Applicability of the melanger for chocolate refining and Stephan mixer for conching as small-scale alternative chocolate production techniques

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\textbf{A B S T R A C T}

Conventional dark chocolate production methods that are applied in the industry consist of several steps, which require equipment with big investment costs. There are however few cost-effective alternatives suggested for small-scale production. Meanwhile, knowledge on these alternative equipment/techniques are insufficient to promote miniature production of high quality chocolates, either for research purposes in an industrial context, or for producers in developing countries, where the cost of investing in conventional equipment seems to be an impediment. The aim of this study was two-fold; first, to assess the feasibility of utilizing the ECGC-12SLTA CocoaTown melanger as an alternative to the conventional 3-roll refiner at different settings and fat content. Thereafter, one optimal setting was selected for each equipment for further investigation on the impact of the refining on some quality attributes of the final dark chocolate (70% cocoa). Secondly, the Stephan mixer; being used to mimic a conching-like process, was assessed with respect to two processing factors; the dry conching temperature (60 °C, 80 °C) and the duration of vacuum pump connection (0, 30, 60 min). The latter was to facilitate adequate moisture removal. The melanger proved to be a suitable alternative to the 3-roll refiner, provided that refining was carried out at moderate/high (ca. 40%) fat content, as is often the case for “high-percentage-cocoa” chocolates. Refining for 180 min with the mini drum at 40% resulted in D (v,0.9) significantly (p < 0.05) lower than when the 3-roll refiner was used. Nonetheless, a comparative advantage of the latter would be its short throughput time (5–10 min). Due to a resultant linear speed gradient of the chocolate mass due to the cylindrical roller stones, a more efficient refining was achieved with the mini drum than with the big drum. More so, refining in excess fat (40%) may have contributed to a more efficient coating of the newly created hydrophilic sugar surfaces, thus, limiting the possibility for moisture-induced agglomeration as may have been the case for the recipe with 27% fat. In spite of trivial difference in moisture content, chocolates manufactured following melanger and 3-roll refining showed significant (p < 0.05) differences in terms of particle size, flow parameters and color. For chocolates that were conched with the Stephan mixer, the vacuum duration had a significant (p < 0.05) impact on moisture content and D (v,0.9). Also, an impact of all factors and their interaction on the Casson yield values of the chocolates was observed. However, these factors proved to be less important in dictating the final viscosities of the chocolates. Among others, it is suggested that the influence of the high fat content of the chocolates may have played a more important role. Although all chocolates exhibited less thixotropic behavior, a direct proportional relationship between the particle surface area and thixotropy was observed. Finally, the interaction effect of both factors also significantly (p < 0.05) influenced the color of the chocolates.

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1. Introduction

Chocolate is a complex suspension of solid particles (sugar, cocoa particles) dispersed in a continuous fat phase, mainly cocoa butter (Afoakwa et al., 2008). The growing popularity of chocolate revolves around its unique sensory and textural properties (Fowler, 2009; Torres-Moreno et al., 2015). However, the acclaimed health benefits attributed to chocolate in recent times have further promoted its demand among consumers (Steinberg et al., 2003; Latif, 2013).

The main conventional chocolate manufacturing process encompasses steps such as mixing, refining and conching. However, most classical processing equipment are designed to handle huge quantities either in a continuous or batch process. Regardless of some attempts to scale-down production, in practice, only a handful of equipment on the market have been recognized as suitable alternatives, considering the similitude of their final products to chocolates from a classical industrial process in such areas as quality and flavor attributes. Common examples of these include the ball mill and the ELK'olino conche. Whereas the former is often applied for grinding nibs into liquor, and in some cases, for alternative processing of chocolates/compounds, the latter has served as an ideal conching equipment handling approximately 5 kg per batch (Saputro et al., 2016a,b). The cost of investment associated with these equipment have made them unfavorable for simple laboratory and even miniature industrial productions - where only a small amount of chocolate needs to be produced from a limited quantity of available beans. Notably, this also seems to be the challenge for most small-scale bean-to-bar producers in various developing countries, such as Ghana, Ivory Coast, Indonesia and India. It is of no surprise that the idea of small-scale alternative processing has gained a lot of attention with some recent studies have been carried out on this topic (Bolenz and Manske, 2013; Fistes et al., 2013; Pajin et al., 2013; Saputro et al., 2016a,b; Tan and Balasubramanian, 2017; Saputro et al., 2018).

For instance, the introduction of coarse conching using the ball mill by Bolenz et al. (2014). It is apparent that the advantages associated with alternative processing, such as its time and energy efficiency, compact nature (ability to combine several processing steps), cost efficiency, as well as the need for less trained personnel may be some of the underlying factors driving its growing popularity and/or acceptance.

In this study, two types of such alternative processing equipment; the EGCG-12SLTA CocoaTown melanger (CocoaTown, Roswell, USA), and the Stephan mixer (Stephan food service equipment GmbH, Hameln, Germany) were explored to evaluate their feasibility and performance at different stages of the chocolate production process.

The Stephan mixer is an all-purpose, robust system, equipped with a double jacket and a tightly fitted lid for all kinds of food processing between 0.5 and 2.5 kg batch sizes. Here, mixing is achieved by means of a set of rotating knives which is propelled by a shaft through the bottom of the vessel and a reverse-acting scraper arm fitted through the lid. The equipment is also fitted with steam and pressure nozzles which allow both temperature and pressure to be controlled in the vessel during processing (Stephan, 2018). By studying the rheological properties of chocolates, Aidoo et al. (2014) provided some indication supporting the possibility of optimizing this equipment for use as an alternative conching device. They concluded that optimum settings of 65 °C for 10 min dry conching followed by 50 °C for 15 min wet conching both at blade rotary speed of 443 g yielded similar flow properties as the reference. However, information on some quality parameters such as moisture and particle size distribution (PSD), which have been demonstrated to have a huge impact on the flow behavior of chocolates, were missing. Saputro et al. (2016b) also successfully applied a combination of the Stephan mixer and ball mill in an alternative production of dark chocolates sweetened with palm sap-based sugar. Here, they used the Stephan mixer for both mixing and liquefaction, whereas refining was carried in the ball mill. The first stage of the mixing process was carried at 70 °C for 60 min with blade speed of 750 rpm. During the last 10 min of this process, a vacuum pump was activated in order to facilitate moisture removal. Thereafter, a second mixing step was carried out at 50 °C for 30 min with a blade speed of 1500 rpm. Next, the chocolate mass was refined with the ball mill for 30 min at 50 °C with maximum speed. Finally, liquefaction was performed at 50 °C for 15 min with a blade speed of 1500 rpm using the Stephan mixer.

The melanger, on the other hand, was initially developed for grinding, among others, dried seeds and nuts, as well as cocoa nibs. The equipment can either be operated with a big drum (with a maximum capacity of ca. 3.6 kg) or mini drum (with a maximum capacity of ca. 1 kg) (Fig. 1). The big drum is equipped with a set of conical granite roller stones, whereas, the mini drum operates with a set of cylindrical granite roller stones that rotate at 135–140 rpm on a granite slab - creating shearing forces - that crushes and reduces the particle sizes of cocoa solids during the grinding process (CocoaTown, 2017). This could result in particle sizes less than 20 μm after grinding/refining for about 8 h as indicated by Tan and Balasubramanian (2017). The intensity of the grinding/refining can be adjusted by regulating the tension between the roller stones and the slab. The minimal loss of sample makes the melanger more efficient in terms of sample recovery. Besides, its low investment cost, its compact configuration (takes up less than 0.14 m² of space) and acclaimed suitability for use at different stages of the chocolate production process, may account for its growing popularity and demand among small-scale chocolate manufacturers in recent times. The melanger however has some obvious flaws; chief of which is the lack of a temperature control unit. Hence, operating this equipment at room temperature in a conching-like process may limit the removal of moisture and undesirable flavors.

Fig. 1. CocoaTown melangers (a) mini drum with cylindrical roller stones and (b) big drum with conical roller stones.
the melanger, more comprehensive information including the different possibilities by which the melanger along with other equipment could be combined for small-scale chocolate production is scarce. The aim of this study was therefore to assess the applicability of the melanger and Stephan mixer for chocolate refining and conching respectively in various small-scale alternative chocolate manufacturing techniques whilst comparing their outcome to those from conventional processes.

2. Materials and methods

2.1. Sample preparation (from bean to liquor)

Fully fermented and sun-dried cocoa beans (hybrid type Forastero) were obtained from a farm in the Brong-Ahafo region of Ghana. Roasting was carried out at 135 °C for 35 min in a MIWE Roll-in-1.0608-TL baking oven (Miwe, USA). After cooling down to room temperature, beans were deshelled using a Winn-15 winnower (Cacao Cucina, U.S.A). Cocoa nibs (1.5 kg batch size) were first pre-broken using the Stephan mixer at 45 °C. First, 8 min at 50% speed, then, 6 min at 75% speed. Thereafter, nibs were then ground into liquor using ECGC-12SLTA CocoaTown melanger (CocoaTown, Roswell, USA). For this, the big drum (1.5 kg batch size) was used for a duration of 150 min in order to achieve a final particle size (D<sub>90</sub>) of 26.5 μm based on the finding from preliminary trials with both the mini and big drums (Appendix A).

2.2. Experimental design

The study was conducted in two set-ups. The first set-up was focused on the stage of refining, where both the mini and big drums of the melanger were studied in comparison to the 3-roll refiner for particle size reduction. The outcome were evaluated on the basis of particle size and microscopic imaging analyses. Thereafter, an optimal setting for the melanger was selected and further investigated in reference to the 3-roll refiner for the impact of the different refining equipment on some quality attributes of the final chocolate. The second set-up was also focused on the conching stage, where two processing parameters were explored using the Stephan mixer in a conching-like process. Hitherto, the impact of these processing parameters were also evaluated with respect to the same quality attributes of the end chocolates as in set-up 1. Summary of the processes involved in both set-ups have been outlined in Figs. 2–4.

2.2.1. Experimental set-up 1

2.2.1.1. Comparing melanger and 3-roll refiner for particle reduction during chocolate refining. Two different recipes were tested at different settings of the ECGC-12SL CocoaTown melanger (CocoaTown, Roswell, USA) and the Exakt 805 3-roll refiner (Exakt Technologies, Inc., USA) (Fig. 2). Thereafter, particle size distribution (PSD) of samples before and after refining were analyzed and compared. The first recipe had a total fat content of 27% - being optimal for the 3-roll refiner, whereas the second recipe, also referred to as “full fat”, had a total fat content of 40% as is recommended for the smooth running of the melanger. The amounts of cocoa liquor and sugar needed were calculated on the account of the previously determined fat content of the cocoa liquor. For the recipe 1 (27% fat), 900 g of sugar and 780 g of cocoa liquor were mixed for 20 min in a VEMA BM 30/20 planetary mixer (Machinery Verhoest NV/Vema Construct, Izegem, Belgium) at a constant temperature of 45 °C. Whereas, for recipe 2 (40% fat), 900 g sugar was mixed with 1939.5 g cocoa liquor using the same mixing equipment/process.

The 3-roll refiner was operated at a constant temperature of 35 °C and roller speed of 400 rpm with two different gap settings 3 - 1 and 2 - 1. This means that the gap in between the first two rolls was adjusted to either 3 or 2, while the gap in between the second and last roll was always kept constant at 1. However, refining in the melanger (both mini and big drums) was also carried out at 1.5, 3.0 and 4.5 h durations. Since the melanger operates at room temperature (ca. 27 °C), it was necessary to monitor the temperature of the refining chocolate mass during the entire process. Generally, temperature rose to approximately 40 °C due to internal friction. However, at fat content of 27%, refining beyond 3 h with the mini drum resulted in a dramatic rise in temperature (= 78 °C) of the chocolate mass. Meanwhile, the maximum temperature allowed for the operation of the melanger, according to the manufacturer is 80 °C. For this reason, the process was discontinued beyond 3 h. For the same reason, refining with the big drum at 27% fat was not carried out in order to avoid the risk of damage through overheating. Following investigations by both PSD and microscopic analyses, one optimal setting from each equipment was selected for further chocolate production. For the 3-roll refiner, the gap setting 2–1, at 27% fat was selected, whereas, for the melanger, refining with the mini drum for 3 h at 40% fat was selected. Selection was done on the basis of maximum particle reduction in both equipment, and additionally, in the case of the melanger, for the shortest possible refining time.

2.2.1.2. Chocolate production. Two batches of 70% dark chocolate (total fat = 43%) consisting of 30% pre-broken sugar (Barry Callebaut Belgium, Wieze, Belgium), 64.65% cocoa liquor, 5% cocoa butter (Puratos - Belocola, Eremondgelem, Belgium) and 0.35% soy lecithin (Soya International Ltd, Cheshire, U.K.) were produced on a 5 kg scale (Figs. 3 and 4). Mixing was carried out using the VEMA BM 30/20 planetary mixer (Machinery Verhoest NV/Vema Construct, Izegem, Belgium) for a duration of 20 min at 45 °C. For the first batch, the mixed ingredients (27% fat) was refined with the Exakt 805 3-roll refiner (Exakt Technologies, Inc., USA) at gap setting 2–1, roller speed of 400 rpm and temperature of 35 °C (referred to as choc 1). However, the second batch of mixed ingredients (40% or full fat) was refined using the mini drum of the ECGC-12SLTA CocoaTown melanger (CocoaTown, Roswell, USA) for a duration of 3 h (referred to as choc 2). In each case, the resulting refined chocolate mass was conched in a Bühler ELKólino conche (Richard Frisse GmbH, Bad Salzuflen, Germany) in two phases. The dry phase was carried out at 60 °C with 1200 rpm for 2 h (clockwise) and 80 °C with 1200 rpm for 4 h (anti-clockwise). At the liquid phase, calculated amounts of pre-conched cocoa liquor, cocoa butter and the soy lecithin were added, such that, the final fat content of the chocolate was 43%. Here, the process was carried out as follows; 45 °C with 2400 rpm for 15 min (clockwise) and 15 min (anti-clockwise). Specifically in the case of choc 1, pre-conching of part of the cocoa liquor was necessary since the entire amount of cocoa liquor required to produce final chocolate consisting of 70% cocoa could not be included in the recipe before refining. This is because; for the refining mass, a final fat content of 27% was required for optimal processing by the 3-roll refiner. Hence, for this batch, a calculated amount of cocoa liquor was previously dry-conched using the same dry conching procedure, which was then subsequently added at the stage of liquefaction in order to make up for this final concentration. However, for choc 2, this was not the case because, here, since the melanger refines at full fat, the entire amount of cocoa liquor required for the production of 70% cocoa chocolate could be included at the mixing stage prior to refining.

2.2.2. Experimental set-up 2

Six 70% dark chocolates were produced with the same composition of ingredients just like choc 2. However, these were produced on a 1 kg scale due to the capacity of the Stephan mixer. Mixing of the sugar and cocoa liquor was carried out in a Hobart mixer (Troy, USA) for a duration of 20 min at 45 °C just like in the VEMA mixer. However, here, the temperature of the mixing ingredients was maintained by means of a heat gun (BOSCH, Germany). Thereafter, the mixed ingredients was refined in the mini drum of the ECGC-12SLTA CocoaTown melanger (CocoaTown, Roswell, USA) as described in set-up 1. Next, the Stephan
Fig. 2. Chocolate refining using melanger and 3-roll refiner.

![Diagram of chocolate refining](image1)

NOTE: “Full fat” refers to fat content of 40% as is recommended for smooth running of the melanger.

Fig. 3. Outline of chocolate productions using conventional and alternative means.

![Diagram of chocolate production](image2)

NOTE: “Full fat” refers to fat content of 40% as is recommended for smooth running of the melanger.
mixture (STEPHAN food service equipment GmbH, Hameln, Germany) was used for mimicking the conventional conching process. For dry conching, it was operated at blade speed of 1500 rpm for a duration of 60 min. In order to facilitate moisture reduction and the loss of unwanted volatiles during the process, a vacuum pump (KNF Neuberger, Inc., USA) was connected to the Stephan mixer as shown in Fig. 4. The pump was operated at –0.7 bar. Here, a 2 × 3 full factorial design was used, consisting of dry conching temperatures; 60 °C and 80 °C, and vacuum durations, 0, 30 and 60 min. After the dry conching, the vacuum pump was detached, the required amount of cocoa butter and lecithin were then added to the chocolates. Liquefaction was then carried out at 50 °C for 15 min with blade speed of 1500 rpm according to Saputro et al. (2016b). The resulting chocolates were hereby referred to as choc 3A, 3B, 3C, 3D, 3E, and 3F representing dry conching conditions (temperature/vacuum duration); 60 °C/0 min, 60 °C/30 min, 60 °C/60 min, 80 °C/0 min, 80 °C/30 min, and 80 °C/60 min respectively.

2.3. Light microscopy

Microstructures of cocoa liquor, the different refined chocolate masses and finished chocolate were observed using a Leica DM2500 microscope (Wetzlar, Germany) equipped with a temperature controlled sample holder (Linkam Scientific Instrument Ltd, Surrey, UK). Samples were observed under both normal and polarized light (magnification = 20 ×). In the case of the former, 0.5 g of each molten sample was first diluted in 10 ml isopropanol (VWR, Leuven, Belgium), homogenized by shaking and then after, a representative drop was brought to the glass slide using a Pasteur pipette. A cover slip was then carefully placed on the sample. It was then mounted on the sample holder (isothermal at 50 °C) for visualization. However, samples were observed “as-is” under the polarized light.

2.4. Scanning electron microscopy (SEM)

In order to enhance visualization, the samples were first partially defatted by dissolving 0.5 g sample in 10 ml isopropanol (VWR, Leuven, Belgium), filtered over Whatman No. 40 filter paper and dried in an oven at 50 °C for 1 h. The surface morphology of the partially defatted cocoa liquor and chocolate were then visualized using a JSM-7100 F TTLS LV TFEG-SEM (Jeol Europe BV, Zaventem, Belgium) under high vacuum and at an accelerated voltage of 2 keV. Prior to electron beam targeting, the samples were vitrified in liquid nitrogen and transferred to a PP3000T device (Quorum Technologies Ltd., East Sussex, UK) at –140 °C. Here, the samples were allowed to sublime for 15 min at –70 °C in order to remove frost artifacts. Prior to the transfer from the cryo-preparation room to the SEM chamber, a thin layer of a conductive metal (Pt) was deposited on the samples. This sputter coating process prevents charging of specimens with an electron beam.
2.5. Particle size distribution (PSD)

Particle size distribution (PSD) of refined masses and final chocolates were determined with a Master Sizer S (Malvern Instruments Ltd., Worcestershire, UK), a laser diffraction particle size analyzer, equipped with a 300 RF lens and an active beam length of 2.4 to measure particles in a range of 0.05–900 µm. First, 0.5 g of each molten sample was diluted with 10 ml of isopropanol (VWR, Leuven, Belgium) and placed in an oven at 50 °C for an hour. Then the suspension was further diluted with 10 ml of isopropanol and placed in an oven at 50 °C before sampling 20 g for analysis. After, the data was fitted to the Casson model (Equation (1)) from which the Casson yield value (τCA) and Casson plastic viscosity (ηCA) were obtained. The value of thixotropy was also obtained by computing the difference between the shear stress at 5 s⁻¹ for ramp up and ramp down measurements (Afoakwa, 2010). All analyses were done in triplicate.

\[
\sqrt{\tau} = \sqrt{\tau_{CA}} + \sqrt{\eta_{CA} \cdot \dot{\gamma}}
\]  

(1)

Casson model (\(\tau = \) shear stress, \(\tau_{CA} = \) Casson yield value, \(\eta_{CA} = \) Casson plastic viscosity, \(\dot{\gamma} = \) shear rate).

2.8. Color

Color parameters of the molten chocolates were determined using a Minolta Model CM-2500D spectrophotometer (Konica Minolta Sensing, Inc., Osaka, Japan). Only values of the SCE (Specular Component Excluded) were considered as these are claimed to be more correlated with observations of the human eye. The color was expressed in terms of L* (lightness component), a* (green to red component) and b* (blue to yellow component). Here, five repeated measurements were taken per replicate of a sample.

2.9. Statistical analysis

Statistical analysis was performed with Minitab 18 (Minitab Inc, USA). For set-up 1, the different responses were subjected to Analysis of Variance (ANOVA) with a 5% significance level. Assumptions of normality and equality of variance were tested prior to the analysis using Kolmogorov-Smirnov test and Modified Levene's test, respectively. Where assumptions were fulfilled, a non-parametric alternative, Welch was used along with Games Howell post-hoc test. In set-up 2, a general linear model (GLM) was used to explore the impact of factors (conching temperature and vacuum duration) and their interaction effect on the various responses. Here, assumptions of normality of residuals, linearity of the covariate effects and constancy of the variance were also verified.
3. Results and discussion

3.1. Particle properties of refined chocolate mass using melanger and 3-roll refiner

3.1.1. Particle size distribution

From Table 1, a clear downward trend in PSD with refining time was observed. Also, at the different fat contents, both mini and big drums proved to be effective in significantly (p < 0.05) reducing particle size with increasing refining time. Consequently, SSA also increased. Meanwhile, for a given duration, particle size parameters were slightly lower in the mini drum compared to the big drum. This may be attributed to the difference in geometry of the roller stones (Fig. 1 and B.1), as it dictates the linear speed gradient of product during the refining process. Tan and Balasubramanian (2017), who reported similar findings, suggested that at constant rotational speed of the roller stones, the linear speed of the product at the outer edge of the cylindrical roller stones in the mini drum is greater than that at the inner edge due to the difference in distance between the two edges of the roller stones to the center of the drum. This therefore creates a resultant linear speed gradient between the refining mass at the center and that at the walls of the rotating drum. It is suggested that this gradient consequently introduces shearing forces which may have contributed to the crushing of more cocoa and sugar particles. Meanwhile, in the case of the big drum, which is equipped with conical roller stones, an opposite scenario is observed where almost no or very limited gradient is created during the refining process, hence, less shearing forces are generated leading to relatively minimal particle reduction for the same duration of refining. Although the differences in the amount of samples used in both drums may have also contributed to this observation, early work by Tan and Balasubramanian (2017) during grinding of cocoa nibs proved that this was indeed insignificant, in comparison to the impact of the geometry of the roller stones.

Comparing the different settings of the 3-roll refiner, it was evident that the smaller the roller gap, the smaller the particle size due to a more effective crushing of the solid particles. According to Do et al. (2007), optimum particle size of dark chocolate after refining should be < 30 μm, as larger particles result in gritty mouth feel. Among other PSD parameters, the D (v, 0.9), being an estimation of the largest particle sizes, may be used to represent the fineness of the chocolate, since this has been demonstrated to correlate well with human perception (Beckett, 2009). Considering the values of D (v, 0.9) from Table 1, for the full fat recipe, these were obtained after 3 h and 4.5 h of refining with the mini and big drums respectively. Additionally, at both fat contents, the D (v,0.9) from setting (2-1) of the 3-roll refiner were also sufficiently reduced. The differences in particle sizes of refined chocolate masses from both melanger and 3-roll refiner could be due to the differences in both the equipment and the throughput time. Whereas the former was operated between 1.5 and 4.5 h, it only took 5–10 min to refine the same amount of product with the latter.

3.1.2. Microscopy

Since dark chocolate consists of 65–75% suspended solid particles, the sizes of these particles will have a huge influence on the microstructure, and subsequently, other physical and flow properties of the final chocolate (Afoakwa, 2010). The shapes and sizes of the particles that form the chocolate matrix originate from the ingredients; in this case, the refined cocoa solids and sugar particles in the matrix. Hence, in order to understand the evolution of the microstructural changes during the refining process, three microscopic techniques were applied, namely; Normal Light Microscopy (NLM), Polarized Light Microscopy (PLM) and Scanning Electron Microscopy (SEM). First a deeper understanding of the microstructure was sought by comparing the initial cocoa liquor to that of the final chocolate, obviously, with the presence of sugar particles being the difference between the two matrices. After this one technique was chosen to follow through the different refining processes.

From Fig. 5, a clear difference is seen. NLM revealed the microstructure of cocoa liquor being dominated by black circular spots representing solid cocoa particles with sizes range < 20 μm. However, in the chocolate, additional sugar particles are represented by clear crystals with jagged irregular shapes and sharp edges which consist of approximate sizes ≤ 30 μm. This irregular breaking pattern is due to their brittle nature under mechanical stress during the process of refining (Beckett, 2009). Similarly, this difference is also seen from PLM although under polarized light, only the presence of sugar crystals are seen in the chocolate with a dense microstructure. Images from SEM were more detailed; consisting of a more loose packing of cocoa particles of different sizes and shapes overlaid with some amount of fat crystals due to partial defatting of the samples. Additionally, other ellipsoidal components within the matrix which appeared to retain their shape in spite of the grinding and refining processes were observed. According to Beckett (2009), milled cocoa particles appear as small platelets, but also included are cocoa starch granules. Of these, the starch granules are known to contribute roughly 7% of the weight of the liquor. Their sizes range from 2 to 12.5 μm, thus, due to their small sizes, making it possible for them to retain their ellipsoidal shape even after milling or refining. The same was confirmed by Afoakwa (2016), who reported 6.1% starch granules in dried Forastero cocoa beans. Unlike the cocoa liquor, a large (≤ 30 μm), broken sugar particle with a somewhat rectangular shape was additionally found in the SEM image of the chocolate. Similar crystalline sugar particles have also been described by Saputro et al. (2017) who studied sugar crystals with the SEM.

Indeed, as long as cocoa liquor has been sufficiently grinded, the sole purpose of refining is to reduce the size of sugar particles. As indicated earlier, the initial liquor consisted of an average particle size; D (v,0.9) = 26.5 μm. This means that except for the samples refined beyond 3 h at full fat using the mini drum, the other processes were less effective in further reducing the particle size of the cocoa solids during the process. Notwithstanding, NLM images (Figs. 6 and 7) of the various samples were also found to be in agreement with the earlier trends from Table 1.

3.2. Chocolate quality attributes as affected by different processing equipment or techniques

3.2.1. Moisture content

The moisture content of chocolate is known to be a key factor influencing its rheological and textural attributes. Comparing the chocolates from set-up 1, no significant (p > 0.05) difference between moisture contents of the final chocolates was found in spite of the refining technique used. This is probably due to the fact that both chocolates were conched with the same equipment. However, in the case of set-up 2, a demonstration of the removal of moisture due to the conching parameters was observed in spite of the initial moisture contents of the cocoa liquor and pre-broken sugar, being (1.99% ± 0.11) and (0.26% ± 0.01) respectively. Here, the moisture contents of the chocolates ranged from 0.52% ± 0.03 in choc 3F to 1.03% ± 0.09 in choc 2F (Table 2). A significant (p < 0.05) decline in moisture content was observed with increasing duration of the vacuum pump (Table 3). This is because at fixed temperature, the decrease in pressure due to the vacuum, may have resulted in a consequent decrease in the boiling point of water, thereby facilitating its evaporation from the chocolate matrix. As such, the longer the duration of the vacuum created by the pump, the higher the tendency for moisture removal, hence, the lower resulting moisture content of the chocolates. Nevertheless, the moisture content was trivial between the two conching temperatures for the same duration of vacuum pump connection. This is evident from Table 3, where, only the vacuum duration showed significant (p < 0.05) impact on the moisture content. Also, there was no significant (p > 0.05) interaction effect. Interestingly, the moisture
contents of all chocolates were within the range of 0.5–1.5% as recommended by Afoakwa (2010) to be an acceptable range without drastic effects on their flow properties.

3.2.2. Particle size distribution

Considering set-up 1 from Table 4, the chocolate refined with the melanger recorded significantly (p < 0.05) smaller particle parameters than the chocolate refined with the conventional 3-roll refiner. This obviously implies a notable impact of the type of equipment/technique in dictating the final particle size of the chocolate. Similar to an earlier observation in Table 1 and Fig. 7, it is possible that the presence of excess fat available within the matrix of choc 2 (refined at full fat) may have been responsible for coating the newly created sugar surfaces in the system, thereby, limiting the possibly of water-induced agglomeration through moisture reabsorption from either the ingredients or the surrounding (Beckett, 2009; Saputro et al., 2016a,b). However, this was likely the case in choc 1 which was roll refined at 27% fat content. Additionally, as earlier suggested, the different durations employed for the refining processes could have also contributed partially to this observed difference.

Considering set-up 2, it can be observed from Table 3 that whereas dry conching temperature only had significant (p < 0.05) impact on D (4,3), D (v, 0.1) and D (v, 0.5), all particle parameters but D (v, 0.1) were significantly (p < 0.05) influenced by vacuum duration. Of these, only D (3,2), D (v, 0.1) and the SSA recorded significant (p < 0.05) interaction effects of the two factors. As stated earlier, the D (v, 0.9) generally shows a direct proportional relationship with other PSD parameters, except for the SSA, in which case, the opposite is observed. It is therefore highly essential in explaining the finesses of the chocolates as perceived by consumers. The D (v, 0.9), decreased significantly (p < 0.05) with increasing vacuum pump duration for the two conching temperatures (Table 4). In retrospect, a similar decreasing trend in moisture content with increasing vacuum pump duration was initially observed (Table 2). Afoakwa (2010) explained that the presence of moisture in chocolate promotes various interactions among hydrophilic particles, such as aggregation of sugar particles resulting in lump formation with a consequential increase in particle sizes. This idea may explain the possible effect of the moisture content on the decline in particle sizes as the duration of vacuum pump connection was increased. Hereby, the more moisture was removed by the action of the

Fig. 5. Images of NLM (top), PLM (middle) and Cryo-SEM (bottom) showing the microstructure of cocoa liquor (left) and chocolate (right). A: cocoa particles overlaid with fat crystals; B: cocoa starch granules; C: sugar particle.
vacuum pump, the lesser the tendency to promote agglomeration among the hydrophilic sugar particles. Interestingly, the aforementioned trends are fairly reflected in the other particle size parameters as well.

### 3.2.3. Flow behavior

The rheology of chocolate is one of the crucial quality attributes as it gives an indication of its flow behavior under various conditions. Molten chocolate is a non-Newtonian fluid whose flow properties; e.g. yield stress and viscosity, are largely dependent on the composition and interactions between the constituents of the chocolate. Of these, the importance of such factors as PSD, fat content, moisture, amount and type of emulsifier as influenced through various processing steps (refining, conching, tempering) cannot be overemphasized (Vavreck, 2004; Schantz and Rohm, 2005).

The yield value according to Beckett (2009), corresponds to the stress needed to initiate flow and it is mainly determined by the particle-particle interactions in the microstructure of the chocolate. Different chocolates may have different yield values, depending on their specific composition and application. From set-up 1 (Table 2), an impact of the refining equipment/technique on yield value is exhibited, whereby choc 2 recorded a significantly ($p < 0.05$) higher yield value than choc 1. This is due to the smaller particle size with higher SSA in the former. The work of Bolenz and Manske (2013) provided insights on the linear relationship between the yield value and SSA of the chocolate. They explained that a higher yield value was as a result of the existence of smaller particle sizes with higher SSA, engaging in various particle-particle interactions. This results in a more rigid microstructure which is resistant to the initiation of flow. Unlike, in set-up 1, no clear trend in yield value was found from the chocolates in set-up 2. The reasons for this could have been due to the combined effect of moisture and particle size distribution of the chocolates, both of which have opposite influence on yield value. Notwithstanding, choc 3D – 3F seemed to reveal a slight decreasing trend in yield value with increasing vacuum duration. With this trend being similar to that of the moisture content, we may suggest that in this instance, the impact of moisture on the matrix may have played a more important role in dictating the yield value of the chocolates. This is also evident from Table 3, where all factors and their interaction effect – both contributing to moisture reduction – also contributed significantly ($p < 0.05$) to the observed

![Fig. 6. NLM images revealing sugar and cocoa particles during refining with melanger.](image-url)
outcome.

The viscosity of the chocolate describes its resistance to flow once the motion has been initiated. Contrary to the yield value, chocolate 2 with lower particle size also recorded the lowest viscosity. According to Chevalley (1999), the PSD is more pronounced on the yield value than the plastic viscosity of the chocolate. Hitherto, the free fat content, consistency and arrangement packing arrangement of the particles play a major role. Withal, Beckett (2009) recognized that unlike the yield value, a phenomenon where the viscosity decreases amidst fine particles is not surprising. He ascribed this to the packing arrangement of the suspended solids and the increasing amounts of unbound fat in the chocolate system making it possible for particles to slide over each other with ease during motion. Whilst studying the impact of particle size on the Casson yield and viscosity of chocolate, it was observed that a unimodal distribution resulted in a much lower effect on the flow parameters than in the case of a bimodal distribution. Moreover, whereas no effect was seen on the viscosity at fat content beyond 34%, the impact on the yield value rather persisted to 45% fat content. Thus, at high fat content, the excess amount of free fat in the system plays a more important role in determining the viscosity of the chocolates. In this study, the chocolates, were manufactured with a total fat content of 43%. This suggests that the chocolate with smaller particles may be less likely to impede motion within the chocolate. More importantly, contrary to the 3-roll refiner, it appears that the ability of the melanger to sufficiently coat most of the newly formed sugar surfaces with the extra fat, may have played an additional role in viscosity reduction by way of keeping the hydrophilic sugar particles further apart. This may also be responsible for the observed outcome of chocolates from set-up 2, where all chocolates had comparable viscosities. Here, in spite of the slight differences in particle sizes (Table 4), the more efficient coating of particles due to the same refining technique using the melanger and the additional impact of high fat content of the chocolate may have been chiefly responsible for the viscosities of these chocolates. Hitherto, the difference between the observed trend in set-up 1 as opposed to that of set-up 2, can be attributed to the different refining and conching equipment used. From Table 3, it was therefore not surprising both factors and their interaction appeared to have no significant (p > 0.05) impact on the Casson viscosities of the chocolates. Obviously, only 29%

Table 2

<table>
<thead>
<tr>
<th>Chocolates</th>
<th>Moisture (%)</th>
<th>Casson yield value (Pa)</th>
<th>Casson viscosity (Pa.s)</th>
<th>Thixotropy (Pa)</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Set-up 1</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Choc 1</td>
<td>0.63 ± 0.11a</td>
<td>1.8 ± 0.0b</td>
<td>1.8 ± 0.0a</td>
<td>0.8 ± 0.0b</td>
<td>19.4 ± 0.4a</td>
<td>12.7 ± 0.2a</td>
<td>13.7 ± 0.7a</td>
</tr>
<tr>
<td>Choc 2</td>
<td>0.62 ± 0.16a</td>
<td>3.4 ± 0.1a</td>
<td>1.3 ± 0.0b</td>
<td>1.0 ± 0.1a</td>
<td>22.5 ± 0.6a</td>
<td>16.6 ± 0.3a</td>
<td>9.4 ± 0.5a</td>
</tr>
<tr>
<td><strong>Set-up 2</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Choc 3A</td>
<td>1.03 ± 0.09a</td>
<td>3.0 ± 0.1BC</td>
<td>1.4 ± 0.0a</td>
<td>0.9 ± 0.0b</td>
<td>18.9 ± 0.2a</td>
<td>13.2 ± 0.1a</td>
<td>14.7 ± 0.4c</td>
</tr>
<tr>
<td>Choc 3B</td>
<td>0.82 ± 0.05b</td>
<td>1.4 ± 0.0a</td>
<td>0.9 ± 0.0b</td>
<td>19.6 ± 0.3a</td>
<td>13.6 ± 0.1a</td>
<td>16.1 ± 0.4a</td>
<td></td>
</tr>
<tr>
<td>Choc 3C</td>
<td>0.62 ± 0.03c</td>
<td>1.4 ± 0.0a</td>
<td>0.9 ± 0.0b</td>
<td>18.8 ± 0.1a</td>
<td>13.2 ± 0.1a</td>
<td>15.4 ± 0.2c</td>
<td></td>
</tr>
<tr>
<td>Choc 3D</td>
<td>1.01 ± 0.02a</td>
<td>1.4 ± 0.0a</td>
<td>0.7 ± 0.1c</td>
<td>18.9 ± 0.1a</td>
<td>13.6 ± 0.1a</td>
<td>16.1 ± 0.4a</td>
<td></td>
</tr>
<tr>
<td>Choc 3E</td>
<td>0.74 ± 0.03c</td>
<td>1.4 ± 0.0a</td>
<td>0.9 ± 0.1ab</td>
<td>19.4 ± 0.1a</td>
<td>13.1 ± 0.0a</td>
<td>14.3 ± 0.2c</td>
<td></td>
</tr>
<tr>
<td>Choc 3F</td>
<td>0.52 ± 0.03d</td>
<td>2.9 ± 0.1CD</td>
<td>1.4 ± 0.0a</td>
<td>1.0 ± 0.1a</td>
<td>19.0 ± 0.9a</td>
<td>13.3 ± 0.1a</td>
<td>15.4 ± 0.2b</td>
</tr>
</tbody>
</table>

Different alphabets (lowercase: set-up 1 and uppercase: set-up 2) in each column indicate significant differences (p < 0.05).
of the variability could be explained, leaving 71% to other factors (suggestively, the impact of fat content and refining equipment) which were not considered in the model.

Thixotropy is a unique property used to describe the efficiency of the processing equipment/technique to coat the surfaces of the suspended particles in the system with the continuous fat phase. Generally, Table 2 revealed that chocolates with smaller particles (higher SSA) also exhibited high thixotropic behavior, although, statistically, there is no clear trend in the case of the chocolates from set-up 2. It is however clear that the higher the extent to which larger particles are fragmented into smaller ones during the processing, the greater the need to coat these new surfaces with the continuous fat phase. It is expected that a well-conched chocolate exhibits minimal thixotropic behavior (Servais et al., 2002; Afoakwa, 2010). Interestingly, in spite of the equipment/technique applied, all chocolates proved to be less thixotropic with values ≤ 1 Pa.

3.2.4. Color

The color is a key contributor to the appearance of the chocolate and influences the perception of consumers. In comparison to the values in Table 2, the initial liquor (L* = 15.24 ± 0.10, a* = 12.58 ± 0.08, b* = 12.36 ± 0.07) appeared much darker (lowest L* value) than the chocolates. Similarly, a* and b* components had lower values. This is obviously due to the composition as no sugar has been added to the liquor. As sugar is added, there appears to be a "dilution" effect on the intensity of the color of the chocolates. From set-up 1, choc 2 (refined with the melanger), which recorded smaller particle size, also appeared lighter (higher L* value) with lower redness (a*) and yellowness (b*). Afoakwa et al. (2008) explained that smaller particles promote a more dense packing that scatters more light, and therefore leads to a lighter appearance. Chocolates from set-up 2 - which were conched with the Stephan mixer - showed no clear trend in terms of the color components. This may be indicative of the comparable color attributes of these chocolates.

4. Conclusions

The CocoaTown melanger could be considered as an ideal alternative to the 3-roll refiner at the refining step during small-scale chocolate production, provided that a recipe with moderate to high fat content (ca 40% fat) is targeted. Refining for 3 h with the mini drum at full fat resulted in D (v,0.9) significantly (p < 0.05) lower than when the 3-roll refiner was used. Nonetheless, a comparative advantage of the latter would be its short throughput time. Whereas the former required 3 h of refining time, the latter required about 5–10 min of operation time to refine the same amount of chocolate. Generally, a better refining was achieved with the mini drum than for the big drum. This is due to the difference in linear speed gradient of the product during the refining process, owing to the difference in geometry of the roller stones. Also, refining with a full fat recipe (40% fat) may have contributed to a more efficient coating of the newly created sugar surfaces in the presence of excess fat in the system. In spite of trivial difference in moisture content, chocolates manufactured following melanger and 3-roll refining showed significant (p < 0.05) differences in terms of PSD, flow parameters and color. In this study where the Stephan mixer was employed on a small-scale for conching dark chocolate, both processing factors; dry conching temperature and vacuum pump duration proved to be significant in dictating the various quality attributes of the end chocolates. Whereas the vacuum duration had a significant (p < 0.05) impact on moisture content and D (v,0.9), a consequential impact of all factors and their interaction on the Casson yield was observed. On the contrary, these factors proved to be less important in dictating the final viscosities of the chocolates. Among others, it is suggested that the role of the high fat content of the chocolates may have played a more important role. Although all chocolates exhibited less thixotropic behavior, a direct proportional relationship between the particle SSA and thixotropy was observed. Finally, it is possible that the impact of the vacuum pump on the rigidity of the chocolate matrix through moisture removal during conching may have contributed to the various color intensities of the chocolates. However, the interaction effect of both factors also proved significant (p < 0.05) for all color

Table 3

ANOVA summary showing F-values of chocolate quality attributes with varying dry conching temperature and vacuum pump duration using the Stephan mixer.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Moisture (%)</th>
<th>PSD profile</th>
<th>Flow parameters</th>
<th>Color values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>D (4,3) (μm)</td>
<td>D (3,2) (μm)</td>
<td>D (v,0.1) (μm)</td>
</tr>
<tr>
<td>T</td>
<td>8.65</td>
<td>19.11*</td>
<td>2.67</td>
<td>8.53*</td>
</tr>
<tr>
<td>V</td>
<td>138.44*</td>
<td>27.35*</td>
<td>4.48*</td>
<td>4.56</td>
</tr>
<tr>
<td>T x V</td>
<td>1.5</td>
<td>0.56</td>
<td>7.13*</td>
<td>4.16*</td>
</tr>
<tr>
<td>R² (%)</td>
<td>96.0</td>
<td>86.2</td>
<td>68.3</td>
<td>64.4</td>
</tr>
</tbody>
</table>

T = dry conching temperature (°C); V = vacuum pump duration (min); regression coefficients with (*) are significant at α = 0.05.

Table 4

Particle size profiles of chocolates produced through different processing means.

<table>
<thead>
<tr>
<th>Chocolates</th>
<th>Derived Diameter</th>
<th>Distribution Percentiles</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>D(4,3) (μm)</td>
<td>D(3,2) (μm)</td>
<td>D(v,0.1) (μm)</td>
</tr>
<tr>
<td>Set-up 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Choc 1</td>
<td>12.38 ± 0.22a</td>
<td>2.63 ± 0.08a</td>
<td>1.42 ± 0.03a</td>
</tr>
<tr>
<td>Choc 2</td>
<td>11.08 ± 0.05a</td>
<td>2.60 ± 0.07a</td>
<td>1.34 ± 0.01a</td>
</tr>
<tr>
<td>Set-up 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Choc 3A</td>
<td>12.69 ± 0.08a</td>
<td>2.68 ± 0.04ab</td>
<td>1.38 ± 0.03ab</td>
</tr>
<tr>
<td>Choc 3B</td>
<td>12.50 ± 0.17a</td>
<td>2.70 ± 0.07a</td>
<td>1.41 ± 0.02a</td>
</tr>
<tr>
<td>Choc 3C</td>
<td>11.93 ± 0.04ab</td>
<td>2.51 ± 0.03bc</td>
<td>1.31 ± 0.03ab</td>
</tr>
<tr>
<td>Choc 3D</td>
<td>12.37 ± 0.26ab</td>
<td>2.65 ± 0.02abc</td>
<td>1.36 ± 0.02abc</td>
</tr>
<tr>
<td>Choc 3E</td>
<td>12.03 ± 0.04bc</td>
<td>2.50 ± 0.06</td>
<td>1.29 ± 0.05b</td>
</tr>
<tr>
<td>Choc 3F</td>
<td>11.66 ± 0.27c</td>
<td>2.59 ± 0.11abc</td>
<td>1.31 ± 0.05b</td>
</tr>
</tbody>
</table>

Different alphabets (lowercase: set-up 1 and uppercase: set-up 2) in each column indicate significant differences (p < 0.05).
components.

Declaration of interest

The authors declare that they have no conflict of interest.

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Appendix A

Fig. A1D (v,0.9) in function of grinding time during particle reduction in mini drum.

Fig. A2. D (v,0.9) in function of grinding time during particle reduction in big drum.

Fig. B1. Gradients of linear speed of cocoa product in (a) cylindrical and (b) conical roller stone melangers (According to Tan and Balasubramanian, 2017).
References


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