knowing the differences between these systems can help choose equipment according to the needs of each chocolate manufacturer. This work aimed to evaluate different technologies for the chocolate refining stage and evaluate the impacts on the physical, chemical and sensory characteristics of the chocolates produced. We evaluated the following refining systems: System 1: Double refining; System 2: Refining in ball mill; System 3: Refining in ball mill and BLC conching; System 4: Refining and conching in refining conche; System 5: Refining and conching in stone mill – melanger; System 6: Simple refining. All of these refining systems were evaluated for pH, total titratable acidity, moisture, maximum particle size, particle size distribution, rheology, and sensory characteristics. The refining and conching system resulted in considerable changes in the evaluation of features such as total titratable acidity, moisture, maximum particle size, particle size distribution, and rheological properties. In the sensory evaluation, attributes such as aroma, hardness, melting, and color did not show significant differences. On the other hand, we observed significant differences in attributes such as overall impression, flavor, grittiness, and acidity. It was possible to conclude that the combination of refining systems with homogenizing conche can be favorable for obtaining chocolates due to greater fluidity and better results in sensory evaluation.

Keywords: dark chocolate, particle size, rheology, sensory evaluation

1. Introduction

Traditionally, chocolate manufacturing methods are based on mixing ingredients, grinding in roller refiners (refining stage), conching and tempering (Beckett, 1999; Ziegler & Hogg, 2009; Alamprese, Datei & Semeraro, 2007).

All these steps in the chocolate manufacturing process, in addition to the parameters adopted, affect the properties of the chocolate and determine the behavior and characteristics of the final product. Thus, controlling, determining and knowing the technological parameters is essential to achieve a constant and desirable quality in production (Muller-Fischer & Windhab, 2005; Baixauli, Sanz, Salvadora & Fiszmana, 2007).

Refining as a whole is the step in which particle size is reduced with the main purpose of making sugar, dairy products and cocoa solids physically imperceptible in the mouth, thus not causing a gritty sensation. Moreover, during the refining step, there is also homogenization of ingredients and coating of solid surfaces by the lipid phase (Ziegler & Hogg, 2009). At this step, it is desirable to reduce the size of particles, mainly of sugar and cocoa, to less than 0.03 mm (Beckett, Francesconi, Geary, Mackenzie & Maulny, 2006).

Conching, in turn, has as its main objectives: to remove moisture and undesirable flavors while developing pleasant flavors. However, since the previous refining process will have created surfaces not yet covered with fat, the conching phase coats these new surfaces and improves flow properties (Beckett, 1999).

It is due to the combined effect of mixing, grinding and recirculation that the surface of solid particles is covered by fat, giving the paste its rheological properties (Petković, Pajin & Tomić, 2013; Cavela et al., 2020).

Because, during these steps, the cocoa, milk and sugar solids are broken down, and the smaller the particle size, the greater the surface area for coating by the lipid phase and, consequently, the greater the resistance to flow (Talbolt, 2009). According to Beckett (2009), the lipid phase can be present in two forms in chocolate: as free fat or as fat associated with solid particles. Free fat enables the particles to pass one another, that is, to move, and thus the chocolate to flow. With increased free fat, the distance between the other solid particles increases and, consequently, viscosity decreases.

Refining is usually performed in a five-roll refiner. Thus, there are four vertically aligned grinding rollers, while one roller feeds them. However, in order to meet new requirements from chocolate producers, where compact production plants are needed, new types of plants and equipment are under development (Beckett, 1999; Ziegler & Hogg, 2009).

These new chocolate production systems are associated with advantages that boost growing popularity and acceptance, such as time and energy efficiency, compactness (capability to combine multiple processing steps), cost efficiency, and lower demand for highly-trained personnel (Hinneh, Walle, Tzompa-Sosa & Haeck, 2019).

Among the most common, there is the ball mill, which uses the relative movement of loose elements (balls) for grinding. Normally, these consist of cylinders with a rotating shaft, being filled up to 90% of the volume with grinding elements (such as steel balls, ceramic balls, among others), so grinding occurs by compression and shear. In systems like this, temperature is controlled so as to ensure the melting of solid fats, avoiding excessive heating of the product and executing the replacement action of traditional conching (Beckett, 1999; Ziegler & Hogg, 2009).

Stone mills – called melangers – are also alternatives for producing small batches of chocolate, due to the possibility of small production and low initial investment. These mills can also replace conches, but conches are generally used at temperatures close to 80 ℃ and initial refining is required, since the melanger lacks a temperature control system and conching and refining are carried out in the same equipment (Vishwanathan, Singh & Subramanian, 2011; Aidoo, Clercq, Afoakwa & Dewettinck,, 2014; Vreeland, 2015; Albak & Tekin, 2016; Tan & Balasubramanian, 2017; Bastos, Uekane, Bello, Rezende, Paschoalin & Del Aguila, 2019; Hinneh et al., 2019).

Another system, the refining conche, is an option for the pre-milling, mixing, refining, and conching steps in a single piece of equipment. This type of system enables temperature control, requires little space, and is efficient with different particle sizes of ingredients such as sugar. This system is recommended even for formulations with low fat content, which is possible due to a combination of good grinding performance with high-efficiency product cooling (Duyvis Wiener, 2021; Jaf Inox, 2021).

When choosing equipment or even a refining/conching system, the characteristics of the equipment material, maintenance, product specifications, yield, and the required particle size reduction ratio must be considered. Thus, ideal equipment should have high material processing capacity in relation to power consumed in the operation and output a product with adequate particle size distribution (Ziegler & Hogg, 2009; Mccabe, Smith & Harriott, 2005).

Knowing particle size distribution and shape in chocolate is essential to understand the behavior of the mass, such as flow properties, bulk density, bed porosity, and stability of emulsions. The surface area of these particles depends on the size and shape of these particles, so they have a direct correlation with the sorption, dissolution, and chemical reaction kinetics (Figura & Teixeira, 2007). Several studies have reported the relation between the rheological properties of chocolate and its formulation, fat crystallization kinetics, and particle size (Servais, Ranch & Roberts, 2004; Afoakwa, Paterson & Fowler, 2008c; Afoakwa, Paterson, Fowler & Vieira, 2008b, 2009b; Baldino, Gabriele & Migliori, 2010; De Graef, Depypere, Minnaert & Dewettinck, 2011; Efraim, Marson, Jardim, Garcia & Yotsuynagi, 2011; Fernandes, Muller & Sandoval, 2013).

Therefore, the objective was to evaluate different technologies available for the refining and conching stage by determining the physical, chemical, and sensory impacts on dark chocolate.

2. Methodology

2.1 Chocolate Processing

The chocolate samples were produced between November 2017 and April 2018 at the JAF INOX factory. Dark chocolates were produced containing 65% cocoa mass (Harald Ind. e Com. Alimentos Ltda) and 35% sugar (Mais Doce - Açucareira Boa Vista).

The ingredients were mixed in homogenizing conche for 5 min. Then, the obtained masses were submitted to six different systems to undergo the refining and conching steps, as described below:

- ✓ System 1: double refining (2-roll pre-refiner and 5-roll cooled refiner) and conching in homogenizing conche.
- ✓ System 2: refining in ball mill and conching in homogenizing conche.
- ✓ System 3: refining in ball mill and conching in Batch Liquid Conche (BLC) system.
- ✓ System 4: refining and conching in the same equipment (refining conche) and at the same time.
- ✓ System 5: refining and conching in the same equipment (stone mill – melanger) and at the same time.
- ✓ System 6: Simple refining (single stage) in system with 5 cooled rolls and conching in homogenizing conche.

The processes in systems 1 to 5 employed coarse sugar (particle size ≤ 700 mm) and the process in system 6 employed fine sugar (particle size ≤ 150 mm).

The pieces of equipment used for refining and conching are described in Table 1.

Table 1. Chocolate refining and conching equipment

EQUIPMENT	**BRAND/COUNTRY**	**Model/Capacity/Size**	**System that was used**
2-roll refiner	JAF Inox (Brazil)	300 / 300 mm	System 1
5-roll refiner	JAF Inox (Brazil)	100 X 200 mm	Systems 1 and 6
Boiler	JAF Inox (Brazil)	20 L	Systems 2, 3 and 4
Cooler	Mecalor (Brazil)	RAI-5-220 / 3000 M/ ħ	Systems 1, 2, 3 and 6
Homogenizing conche	JAF Inox (Brazil)	10 kg / 1 shaft	Systems 1, 2 and 6
Refining conche	JAF Inox / Duyvis Wiener (Netherlands)	20 Kg	System 4
Qchoc (ball mill, mixer, and Batch Liquid Conche – BLC)	Duyvis Wiener (Netherlands)	50 to 400 kg / batch	Systems 2 and 3
Stone mill	Mecal (Brazil)	20 Kg	System 5

Source: Prepared by the author, 2021.

After refining and conching, the chocolates were sent to the School of Food Engineering at the State University of Campinas - FEA/UNICAMP to undergo the other steps.

Tempering was carried out in a tempering machine (Dedy GmbH, Essen, Germany). Initially, the chocolate mass was heated to 45 ℃ and then cooled, under Top temper D 45134 constant movement, to 29 ± 1.0 ℃, at a rate of 2 ℃/min, remaining at this temperature for 15 min. Then, the mass was reheated to 31 ℃. To control the tempering step, the tempering index was controlled by a ChocoMeter (Aasted-Mikroverk ApS Farum, Denmark), and tempering index between 4.0 and 6.0 were considered adequate.

The dosage of chocolate was performed manually in polypropylene molds in the shape of rectangular bars. Then, the molds were placed on a vibrating table (JAF Inox, Brazil) to eliminate air bubbles. Subsequently, the chocolates were cooled in a refrigerated counter, demolded, packed in laminated packaging and stored in a chamber (BOD TE-371, Tecnal, Brazil) at 20 ℃ for the analyses described below.

2.2 Physicochemical Analyses

Maximum particle size was determined using a digital micrometer (Mitutoyo, Japan) with a scale of 0–25 mm.

Total titratable acidity and pH were determined by using a digital pH meter (Digimed DM-20, Brazil) by the AOAC 31.1.07 and 942.15 methods, respectively (Association of Official Analytical Chemist [AOAC], 2016).

Moisture content was measured by the AOAC 970.20 gravimetric method (AOAC, 2016) with the aid of an oven with air renewal and circulation (TE-394-2 Tecnal, Brazil) and an analytical scale (Mettler Toledo AB204, Switzerland).

2.3 Particle Size Distribution

Particle size distribution in the chocolates was measured in the Instrumentation Laboratory (FEA/UNICAMP) in a Mastersizer 2000 laser diffraction system with a Hydro 2000S dispersion unit (Malvern Instruments Limited, United Kingdom), whose measurement range comprises particles from 0.02 to 2000 μm. Approximately 0.5 g of each chocolate sample was dispersed in mineral oil. The analyses were carried out at room temperature in triplicate. The analysis model selected was that of general use for irregular particles. Particle size distribution was expressed in micrometers by the values of volumetric mean diameter D [4,3], median $d_v(0.5)$, and surface weighted mean D [3,2]. We also evaluated the parameters $d_v(0.1)$ and $d_v(0.9)$, which represent the diameter values below which are located 10% and 90% of the cumulative distribution (in volume), respectively. The span value was used as indicator of particle size distribution range. The analysis followed a methodology adapted from Toker, Sagdic, Sener, Konar, Zorlucan and Daghoglu (2016) and Afoakwa, Paterson and Fowler (2008).

2.4 Rheological Properties

The rheological properties were analyzed at the Instrumentation Laboratory (FEA/UNICAMP) according to the methodology of De Graef et al. (2011).

The measurements were performed on an AR2000 (ex) stress controlled rheometer (TA Instruments, Belgium). Prior to analysis, the samples were melted in an oven at 40 ℃ and transferred to DIN cup and bob geometry that was adjusted to 40 ℃. There were two different rheological measurements: a stepped flow experiment to record the flow curve and an oscillatory stress sweep test to determine the linear viscoelastic region (LVR). Each rheological analysis was carried out in triplicate.

2.4.1 Flow Curve

After a 2-min conditioning step, the shear rate was increased from 0.1 s^{-1} to 100 s^{-1}. The flow curve was then obtained by plotting the recorded shear stress (Pa) as a function of the applied shear rate (1/s).

2.4.2 Stress Sweep Tests

Oscillatory measurements were performed at a frequency of 1 Hz. After a 2-min conditioning step, the chocolate sample was subjected to oscillatory stress increasing from 0.01 Pa to 50 Pa. Rheograms were obtained by plotting the complex modulus G* (Pa) as a function of oscillatory shear. The complex modulus can be defined as the ratio of stress to the corresponding strain and is considered a measure of system rigidity. As the complex modulus is a measure of system rigidity, it provides information on the microstructure of chocolates.

2.5 Sensory Analysis

The chocolates were evaluated by Acceptance Test with chocolate consumers at the Sensory Analysis Laboratory (FEA/UNICAMP) following a randomized complete block design with 130 tasters with no age, sex and social class restrictions (Moskowitz, 1983; Meilgaard, Civille & Carr, 1999).

The samples were identified using random numerical three-digit codes and presented sequentially in single portions to the tasters in individual booths. The overall impression, aroma, flavor, hardness, melting, grittiness, acidity, and color sensory attributes were evaluated using a 9-point hedonic scale.

The sensory analysis was registered with the research ethics committee with the CAAE number: 00802918.5.0000.5404.

2.6 Data Analysis

The data obtained in the analytical determinations and in the sensory evaluation were submitted to Analysis of Variance (ANOVA) and the means were compared by Tukey Test, at 5% significance level, using statistical package PAST® 3 (Hammer, Harper & Ryan, 2001).

3. Results and Discussion

3.1 Physicochemical Analyses

The results of the pH, total titratable acidity, and moisture determinations are described in Table 2.

Table 2. pH, total acidity, and moisture content of dark chocolates produced in different refining / conching systems

Systems*	pH	Total acidity (g/100g)	Moisture content (g/100g)
1	5.29 ± 0.05^{a}	0.53 ± 0.03^{ab}	0.86 ± 0.21^{b}
2	5.40 ± 0.01^{a}	0.49 ± 0.03^{b}	1.46 ± 0.17^{a}
3	5.09 ± 0.61^{a}	0.55 ± 0.03^{ab}	0.89 ± 0.10^{b}
4	5.22 ± 0.01^{a}	0.61 ± 0.03^{a}	0.75 ± 0.05^{b}
5	5.25 ± 0.03^{a}	0.51 ± 0.06^{ab}	0.64 ± 0.11^{b}
6	5.28 ± 0.02^{a}	0.61 ± 0.29^{a}	0.63 ± 0.06^{b}

Notes: Equal letters in the same column indicate that the samples did not differ statistically in the Tukey Test at 5% significance.

*System 1: Double refining; System 2: Refining in ball mill; System 3: Refining in ball mill and BLC conching; System 4: Refining and conching in refining conche; System 5: Refining and conching in stone mill – melanger; System 6: Simple refining.

Source: Prepared by the author, 2021.

Significant differences can be observed in the evaluations of total titratable acidity and moisture, especially for System 2 (Ball mill / Homogenizing conche), with the lowest and highest values, respectively. Significant differences were not found in the pH evaluation.

The values suggest that the use of the ball mill and the homogenizing conche can reduce the loss of water, with a consequent increase in the moisture content. Thus, the presence of a greater amount of water can influence the lower percentage of acids found.

3.2 Particle Size Distribution

The results obtained for particle size distribution are shown in Figure 1 and Table 3.

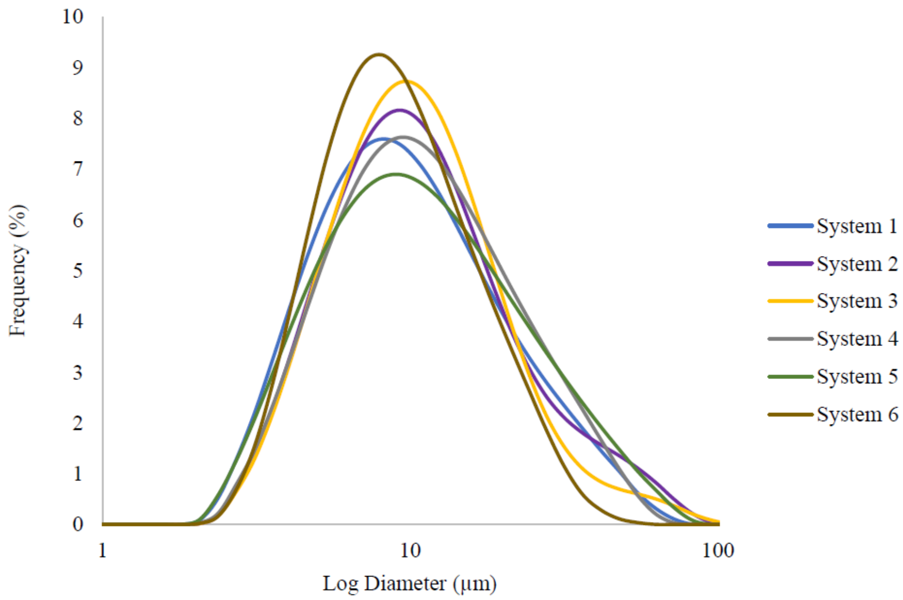

Figure 1. Particle size distribution in dark chocolates produced in different refining / conching systems

Note: System 1: Double refining; System 2: Refining in ball mill; System 3: Refining in ball mill and BLC conching; System 4: Refining and conching in refining conche; System 5: Refining and conching in stone mill – melanger; System 6: Simple refining.

Source: Prepared by the author, 2021.

Table 3. Maximum particle diameter (micrometer) and particle size distribution (laser diffraction) of dark chocolates produced in different refining / conching systems

Systems*	**Maximum particle size (mm)**	**Particle size distribution**					
		D[4,3] ^{1}	**D[3,2]** ^{2}	**dv (0.5)** ^{3}	**dv(0.1)** ^{4}	**dv(0.9)** ^{5}	**Span** ^{6}
1	0.019 ±0.004^{bc}	12.049	7.502	8.834	3.908	24.853	2.371
2	0.017 ±0.003^{c}	13.313	8.250	9.581	4.385	26.944	2.357
3	0.027 ±0.002^{a}	12.221	8.135	9.388	4.448	22.066	1.871
4	0.024 ±0.004^{ab}	12.990	8.358	9.951	4.365	26.200	2.194
5	0.024 ±0.002^{ab}	13.583	7.971	9.759	3.987	28.724	2.535
6	0.016 ±0.002^{c}	10.034	7.311	8.205	4.159	18.623	1.764

Notes: ^{1}Volumetric mean diameter. ^{2}Surface weighted mean. ^{3}Median. ^{4}Diameter values below which are 10% of the cumulative distribution (by volume). ^{5}Diameter values below which are 90% of the cumulative distribution (by volume). ^{6}Particle size distribution range.

*System 1: Double refining; System 2: Refining in ball mill; System 3: Refining in ball mill and BLC conching; System 4: Refining and conching in refining conche; System 5: Refining and conching in stone mill – melanger; System 6: Simple refining.

Source: Prepared by the author, 2021.

All samples showed values below 0.030 mm for maximum particle size. The lowest values were found for samples produced in System 1 (2-roll and 5-roll refiner / Homogenizing conche), System 2 (Ball mill / Homogenizing conche) and System 6 (5-roll refiner / Homogenizing conche). Maximum solid particle size should preferably be less than 25 μm (or 0.025 mm) in order to ensure that the papillae do not perceive the granulation of the particles. Values greater than 35 μm become gritty in the mouth, resulting in less acceptability. However, solid particles should not be too small as they can make the paste viscous (Cavella, Miele, Fidaleo, Borriello & Mais, 2020; Puleo, Miele, Cavella, Masi & Di Monaco, 2020).

The particle volumetric mean diameter (D[4,3]) in the samples presents higher values for Systems 2 and 5 (Ball mill / Homogenizing conche and Stone mill). On the other hand, Systems 2 and 4 (Ball mill / Homogenizing conche and Refining conche) present a higher surface weighted mean of the evaluated particles (D[3,2]).

The volume-based diameter (D[4,3]) is mainly determined by the presence of large particles, while the area-based diameter (D[3,2]) also considers small particles. These small particles can also be important for influencing physicochemical and textural characteristics, as they fill spaces between larger particles (Glicerina, Balestra, Rosa, Bergenhstal, Tornberg & Romani, 2014; Bayod, 2008).

The median (dv (0.5)), which can be understood as the central value that separates the numerical set, that is, the value in which half of the population is above and half below this point, presented higher values for System 5 (Stone Mill), which justifies this treatment being the second system with the lowest dv (0.1) – diameter values below which are 10% of the cumulative particle distribution in volume – and the first with the highest diameter value below which are 90% of the cumulative distribution in volume (dv (0.9)). Figure 1 shows that this sample is the one with the flattest peak. According to Glicerina et al. (2014), this trend involves a large volume also occupied by very small particles.

Evaluating the particle size distribution range (Span Index) enabled finding that System 6 (5-roll refiner / Homogenizing conche) had the lowest value, in contrast to the samples from System 1 (2-roll and 5-roll refiners / Homogenizing conche) and System 5 (Stone mill).

System 6 presented the lowest values for volumetric mean diameter (D[4,3]), surface weighted mean (D[3,2]), and median (dv (0.5)), diameter values below which are 90% of the cumulative distribution in volume (dv (0.9)) and the particle distribution range (Span Index). The only exception are the diameter values below which are 10% of the cumulative distribution in volume (dv (0.1)), where this sample presents intermediate values. This behavior can be seen in Figure 1, where the same sample is the first to reach the highest point and the highest decline in relation to the peak. It should be noted that this sample was the only one that used sugar with smaller particle size (fine sugar) in its formulation, since sugars with larger particle size (coarse sugar) were used for the others.

System 6 also showed a unimodal trend in Figure 1, while the other samples showed bimodal trend. These behaviors are similar to those found by Glicerina et al. (2014), who reported the influence of different stages on the distribution, leading some systems to change from a bimodal to a unimodal trend.

The dv (0.9) is normally considered acceptable if below 23 μm, as it is believed that, above this size, particles can produce an unpleasant gritty mouthfeel (Beckett, 1999; 1994). The present study showed values ranging between 18.623 and 28.724 μm, which indicates some points as inadequate. However, the results are similar to those found in a study conducted by Alamprese, Datei and Semerado (2007), in which the values also ranged between 14.5 and 28.5 μm.

Particle size evaluation by light scattering consists in a monochromatic laser beam that is directed to the particles, undergoing scattering caused by diffraction, reflection or refraction. The angle formed by the scattering depends on the size and shape of these particles (Figura & Teixeira, 2007). These techniques provide a weighted distribution, as the contribution of each particle present is related to the intensity of the light that was scattered by the particle.

Therefore, it was found that System 6 (5-roll refiner / homogenizing conche) presents the lowest variation in particle size, making it a more homogeneous process in relation to the other evaluated systems. On the other hand, Systems 2 and 3 (Ball mill/BLC and Ball mill/Homogenizing conche) and System 5 (Stone Mill) showed the greatest variation in particle diameter.

A study conducted by Henneh et al. (2019), which compared refining systems that employed stone mill (melanger) and conventional 3-roll refiner, also observed significant changes in parameters related to particle size distribution. According to the authors, these changes imply a significant impact of the type of equipment/system on the final chocolate particle size.

3.3 Rheological Properties

Viscosity is extremely important in the production of chocolates because it impacts the production cost, as the efficiency in mixing, pumping and mold filling is directly affected by flow characteristics (Afoakwa, Paterson & Fowler, 2008c).

Particle size distribution is also fundamental in the flow characteristics of chocolate mass. In the refining process, particles such as sugar and cocoa solids are broken up consecutively, and the smaller they become, new surfaces appear and are covered by fats and emulsifiers (lipid phase) and the greater the resistance of the mass to flow (Talbot, 2009).

According to the International Office of Cocoa, Chocolate and Sugar Confectionery (IOCCC), it is recommended the use of BOB geometry or Cup (such as DIN cup) in rotation to evaluate the viscosity of cocoa and chocolate-based masses. These methodologies have been widely studied and accepted (Chevalley, 1975, 1991, and 1994; Bouzas & Brown, 1995; IOCCC, 2000).

The rheological characteristics of Flow Curve and Stress Sweep of the evaluated samples are shown in Figures 2 and 3.

The curvature of the line seen in Figure 2 between shear rate and stress had a similar behavior for all analyzed samples, which indicates a shear dilution behavior similar to that reported by Toker et al. (2016). This shear dilution behavior may result from structural breakage and molecule alignment due to the shear rate applied during the analyses. Thus, the shear rate level applied during a process (or even post-process such as molding and packaging) must be carefully selected in order to maintain the structural quality of the products (Toker et al., 2016; Izidoro, Scheer, Sierakowski, & Haminiuk, 2008; Fernandes, Müller & Sandoval, 2013).

Systems 2, 3, and 5 (Ball mill / BLC; Ball mill / Homogenizing conche; and Stone mill) showed the highest shear stress (Pa). On the other hand, Systems 1 and 6, which combined the refiner with homogenizing conche (2-roll and 5-roll refiners / Homogenizing conche and 5-roll refiner / Homogenizing conche) showed the lowest shear stress (Pa).

According to Afoakwa, Paterson, Fowler and Vieira (2008a), dv (0.9) is important in the grittiness and textural properties of chocolate. It is observed that System 5 (Stone mill) with the highest value of (dv (0.9)) (Table 3) also presents the highest shear stress (Figure 2).

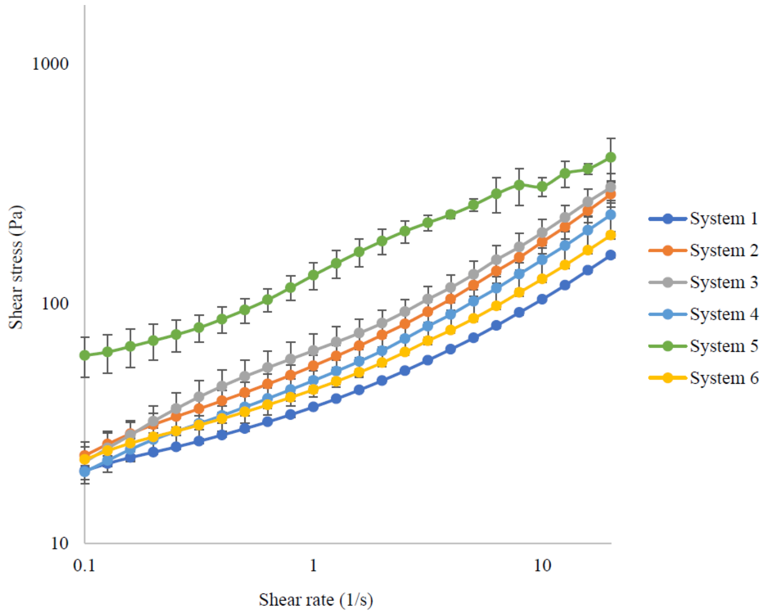

Figure 2. Flow curve obtained in analysis of rheological properties of dark chocolates produced in different refining / conching systems

Note: System 1: Double refining; System 2: Refining in ball mill; System 3: Refining in ball mill and BLC conching; System 4: Refining and conching in refining conche; System 5: Refining and conching in stone mill – melanger; System 6: Simple refining.

Source: Prepared by the author, 2021.

Usually, materials that possess shear stress are considered multiphase systems. For example, chocolate, which is a suspension of sugar and cocoa solids dispersed in cocoa butter. With the condition of mutual attraction, the particles interact and form flakes, which, in turn, interact to create a continuous three-dimensional network, which can prevent flow at low stress. The initial shear stress is associated with the transition between solid-elastic and liquid-viscous behaviors; however, this transition occurs in a stress range, in which the material exhibits viscous and elastic properties. A suspension only flows when the stress is sufficient to break the structure (Wilson, Speers & Tung, 1993; Liddell & Borger, 1996).

In the stress sweep test (Figure 3), similar behaviors can be observed because higher complex modulus (Pa) values are observed in the samples generated by Systems 3 (Ball mill/BLC) and 5 (Stone mill), in contrast with System 1 (2-roll and 5-roll refiners / Homogenizing conche), which presented lower values. The figure shows clearly that the refining system influences the curves of the complex modulus.

As the complex modulus is a measure of system rigidity, that is, a measure of the total resistance of a sample to deformation, it is observed that the structure of the chocolates produced by System 1 (2-roll and 5-roll refiners / Homogenizing conche) presents lower resistance to deformation. Systems like this also lead to reduced linear viscoelastic region (LVR), that is, the stress range within which the structure does not break.

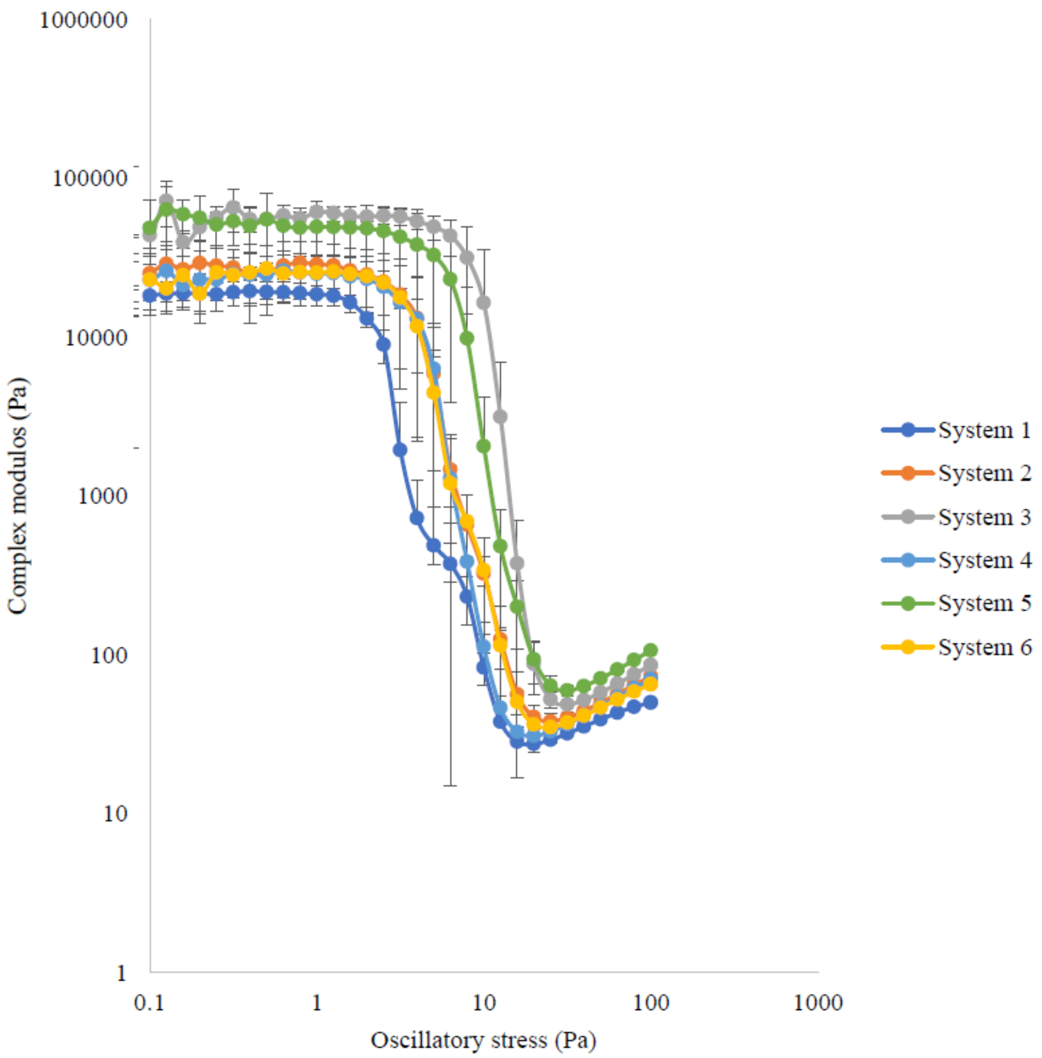

Figure 3. Stress sweep obtained in analysis of rheological properties of dark chocolates produced in different refining / conching systems

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Source: Prepared by the author, 2021.

By correlation with the results for particle size distribution (Figure 1 and Table 3), it is observed that System 1 (2-roll and 5-roll refiners / Homogenizing conche) and System 6 (5-roll refiner / Homogenizing conche) produced samples with lower viscosity, even not being the samples with higher values for dv (0.9). However, the chocolate produced in System 6 presented higher values for D[3,2] and dv (0.5), which suggests a strong correlation between these parameters and the decreased viscosity in the chocolates.

On the other hand, the samples produced in Systems 2 and 3 (Ball mill / Homogenizing conche and Ball mill / BLC) and System 5 (Stone mill) presented the highest values for D[4,3], dv (0.1) and dv (0.9), in addition to the highest values for shear stress (Pa) and complex modulus (Pa), which was already expected, since particle size distribution (in addition to the composition of ingredients) influences the rheological properties of chocolates, also affecting the final texture and melting profile, as already widely reported in the literature (Beckett, 2009; Glicerina, Balestra, Rosa & Romani, 2013; Toker et al., 2016). The basic ingredients of the chocolate formulation measure about 1 mm in diameter; however, particles larger than 0.03 mm result in the characteristic of a gritty chocolate. On the other hand, in the refining process, very fine particles can also be formed (less than 0.005 mm in diameter), but these particles also need to be coated with fat in order to flow, which means that if more fat is not added in the process, the chocolate becomes highly viscous and tends to melt in the mouth less easily.

Other studies, such as that conducted by Afoakwa, Paterson and Fowler (2008c), report that chocolate mass with 25% fat and 0.3% lecithin, which had particles of D90 equal to 50 μm (90% of the total and particles with diameter below 50 μm), had the viscosity doubled when the particles were reduced to 18 μm. The effect is due to greater dispersion of solids, causing a separation of particles, and less contact between solid particles.

Another study with a model system with different amounts of fats equivalent to cocoa butter and with mixtures of sugars of different particle sizes demonstrated that apparent viscosity at 40 ℃ decreases with increasing particle size in samples with more than 25% fat. Moreover, in samples with 22% fat, apparent viscosity decreased as the amount of larger particles increased (Do, Hargreaves, Wolf, Hort & Mitchell, 2007).

In fact, numerous studies have shown differences in rheological characteristics and mechanical strength as to chocolates. However, other studies also note that some characteristics of chocolates may not show a significant correlation with particle size distribution (Afoakwa, Paterson & Fowler, 2007b; Do et al., 2007; Afoakwa et al., 2008a; Afoakwa, Paterson, Fowler & Vieira, 2009a; Bolenz, Holm & Langskr är, 2014).

This reinforces the fact that dark chocolate has a complex rheological behavior, that is, it shows a flow stress (minimum amount of energy to start the flow) and a plastic viscosity (energy to keep the fluid in motion) that is very dependent on the manufacturing process (Bourne, 2002; Servais, Ranch & Roberts, 2004; Afoakwa, Paterson & Fowler, 2008c).

3.4 Sensory Analysis

The results of the sensory evaluation of chocolates subjected to different refining and conching methods are shown in Table 4.

The aroma, hardness, melting, and color attributes were not influenced by the refining/conching system used.

We observed higher scores for the “overall impression” attribute for samples produced in System 6 (5-roll refiner / Homogenizing conche), System 3 (Ball mill/BLC), and System 4 (Ball mill / Homogenizing conche).

In the evaluation of flavor, in addition to the three samples above, System 1 (2-roll and 5-rolls refiners / Homogenizing conche) also did not show statistical differences from those best evaluated. These values demonstrate changes in the sensory evaluation in relation to the different refining systems, even though they do not show a direct correlation with particle size distribution and rheology.

Glicerina et al. (2014) note that dark chocolate – due to being a complete matrix – is a product whose entire appearance depends strictly on the process steps, especially those related to particle size distribution.

Afokwa et al. (2009b) states that, in sensory evaluations, aroma release can also be affected by particle size distribution, as most of the identified aromatic components can increase with decreasing particle size.

Rheological changes can affect mouthfeel and flavor, because the time solid chocolate particles take to reach sensory receptors depends on chocolate viscosity (Beckett, 2001). Melting in the mouth is determined by the characteristics of the fat phase, which influence the characteristic flavor and textural attributes of chocolates. The intensity of flavor can change systematically as chocolate is melted, manipulated and mixed with saliva for swallowing (Beckett, 1999).

The evaluation of grittiness only showed a significant difference for System 5 (Stone mill), which did not differ from System 3 (Ball mill/BLC), but differed statistically from the other samples. These two samples, together with System 4 (Refining conche) showed the highest maximum particle sizes, as shown in Table 2.

As for the acidity attribute, System 4 (Refining conche) presented the worst results evaluated. On the other hand, the other samples did not differ statistically from one another, but with the main emphasis on Systems 2 and 3 (Ball mill / Homogenizing conche and Ball mill/BLC). These last two samples also showed lower value for total titratable acidity, differing statistically from the first sample, as shown in Table 2.

Table 4. Mean scores assigned by the evaluators in the sensory analysis of dark chocolates produced in different refining/conching systems

Attributes	**Mean sensory scores / Systems***						**F ****
	1	**2**	**3**	**4**	**5**	**6**	
Overall Impression	6.84^{b}	7.41^{a}	7.08^{ab}	6.57^{b}	6.54^{b}	6.99^{ab}	5.59
Aroma	6.95^{a}	7.10^{a}	7.20^{a}	6.84^{a}	6.97^{a}	6.95^{a}	1
Flavor	6.59^{abc}	7.20^{a}	6.96^{ab}	6.02^{c}	6.31^{bc}	6.64^{abc}	6.74
Hardness	7.28^{a}	7.02^{a}	7.06^{a}	7.16^{a}	6.88^{a}	7.20^{a}	1.01
Melting	7.26^{a}	7.26^{a}	7.03^{a}	7.08^{a}	6.82^{a}	7.16^{a}	1.40
Grittiness	7.16^{a}	7.34^{a}	6.95^{ab}	7.05^{a}	6.4^{b}	7.25^{a}	5.23
Acidity	5.89^{ab}	6.42^{a}	6.49^{a}	5.66^{b}	5.81^{ab}	5.85^{ab}	4.09
Color	7.78^{a}	8.09^{a}	7.88^{a}	7.80^{a}	7.68^{a}	7.98^{a}	1.62

Note: Equal letters in the same row indicate that the samples did not differ statistically in the Tukey Test at 5% significance.

*System 1: Double refining; System 2: Refining in ball mill; System 3: Refining in ball mill and BLC conching; System 4: Refining and conching in refining conche; System 5: Refining and conching in stone mill – melanger; System 6: Simple refining.

Source: Prepared by the author, 2021.

Overall, there is greater acceptance of System 2 (Ball mill / Homogenizing conche) in all aspects evaluated, since the sample differs statistically from the others with the highest scores, or at least in the best-evaluated group. This system also has the highest values for D[4,3], dv (0.1), and dv (0.9) in particle size distribution; in rheology, it has the highest values for shear stress (Pa) and complex modulus (Pa). Apparently, the value of humidity above the other treatments, does not provoke undesirable alterations in the mentioned parameters.

Ziegler, Mongia and Hollender (2001) observed that particle size and rheology significantly influenced melting time and sweetness of milk chocolate samples through the methodology of time and intensity.

The development of new chocolate-based products requires care as to their texture and melting profile, according to current knowledge on particle size distribution and ingredient composition. Because, even though textural perception is a dynamic oral process, individuals also perceive texture by means of vision, touch and hearing. Furthermore, chocolate consumers have well-formed opinions and expectations regarding the appropriate texture and melting characteristics (Wilkinson, Dijksterhuis & Minekus, 2000; Bourne, 2002; Varela, Salvador & Fiszman, 2007).

4. Conclusions

The evaluated refining systems did not lead to significant changes in the evaluated pH. On the other hand, total titratable acidity and moisture underwent changes only in System 2 (Ball mill/Homogenizing conche) with the lowest and highest values, respectively. The samples whose refining was combined with homogenizing conche (Systems 1, 2, and 6) also showed the smallest maximum particle size.

We observed considerable difference in particle size distribution by refining systems, and System 6 (5-roll refiner / Homogenizing conche) showed the lowest variation, that is, a more homogeneous process. This same system, with System 1 (Double refiner (2 and 5 rolls) / Homogenizing conche) also presented the lowest shear stress (Pa). This last one also showed the lowest value for complex modulus (Pa), which indicates a weakened sample with reduced linear viscoelastic region (LVR), which may be characteristic of refining systems with cooled rollers.

On the other hand, Systems 2 and 3 (Ball mill / Homogenizing conche and Ball mill/BLC) and System 5 (Stone mill) were more heterogeneous for particle size distribution. These samples also showed the highest shear stress (Pa) values in the rheological evaluation. In turn, we verified in the evaluation of complex modulus that two of the mentioned samples – Systems 3 and 5 (Ball mill/BLC and Stone Mill) – also presented the highest values found. While System 6 (5-roll refiner / Homogenizing conche) was the most homogeneous.

Sensory evaluation showed no significant differences for the refining systems as to the aroma, hardness, melting, and color attributes. In general, the worst results for overall impression and flavor attribute were observed for samples produced by Systems 4 and 5 (Refining conche and Stone mill). Moreover, System 4 (Refining conche) presented the worst results for the grittiness and acidity attributes.

Overall, Systems 1, 2, and 6 – which combine refining systems with homogenizing conche – proved favorable for obtaining chocolates with greater fluidity and better results in sensory evaluation.

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