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SHORT COMMUNICATION

Rheological of chocolate-flavored, reduced-calories coating as a function of conching process

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Abstract Continuous flow and linear viscoelasticity rheology of chocolate coating is studied in this work using fat substitute gums (xanthan, GX). An alternative conching process, using a Rotor-Estator (RE) type impeller, is proposed. The objective is to obtain a chocolate coating material with improved flow properties. Characterization of the final material through particle size distribution (PSD), differential scanning calorimetry (DSC) and proximal analysis is reported. Particle size distribution of the final material showed less polydispersity and therefore, greater homogeneity; fusion points were also generated at around 20 °C assuming crystal type I (β'_2) and II (α). Moreover, the final material exhibited crossover points (higher structure material), whereas the commercial brand chocolate used for comparison did not. The best conditions to produce the coating were maturing of 36 h and 35 °C, showing crossover points around 76 Pa and a 0.505 solids particle dispersion (average particle diameter of 0.364 μm), and a fusion point at 20.04 °C with a ΔH_f of 1.40 (J/g). The results indicate that xanthan gum is a good substitute for cocoa butter and provides stability to the final product.

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Introduction

The rheological response of a chocolate coating is determined by the composition of the sample (i.e. amount of fat, amount and type of emulsifiers and particle size distribution) as well as the various stages involved in chocolate processing which include mixing, refining, conching, tempering, molding and packaging (Minifie 1989; Chevalley 1999; Do et al. 2007). Cocoa butter has a polymorphic structure meaning that it is made up of different types of fat, each with different melting points. The challenge with tempering is that all the fat molecules move in a single direction. However, due to this process, we can store chocolate without altering its properties up to 35 °C (Jolly et al. 2003). During the tempering process of chocolate, a correct crystallization of cocoa butter molecules is achieved. This an essential step in the process, as it is responsible for a number of characteristics such as firmness, hardness, melting inside the mouth, flavor, and texture, to name a few. Chocolate has been reported to behave as a non-Newtonian fluid due to a thick suspension consisting of non-fat particles dispersed in cocoa butter, which acts as the continuous phase (Landfeld et al. 2000; Jolly et al. 2003; Afoakwa et al. 2007). A chocolate coating is characterized for being a hard material to the touch, which easily breaks under mechanical stress (Forsyth and Quesnel 1963; De Graef et al. 2011). These properties not only determine the efficiency of stages such as mixing and pumping, but also play an important role in the different chocolate applications such as enrobing, shell formation and moulding steps (Beckett 2009, Servais et al. 2004). These dispersions are commonly used to provide sweetness and have recently been developed to make

low-caloric coatings. It is then clear that on the one hand, the occurrence of various solid phases (i.e., polymorphism) has a significant impact on the product quality of chocolate and confectioneries; on the other hand, the rheological properties of chocolate are very important in chocolate processing for obtaining high quality products with well established texture (Afoakwa et al. 2008a, b, c, d, e). Consequently, detailed knowledge of these aspects is of utmost importance to optimize the process and achieve and maintain product quality. There are few studies of oscillatory tests on chocolate samples, and particle size distribution plays a clear role in process fluidity, but is generally restricted to experience based on empirical knowledge. The purpose of this work was to study the effect of the conching process on the linear viscoelastic properties, the particle size distribution, melting enthalpy and caloric intake of a chocolate coating prepared with fat substitutes using xanthan gum.

Methods and materials

Coating and conching process

The chocolate-flavored coating was prepared by mixing chocolate flavored powder, 22.5 g of powdered sugar (Green Valley ®), 6 g of pasteurized skim margarine (Primavera ®), 6.5 g of dark chocolate (Tile ®), 2.5 mL of pasteurized skim milk (Lala ®), 5 mL of purified water (Ciel ®) and 0.2 % (0.085 g) of xanthan gum (batch 09144KKTLT from JT Baker). The milk and chocolate were first placed in a container and heated in a water bath BÜCHI at approximately 65 °C to melt the chocolate and mix. The margarine was similarly melted in a container at approximately 65 °C and added to the chocolate-milk mix. Then, 2 mL of water was used to recover the margarine adhered to the walls of the container. The powdered sugar was added to the mixture. 3 mL of water were used to dissolve the xanthan gum prior to incorporating it into mixture. Conching is carried out at a constant stirring speed of 166 rpm for 15 min, using a rotor-stator impeller and a digital stirrer IKA Eurostar (Quiñones-Muñoz et al. 2011). Once the conching process is completed, the coating obtained is heated at various temperatures (25, 30 and 35 °C) and it is subjected to different maturing times (12, 24, 36 and 48 h). When the maturing time is completed, the coating is subjected to freezing to stop its thermal history and ready for characterization.

Proximate analysis

A vacuum drying method was employed to control sample humidity. A proximate analysis was performed on the sample following the methods of the Association of Analytical Chemistry (NOM-186-SSA1/SCFI-2002, AOAC; Codex STAN 87–1981): carbohydrates (AOAC 938.18), ash (AOAC 972.15), fat (AOAC 963.15), moisture (AOAC 977.10) and nitrogen (AOAC 970.22).

Rheological tests

Rheological tests were performed in a stress-controlled, model TA Instrument AG-2® rheometer using concentric Plates 25 mm with a 5 mm gap. The evolution of the dynamic mechanical modules was monitored in small amplitude linear oscillatory flow (γ <5 %) tests. The frequency range was from 0.1 to 300 rad/s at different temperature: 25, 30 and 35 °C. The tests were performed at least twice to check for repetibility.

Modeling linear viscoelasticity

The results obtained from the rheometric tests were modeled using a multimode Maxwell model (Calderas et al. 2009; Macosko and Larson 1994) algorithm. The multimode Maxwell model was expressed in terms of elastic moduli G', and viscous G", as indicated by Eqs. (1 and 2):

$$G'(\omega) = \sum_{i=1}^{N} G_{0i} \frac{\omega\lambda_{0i}}{1 + (\omega\lambda_{0i})^2} \quad (1)$$

$$G''(\omega) = \sum_{i=1}^{N} G_{0i} \frac{(\omega\lambda_{0i})}{1 + (\omega_0\lambda_i)^2} \quad (2)$$

Table 1 Proximate analysis of chocolate coatings

Sample	Dry Material (%)	%, Humidity	%, Ethereal Extract	%, Protein	%, Ash	%,Ext. Nitrogen Free	Gross Energy (KJ/100 g) d.m.)
Ganache Coating (Chantilly)	91.34±1.23	8.65±0.08	4.33±0.16	0.96±0.04	0.68±0.04	85.38±1.42	1661.82±62.42
Xantham 12 h, 35°C	95.47±0.96	4.52±0.11	15.25±0.18	1.18±0.05	0.39±0.04	78.65±1.62	1982.92±66.23
Xantham 36 h, 30°C	95.76±0.89	4.24±0.08	10.85±0.15	0.84±0.04	0.28±0.04	83.79±1.20	1889.47±54.26
Xantham 36 h, 35°C	95.91±0.92	4.09±0.08	10.65±0.12	3.15±0.06	0.41±0.04	81.75±0.89	1898.82±65.23
Xantham 48 h, 35°C	91.90±1.65	8.10±0.12	3.37±0.13	6.79±0.08	1.06±0.06	80.67±1.22	1680.29 ±62.82

*Anova, $\alpha < 0.05$

Table 2 Caloric content of two chocolate coatings

Composition	Coating with Xantham Gum kcal g^{-1} (36 h, 35ºC)	Ganache Coating kcal g^{-1}
Proteins	0.03±0.01	0.50±0.04
Carbohidrates	2.70±0.02	0.09±0.02
Fats	0.62±0.01	6.98±0.04
Hydrocolloid	0.001±0.001	4.00±0.02
Total	3.36±0.01	11.58±0.03

*Anova, $\alpha < 0.05$

where, G_{oi} and λ_i represent characteristic values of the rigidity modulus and corresponding relaxation time, respectively. The G_{oi} and λi parameters of each sample was calculated using an algorithm developed in a nonlinear fashion, and solved by Mathematica-7, using experimental data of their corresponding G'(ω) and G″ (ω) flow curves. In principle, every relaxation time is associated with a polymer chain length, i.e. a molecular weight spectrum as an equivalent relaxation time spectrum. In practice, however, we propose a discrete spectrum of relaxation times, that is to say, we only consider dominant modes. In fact, we only model with the minimum number of modes necessary to follow experimental rheological behavior with an error of less than 5 %. Experimental results and modeling are used to infer the complexity of the rheological behavior of the investigated samples.

Particle size distribution, (PSD)

Measurements to analyse the samples PSD were carried out using a MasterSizer Analyzer, Refractive index 1.395 for chocolate coating, this parameter was previously obtained in a refractometer at dissolution 1/100, (Malvern Instruments Ltd., Malvern, England). Approximately 0.1 g of chocolate coating was dispersed in water (refractive index of water 1.333) at room temperature (25 °C). The sample was placed under ultrasonic dispersion for 3 min to ensure particles were independently dispersed and the size distribution was quantified. Each sample was analyzed at least twice.

Differential scanning calorimetry, (DSC)

The differential scanning calorimetry was done using a differential scanning calorimeter DSC TA-60WS acquisition: Status of Shimadzu Corporation with a scan rate of 5 °C min^{-1} as detailed in Medina et al., (2006). The nitrogen gas flow was regulated at a velocity of 30 mL per min^{-1}.

Results and discussion

Proximate analysis of chocolate coatings

Table 1 shows the proximate analysis results of the chocolate coatings and a commercial sample used for comparison (Ganache). Results show that high temperatures and long maturation times (48 h 35°C) render the lowest gross energy content sample similar to that of the commercial sample. It is interesting that the xantham gum provides the chocolate coatings with high protein and ethereal extract content having higher values as compared to those of the commercial sample. Temperature does not seem to have an important effect in the proximate analysis results as samples with the same maturing time but different processing temperature render basically the same gross energy content (36 h, 35 °C and 36 h, 30 °C).

Table 2 shows that the xantham gum chocolate coating (36 h, 35 °C processing conditions) contains less than a third of the calories in comparison to the commercial brand (Ganache). The chemical analysis results indicate a composition of 95.91 % of dry material, 0.41 % ash, 10.65 % of ethereal extract, 81.75 % extract nitrogen free and 3.15 % of protein (Table 1). There is a higher amount

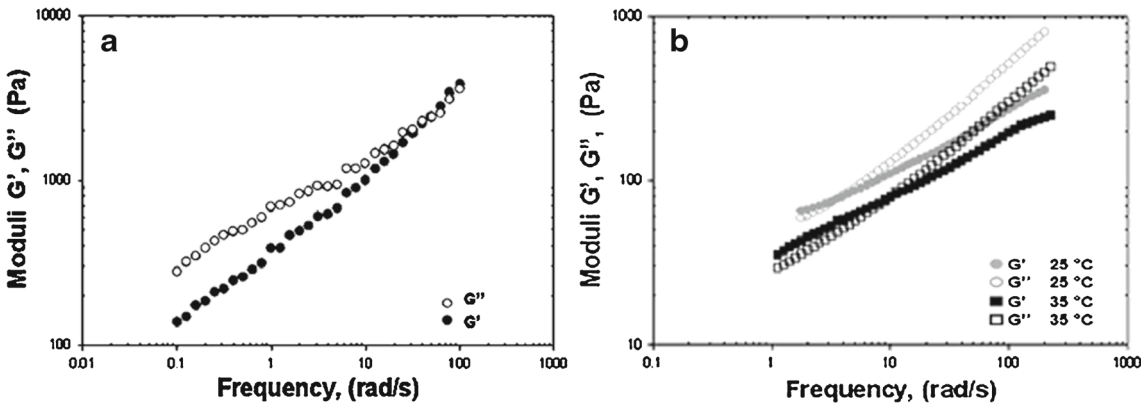

Fig. 1 Dynamic spectrum (G′ and G″) of chocolate coatings with the best viscoelastic behavior for each coating analyzed: **a** Commercial brand; **b** Coating with 36 h and 35 °C tempering at different rheometric test temperatures

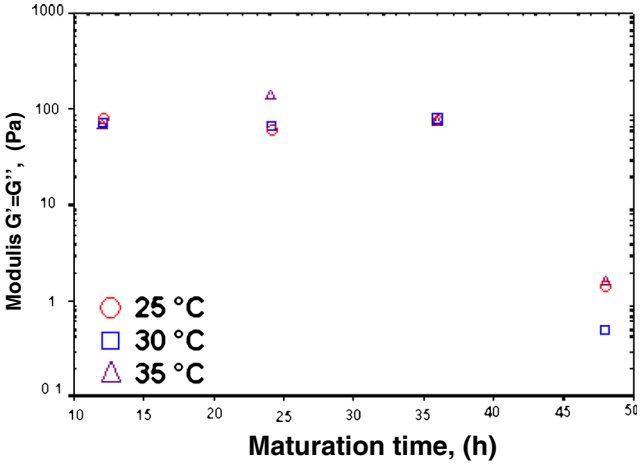

Fig. 2 Crossover points of the dynamic moduli (G′ and G″) as a function of tempering time. (The temperatures correspond to rheometric test temperatures)

of fat in chocoloate coating samples in comparison to those reported in literature for this type of product (30–45 % cocoa butter) (Stortz and Marangoni 2011).

Linear viscoelasticity

The rheological characterization was carried out at different test temperatures (10, 25 and 35 °C) and the results are presented in terms of dynamic moduli (elastic, G′, and viscous, G″) in the linear viscoelastic region. Figure 1 shows the evolution of the dynamic moduli of the coatings produced, particularly those that obtained the best results in mechanical flow in comparison to the commercial brand. Figure 1a shows the evolution of the dynamic modulus of the commercial brand covers, Ganache (Chantilly), showed a crossover point (G′=G″) at 50 rad/s, and after this point, the cover exhibits G′ > G″ at longer times or lower frequencies; this implies that at lower frequencies, the material forms a crystalline structure more stable and rigid (*solid-like* behavior), the crossing point is located at high frequencies and indicates a change in the structure of the final coating. Figure 1b shows the analysis of the chocolate coating produced with at tempering conditions of 36 h and 35 °C.

The rheological behavior of the coatings is influenced by the characteristics of the tempering process. This type of experiment simulates final conditions that could be applied to the sample, thus the applied shear increased the structure into the sample associates to the liquid part (i.e. weakly structure) and in the solid part (more organized structure). This behavior is usually found in systems with high lipid content in liquid state (Toro-Vazquez et al. 2004) and in cocoa butter (Vaeck 1960; Landfeld et al. 2000). This coating showed an improved rheological behavior as compared to the that of the commercial brand, that is to the crossover point of the xantham gum chocolate coating lies at lower which means that this coating has a better mechanical response (higher solid behavior at 25 °C and stability) and also better flow behavior at high temperatures with both modules having lower values as those of the commercial brand indicating a lower viscosity, which is possibly due to the coating composition (due to the enhanced fatty acid chain mobility). This behavior is a function of time and temperature during tempering, and was sufficient enough to form more homogeneous crystals with the ability to coat non-fat particles to form a more stable dispersion, as the one reported by Schantz and Rohm (2005), where they indicated that emulsifier quantity was not the only element to have an effect in order to obtain better mechanical flow, but also the temperature at which the chocolate is processed. Figure 2 shows the crossover point values (G′=G″) at 35 °C tempering temperature for different rheometric test temperature. The crossover point (G′=G″)

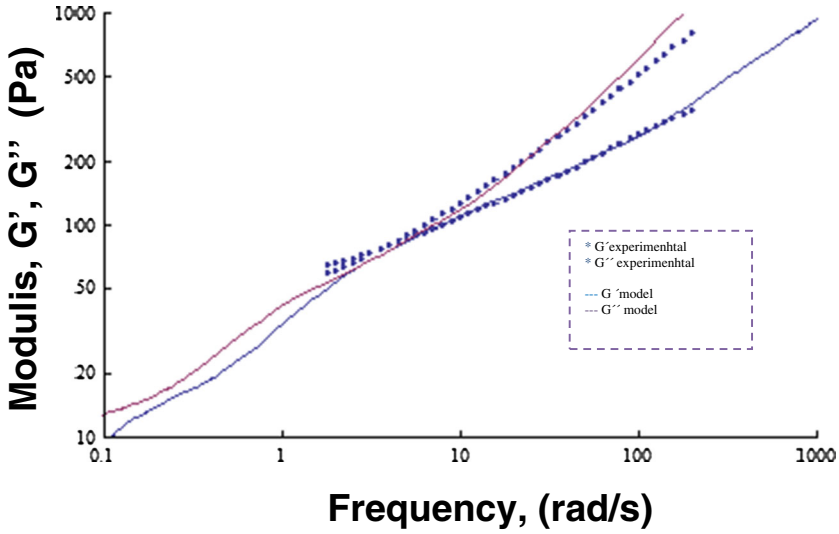

Fig. 3 Comparisons of experimental dynamic data (*dotted lines*) and Maxwell multimode model (*continuous line*) as a function of the frequency for coating at 36 h and 35 °C tempering conditions

Table 3 Moduli and characteristic times used for the multimodal Maxwell model

Modulus	RS 36 h 25 °C	RS 36 h 30 °C	RS 36 h 35 °C	Times	RS 36 h 25 °C	RS 36 h 30 °C	RS 36 h 35 °C
1	1.22×10^{5}	5.6×10^{4}	5.6×10^{4}	1	3.1×10^{-5}	4.8×10^{-5}	4.8×10^{-5}
2	142	98	98	2	0.0222	0.0274	0.02
3	41.2	64	64	3	0.168	0.174	0.174
4	50.6	47	47	4	0.52	0.74	0.74
5	56	480	480	5	0.00282	0.00286	0.0028
6	1.58×10^{5}	4.18×10^{6}	4.8×10^{6}	6	2.7×10^{-7}	2.6×10^{-7}	3×10^{6}
7	15	14.2	14.2	7	6.6	12.4	14.6
8	10	10	36	8	0.0024	0.48	36

represents a solid–liquid transition. Figure 2 showed that the crossover point value is approximately 76 Pa disregarding test temperature which indicates that there high stability in the strucuture of these the materials (homogeneous and ordered crystals). An optimum tempering time of aproximately 36 h is observed since the crossover point value decays after this maturing time to aproximately 1 Pa at 48 h indicating strucutural modifications (degradation).

The Fig. 3 shows the experimental and modeled curves (Maxwell multimode model) of the G′ and G″ modulus in the investigated frequency range for the 36 h and 35 °C tempering conditions.

In order to reproduce the curves for each sample, it was necessary to employ a multimode Maxwell model with 8 modes (the G_i y λ_i pairs, complex modulus and relaxation time respectively). The values for said parameters are shown on Table 3.

It should be emphasized that the model was based on the minimum number of relaxation times necessary to follow experimental rheological behavior of these materials with and error of less than 5 %. Each relaxation time would be associated with a polymer chain relaxation or polymer-polymer interaction within the chocolate matrix.

Particle size distribution (PSD) analysis

Table 4 summarizes the mean particle size and the dispersion index (DI) for the different tempering conditions studied and the commercial sample at 35 °C tempering temperature for comparison. The DI value is non-dimensional and when the value is closer to the unit, it implies greater dispersion and, in principle, less homogeneity of the particles in the continuous medium. Table 4 shows a tendency to reduce the mean particle size as tempering temperature is increased with particle mean size values of around 1 μm and up at 25 °C and decreasing to around 0.6 μm at 35 °C. The best conditions to obtain small and homogeneous particles were found at 35 °C and 36 h

Table 4 PSD results for coatings produced with xantham gum

Maturing Temp. °C	Maturing time, (h)	PSD (μm)	DI (−)
25	12	1.88±0.07	0.40±0.10
	24	1.19±0.08	0.53±0.11
	36	1.01±0.07	0.72±0.08
	48	1.17±0.09	0.52±0.10
30	12	0.87±0.10	0.72±0.09
	24	0.63±0.11	0.95±0.04
	36	0.68±0.07	0.78±0.08
	48	0.63±0.08	0.85±0.06
35	12	0.65±0.08	0.61±0.06
	24	0.69±0.07	0.55±0.09
	36	0.36±0.08	0.51±0.07
	48	1.18±0.06	0.89±0.06
35	36	0.56±0.08	0.67±0.07

* Anova, $\alpha < 0.05$

Table 5 Results of DSC for coatings produced with xantham gum

Maturing temp (°C)	Maturing time, (h)	Peak temperature, (°C)	ΔH_f (J g^{-1})
25	12	20.13±0.16	1.33±0.01
	24	20.27±0.20	2.76±0.02
	36	20.15±0.14	1.68±0.04
	48	20.18±0.22	1.63±0.06
30	12	20.24±0.18	1.07±0.01
	24	20.22±0.22	1.55±0.04
	36	19.97±0.20	2.65±0.06
	48	20.11±0.24	1.85±0.08
35	12	19.93±0.18	1.46±0.02
	24	20.20±0.22	1.37±0.04
	36	20.04±0.20	1.40±0.06
	48	19.99±0.24	1.68±0.07
[a]35	[a]36	[a]23.04±0.22	[a]2.01±0.10

[a] Values determined for the commercial sample, which was obtained via traditional conching process

maturing conditions with a particles size of 0.36 μm and a dispersion index of 0.5. Conclusively, there exists a linear relationship between rheological parameters and particle size diameter of chocolate coatings.

Differential scanning calorimetry results, (DSC)

Table 5 shows the peak melting temperatures for the materials, all materials show a peak around 20 °C. For coatings at 25 °C tempering temperature, it is observed that enthalpy values vary between 1.33 and 2.76 J g^{-1}, which indicates that there are crystals that comprise type I and II, as reported by Afoakwa, et al. (2007) & Marangoni and McGauley (2003). The same crystal type I and II behavior is observed for coatings with a tempering, temperature of 30 °C and those at 35 °C (Beckett 2009). However, most of the ΔH_f showed values smaller than the commercial brand, which implies that they require a lesser amount of energy to melt the sample, and above all, in comparison to products out in the market, it offers potential advantages, due to a window of mechanical flow behaviors, with greater maleability and stability with time (indicating that xantham gum incorporation into the system did not affect the polymorphism generated). This would correlate with the rheological behavior which indicated materials with lower complex viscosity values but higher elastic properties.

As seen in Table 5, none of the samples analyzed in this work showed the most stable crystalline form β_1 (melting temperature above 20 °C). Although this crystalline phase is the most stable from a thermodynamic point of view, it generates undesirable changes in chocolate texture and appearance (Timms 2002; Afoakwa et al. 2007, 2008a, b, c, d, e). The effect of composition on the crystallization of chocolate samples can be seen in Table 5. The melting profiles samples crystallized at <20 °C, and at three different times.

The results of using xantham gum in chocolate coatings exhibit a mechanical flow behavior with improved stability. The use of alternative impellers during the study has probe to be a good alternative to the traditional conching process obtaining a homogeneous chocolate product with high temperature stability.

Conclusions

An alternative conching process using a rotor stator impeller has been probed to render a chocolate coating with improved viscoelastic properties over a commercial sample (Ganache) used for comparison. The rotor stator alternative conching produces a more homogeneous particle size and lower mean particle size depending on the tempering conditions. The best tempering conditions were found at 35 °C and 36 h. The chocolate coating also showed lower complex viscosity and melting temperature values as those of the commercial sample which would be a good feature from the end product application point of view. The multimode Maxwell model satisfactorily reproduced viscoelastic curves of the chocolate coatings with eight modes.

Xanthan gum was shown to be a good replacement of cocoa butter, giving stability to flow, which implies that cocoa butter can very well be substituted with a lower caloric content compounds which could have a positive impact on the consumer health.

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