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RHEOLOGICAL PROPERTIES OF A COMMERCIAL FOOD GLAZE MATERIAL AND THEIR EFFECT ON THE FILM THICKNESS OBTAINED BY DIP COATING

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ABSTRACT

Glazing refers to the application of a coating material onto the surface of foods to enhance their shine and appearance. The objectives of this work were to analyze the rheological properties of a commercial food glaze material and to study their effect on the film thickness obtained by dip coating. Glazing suspension (83.33% total solids content) was obtained using a commercial powder product. Apparent viscosity was determined (shear rate 0.6–50/s) and yield stress was estimated with a creep test (0.5–10 Pa for 5 s). Experimental data were analyzed by applying the generalized Herschel–Bulkley model. A comparison between average thickness values obtained by dip coating and using a phenomenological mathematical model was carried out. All determinations were carried out at 20, 30, 40 and 50C. Rheological parameters were obtained with satisfactory root mean square errors. Good agreement between experimental and theoretical film thickness as affected by temperature was obtained.

PRACTICAL APPLICATIONS

Glazing refers to the application of a coating glaze material onto the surface of foods to enhance their shine and appearance, contributing to extend the shelf life of foods (like in bakery and confectionery products). In the present paper, the rheological properties of a commercial food glaze material were analyzed and their effect on the film thickness obtained by dip coating was evaluated. This study provides new experimental data to validate the mathematical modeling of the dip-coating process developed in our research group. The general approach proposed to face the food glazing process will be of great significance for industry mainly to improve the control of film thickness of glazes.

INTRODUCTION

Glazing refers to the application of a coating glaze material onto the surface of foods to enhance their shine and appearance. Besides improving this aspect, glazing also contributes in extending the shelf life of foods, like in bakery and confectionery products (Chin *et al.* 2011; Jahromi *et al.* 2012). Many types of glaze materials can be used as film-forming fluids and can be made of protein-based (milk, egg, etc.) and fat-based (shortening, vegetable oil, etc.) components. However, the main ingredient of a glazing suspension is usually table sugar (Nieto 2009).

Rheological characterization is an important variable for coating technology (Chan and Venkatraman 2006). Many physical phenomena that occur during coating of vertical surfaces, such as draining and leveling, are influenced by the rheological properties of the film-forming fluid. For this reason, the design of unit operations used in coating processes requires accurate data on the rheological properties of film-forming solutions and dispersions (Brinker and Hurd 1994; Peressini *et al.* 2003). Specifically, in dip-coating technique, the quality of the final film thickness was related to the viscosity and the yield stress of the film-forming fluid (Patel and Bhattacharya 2002; Karnjanolarn and

McCarthy 2006; Bhattacharya and Patel 2007; Ghorbel *et al.* 2011).

Recently, Peralta *et al.* (2014a) performed a theoretical study of the fluid dynamic phenomena in a dip-coating process, considering the withdrawal and draining steps and that the film-forming fluid behaved as a generalized Newtonian fluid (Reiner 1969; Bird *et al.* 1987). This model was developed using rigorous momentum and mass balances applied to an isothermal, monophasic and non-evaporative system, considering that the main forces are viscous and gravitational. The analytical solutions obtained with the model can be used to predict the principal process variables in the dip-coating technique, such as average film thickness. In addition, to complete the theoretical study, experimental validation and sensitivity analysis of the mathematical model were carried out (Peralta *et al.* 2014b). Model validation was performed using experimental average film thickness data obtained from the literature at constant temperature.

In recent years, there have been little studies about glazing application on food products, although glazing is a common practice in baking and confectionery processes (Nieto 2009; Chin *et al.* 2011; Jahromi *et al.* 2012). However, the rheological characterization of food glaze materials was not found in the literature. This information is useful to control and predict the film thickness during an industrial food glazing process. The objectives of this work were to analyze the rheological properties of a commercial food glaze material and to study their effect on the film thickness obtained by dip coating. In addition, this study provides new experimental data to validate the mathematical modeling of the dip-coating process developed in our research group, considering the temperature dependence of the rheological parameters of a commercial food glaze material (Peralta *et al.* 2014a,b).

MATERIALS AND METHODS

Commercial Food Glaze Material

A commercial food glaze powder was used (Ikebana Lheritier, San Carlos Centro, Santa Fe, Argentina). The product consisted of 95% icing sugar and 5% vegetable protein. A glazing suspension was obtained by dissolving 100 g of powder in 20 mL of tap water at 50C, according to the manufacturer's recommendations. The final suspension (83.33% total solids content) was stored at 5C for 24 h for further analysis. The composition of the suspension supplied by the manufacturer was 79.17% sucrose, 16.67% water and 4.17% protein. Glazing suspension was stable, without phase separation, within the experimental time.

Rheological Characterization

Rheological Measurements. All rheological measurements at 20, 30, 40 and 50 ± 0.5C were carried out in triplicate using a stress-controlled rheometer (RheoStress 80, Haake, Inc., Instruments, Karlsruhe, Germany) with a parallel-plate geometry (diameter: 35 mm, distance between plates: 1 mm). A thin film of silicone oil (100 cP) was applied to the exposed sample edges to prevent water vaporization during measurements.

Rotational rheometry was performed in the shear rate range of 0.6–50/s. Values of the apparent viscosity as a function of shear rate were determined.

Creep tests were carried out by applying an increasing controlled stress for 5 s in the range of 0.5–10 Pa. Strain values, obtained as a response of the applied shear stress on the sample, were determined as a function of shear stress. Yield stress was estimated using the methodology proposed by Bhattacharya (1999). In the log–log plots of strain versus shear stress, the intersection between the two linear segments (obtained by linear regression) can give an estimation of yield stress for the studied condition (Fig. 1) (Steffe 1996). Due to the fact that yield stress can exhibit dependency on measuring time (Nguyen and Boger 1992), preliminary experiments were carried out to verify that the estimated yield stress at each temperature was independent of the measuring time (data not shown).

Generalized Herschel–Bulkley (GHB) Model. Apparent viscosity as a function of shear rate was analyzed by applying the GHB model of four parameters proposed by Ofoli *et al.* (1987) for a generalized Newtonian fluid (Reiner 1969; Bird *et al.* 1987):

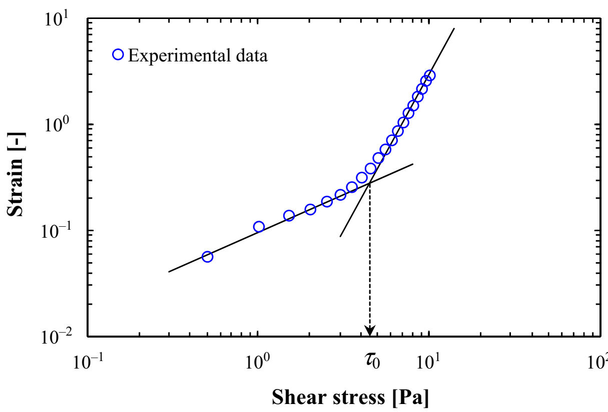

FIG. 1. EXAMPLE OF THE METHODOLOGY USED TO ESTIMATE THE YIELD STRESS BY CREEP TESTS
Symbols are experimental values (one of tree replicates) and lines represent linear regression.

$$\eta = \left(\tau_0^m \dot{\gamma}^{-m} + K^m \dot{\gamma}^{n-m}\right)^{1/m} \qquad (1)$$

where η is the apparent viscosity (Pa·s), τ_0 is the yield stress (Pa), $\dot{\gamma}$ is the shear rate (1/s), K is the consistency index (Pa·$s^{n/m}$), n is the first behavior index (–), and m is the second behavior index (–). This model was selected because it is a generalized expression that includes other rheological models (Bingham, Casson, Heinz–Casson, Herschel–Bulkley, Mizrahi–Berk, Ostwald–de Waele, etc.) (Ofoli *et al.* 1987).

Temperature Dependency of Rheological Parameters. The temperature dependency of τ_0, K and n was analyzed using an Arrhenius-type equation (Yang 2001):

$$A = A_0 \exp\left(\frac{E_A}{RT^*}\right) \qquad (2)$$

where A is the rheological parameter (τ_0, K or n), A_0 is an empirical constant, T^* is the absolute temperature (K), E_A is the activation energy (J/mol) and R is the universal gas constant (J/mol K).

Density

Experimental density of glazing suspension at 20C was determined gravimetrically (10 replicates) by weighing a recipient with known volume (1.83 cm^3) and containing an aliquot of sample. The average density value obtained for these conditions was 1,321 ± 20 kg/m^3.

Theoretical density at each temperature was estimated by considering the contribution of the principal components of the studied food system (water, protein, carbohydrate and air) using the following expression (Michailidis *et al.* 2009):

$$\frac{1-\varepsilon}{\rho} = \sum_{j=1}^{l} \frac{x_j}{\rho_j} \qquad (3)$$

where ε is the porosity (–), ρ is the food density (kg/m^3), x_j is the weight fraction of each food component (–) and ρ_j is the density of each food component (kg/m^3). Values of ρ_j were calculated with the following equation (Choi and Okos 1986):

$$\rho_j = a_1 + a_2 T + a_3 T^2 \qquad (4)$$

where T is the temperature (from –40 to 150C). For water, $a_1 = 997.18$ kg/m^3, $a_2 = 3.1439 \times 10^{-3}$ kg/m^3 C and $a_3 = -3.7874 \times 10^{-3}$ kg/m^3 C^2; for protein, $a_1 = 1{,}330$ kg/m^3, $a_2 = -0.5184$ kg/m^3 C and $a_3 = 0$ kg/m^3 C^2; while for carbohydrate, $a_1 = 1{,}599.1$ kg/m^3, $a_2 = -0.31046$ kg/m^3 C and $a_3 = 0$ kg/m^3 C^2.

Porosity, considered as the air fraction within the glazing suspension, was estimated using the following expression:

$$\varepsilon = \frac{V_b}{V_g} \qquad (5)$$

where V_b is the total volume of air bubbles (m^3) and V_g is the volume of glazing suspension (m^3). Both V_b and V_g were estimated through images captured with an optical microscope (Olympus BH-2, Tokyo, Japan) (Fig. 2). An aliquot of glazing suspension was placed between two microscope slides separated by 0.2 mm and five microphotos were obtained (this procedure was carried out in triplicate). Values of V_g were calculated using the area of the photo (1 mm^2) and the distance between the microscope slides, while values of V_b were calculated taking into account the volume of each bubble considered as a sphere. The porosity value obtained with this methodology was 0.07 ± 0.01.

Average Film Thickness

Experimental Thickness. Experimental average film thickness values were obtained by quintuplicate using the dip-coating methodology proposed by Cisneros-Zevallos and Krochta (2003) with modifications. Glass plates (75 mm high, 25 mm width and 1 mm thick) were used as a substrate. Glazing suspension was conditioned at each temperature (20, 30, 40 and 50C) using a thermostatized bath with recirculation Haake DC 30/Haake W26 (Haake, Inc., Instruments, Paramus, NJ). During dip coating, the

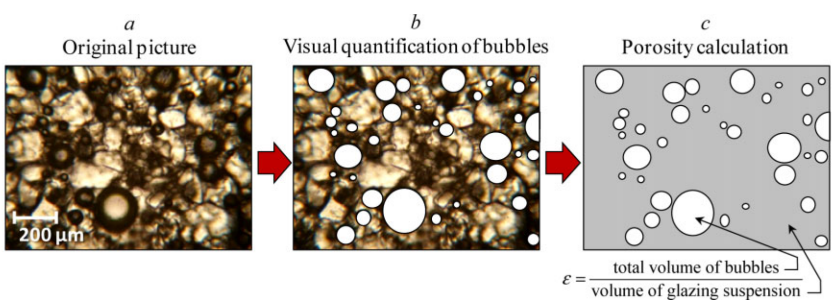

FIG. 2. POROSITY ESTIMATION BY OPTICAL MICROSCOPY

substrate was submerged for 10 s by hand in a glass vessel containing the suspension and then withdrawn quickly, draining the suspension over vessel for 30 s. After that, each substrate was placed in plastic containers and sealed hermetically to prevent water vaporization. The film thickness of the glazing suspension was determined using the following expression (Bhattacharya and Patel 2007):

$$\langle h \rangle_{\exp} = \frac{w}{\rho A_T} \tag{6}$$

where $\langle h \rangle_{\exp}$ is the experimental average film thickness (m), w is the film weight (kg) and A_T is the total area available for coating (m^2) (in this study, 20.8 cm^2). Values of w were determined by the difference between the weight of the substrate before and after dipping.

Theoretical Thickness. Theoretical average film thickness values were estimated using the phenomenological mathematical model proposed by Peralta *et al.* (2014a) for a dip-coating process of a plate during the draining stage based on Eq. (1):

$$\langle h \rangle = {}_2F_1\left[1, -\frac{2}{m}; \frac{1}{n}+1; 1-S_{\tau_0}^m\right]h + \frac{nh}{m(n+1)}\left(1-S_{\tau_0}^m\right){}_2F_1\left[1, 1-\frac{2}{m}; \frac{1}{n}+2; 1-S_{\tau_0}^m\right] \tag{7}$$

$$\left(\frac{\rho g_x h}{K}\right)^m - \left(\frac{\tau_0}{K}\right)^m - \left(\frac{x}{ht}\right)^n = 0 \tag{8}$$

where $\langle h \rangle$ is the theoretical average film thickness (m), h is the film thickness at the x position of the plate (m), g_x is the gravity acceleration (m/s^2), ${}_2F_1$ $[a,b;c;z]$ is the Gauss hypergeometric function (Aomoto and Kita 2011), $S_{\tau_0} = \tau_0/(\rho g_x h)$ is the ratio of the yield stress to the maximum stress and t is the time (s).

Statistical Analysis

Parameters of Eqs. (1) and (2) were estimated by regression (linear or nonlinear regression, when appropriate). Analysis of variance (ANOVA) was used and, when the effect of the factors was significant ($P < 0.05$), the Tukey's honestly significant difference multiple rank test was applied (confidence level 95%). In addition, root mean square errors (*RMSE*) were calculated:

$$RMSE = \sqrt{\frac{1}{N}\sum_{i=1}^{p}\left(\langle h \rangle_{\exp} - \langle h \rangle\right)^2} \tag{9}$$

All statistical analyses were performed using Minitab 13.20 (Minitab, Inc., State College, PA).

RESULTS AND DISCUSSION

Rheological Behavior

Values of the apparent viscosity as a function of shear rate for the glazing suspension are shown in Fig. 3. The shape of flow curves indicates a shear thinning behavior, where apparent viscosity decreases as shear rate increases at all temperatures. According to Rao (2007), shear thinning may occur because of the breakdown of structural units in a food due to the hydrodynamic forces generated during shear. Taking into account the composition, the analyzed glazing suspension has high sucrose content (approximately 80%). Quintas *et al.* (2006) reported that saturated sucrose solutions, with concentrations higher than 60%, presented a Newtonian behavior in a wide range of temperatures. However, those authors noticed a deviation from the Newtonian behavior at some critical values of temperature and concentration of saturated sucrose solutions, probably due to changes in the sample structure, such as crystal nucleation.

Values of the GHB model for the glazing suspension are shown in Table 1. Two particular cases were considered: (1) Herschel–Bulkley ($m = 1$ and $\tau_0 \neq 0$) (Herschel and Bulkley 1926) and (2) Casson ($n = m = ½$) (Casson 1959). Both cases were selected because yield stress was found in the studied experimental conditions. In addition, both models are widely used in the literature to describe the rheological behavior of several types of fluid foods (Steffe 1996; Rao 2007). Thus, yield stress values of case 1 were obtained by creep tests (reducing conveniently the number of parameters for model regression) and yield stress values of case 2 were obtained by linear regression according to Casson

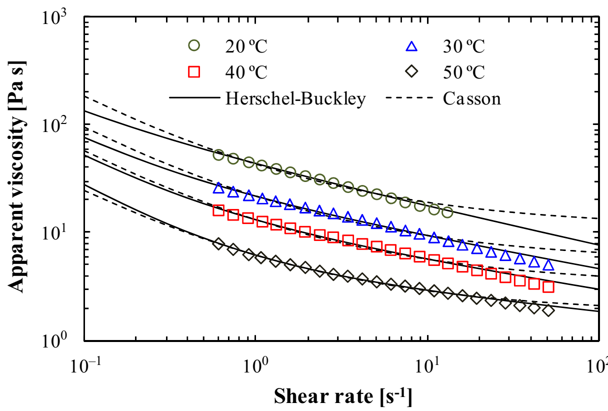

FIG. 3. APPARENT VISCOSITY AS FUNCTION OF SHEAR RATE FOR A COMMERCIAL FOOD GLAZE MATERIAL
Symbols are experimental values (one of tree replicates). Solid line represents case 1 (Herschel–Bulkley) and dash lines represent case 2 (Casson) of the generalized Herschel–Bulkley model.

TABLE 1. VALUES OF GENERALIZED HERSCHEL–BULKLEY MODEL (EQ. 1) OF A COMMERCIAL FOOD GLAZE MATERIAL

Case	*T* (C)	τ_0† (Pa)	*K*† (Pa·s$^{n/m}$)	*n*† (–)	*m* (–)	*RMSE* (Pa·s)
(1) $m = 1$; $\tau_0 \neq 0$ (Herschel–Bulkley)	20	4.46 ± 0.21^{c}*	38.0 ± 1.1^{d}	0.64 ± 0.03^{a}	1	0.48
	30	4.18 ± 0.24^{c}*	16.5 ± 1.1^{c}	0.72 ± 0.02^{b}	1	0.58
	40	3.46 ± 0.63^{b}*	9.70 ± 0.23^{b}	0.74 ± 0.01^{c}	1	0.22
	50	2.22 ± 0.64^{a}*	4.22 ± 0.46^{a}	0.82 ± 0.02^{d}	1	0.08
(2) $m = n = ½$ (Casson)	20	10.3 ± 1.2^{d}	10.7 ± 1.0^{d}	½	½	1.43
	30	5.02 ± 0.59^{c}	5.25 ± 0.23^{c}	½	½	1.16
	40	3.39 ± 0.15^{b}	3.16 ± 0.10^{b}	½	½	0.51
	50	1.40 ± 0.13^{a}	1.84 ± 0.10^{a}	½	½	0.13

Note: Means within a column with different superscript letters are significantly different ($P < 0.05$) within each case.
* Estimated by creep test.
† Mean values and standard deviations of three replicates.

(1959). Values of *RMSE* obtained for Herschel–Bulkley case were lower than values obtained for Casson case, indicating a better agreement between experimental and theoretical data for all temperatures (Fig. 3).

Yield stress values decrease as temperature increases (Table 1). Because yield stress is related to the strength of the network structure, when this value is exceeded during the condition of the experiment, the fluid flows like a truly viscous material with finite viscosity (Nguyen and Boger 1992). Similarly, consistency index values decrease as temperature increases (Table 1), the trend of apparent viscosity (Fig. 3) being similar to *K* for all temperatures. This behavior is expected because *K* can also be defined as the apparent viscosity of a power law fluid at a shear rate of 1/s (Rao 2007). On the contrary, flow behavior indices increase as temperature increases, indicating that the rheological behavior of glazing suspension tends to be closer to Newtonian flow at higher temperatures.

Experimental and Theoretical Film Thickness

A comparison between experimental and theoretical average film thickness obtained by dip coating at 20, 30, 40 and 50C is shown in Fig. 4. Significant difference among experimental values was observed, showing that film thickness decreases as temperature increases. Theoretical data were obtained using Eqs. (7) and (8) with experimental yield stress values obtained by creep test, rheological parameters (*K* and *n*) obtained for the particular case 1 (Table 1) and experimental density value. A satisfactory agreement between experimental and theoretical data was obtained (Fig. 4). Similar results were observed by Peralta *et al.* (2014b), who reported good theoretical predictions with the phenomenological mathematical model used in the present work. The authors estimated the theoretical average film thickness for a dip-coating process of a plate during the draining stage using the rheological parameters of the GHB model obtained from the literature for milk chocolate and deep-fat frying batters (Lee *et al.* 2002; Karnjanolarn and McCarthy 2006).

A comparison between experimental and theoretical average film thickness obtained by dip coating in the range of 10–60C is shown in Fig. 5. Extrapolation of the theoretical average film thickness data was considered in order to evaluate the behavior of the mathematical model at lower and higher temperatures than experimental ones. In this case, theoretical data were obtained using Eqs. (7) and (8) but considering the temperature dependency of rheological parameters and density.

Values of activation energies and A_0 estimated by linear regression analysis of data (Eq. 2) were 19,051, 59,445 and –6,613 J/mol and 3.33×10^{-3}, 4.86×10^{-9} and 8.1 for τ_0, K and n, respectively. In all cases, a good adjustment was obtained ($R^2 > 0.86$).

As mentioned earlier, theoretical density values at each temperature were predicted using Eq. (3). For example, the

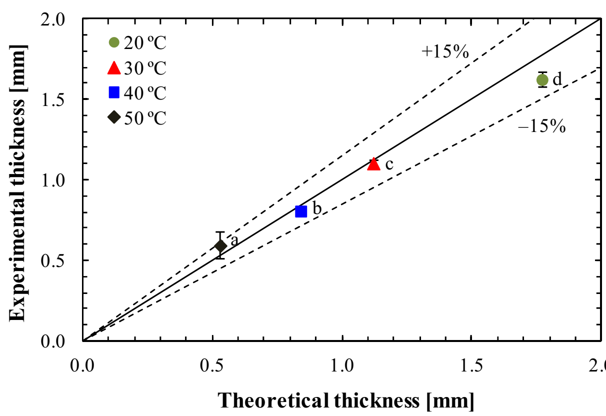

FIG. 4. EXPERIMENTAL AND THEORETICAL AVERAGE FILM THICKNESS VALUES OBTAINED FOR A COMMERCIAL FOOD GLAZE MATERIAL
Bars are standard deviations and lines represent ±15% error. Means with different letters are significantly different ($P < 0.05$).

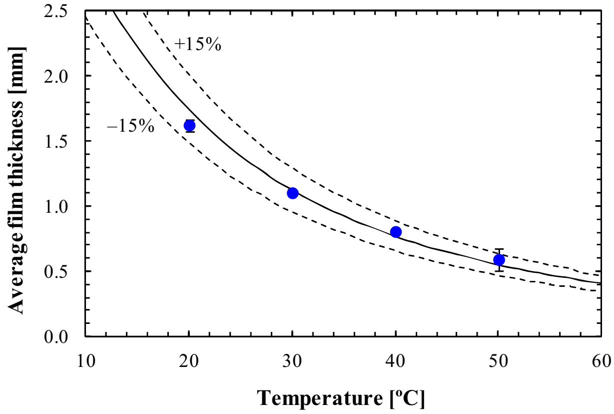

FIG. 5. AVERAGE FILM THICKNESS OBTAINED FOR A COMMERCIAL FOOD GLAZE MATERIAL
Symbols are experimental data and bars represent standard deviations. Solid line represents theoretical data and dash lines represent ±15% error.

theoretical value predicted at 20C was 1,336 kg/m^3, which is a good estimate of the experimental density value of 1,321 kg/m^3. A satisfactory prediction of the average film thickness was obtained at the temperature range of 10–60C (Fig. 5).

CONCLUSIONS

The commercial food glaze material can be analyzed as a generalized Newtonian fluid. A shear thinning behavior was observed for all the studied temperatures. Experimental yield stress values and rheological parameters were obtained, being all dependent on temperature (τ_0 and K decrease while n increases as temperature increases). Experimental average film thickness obtained by dip coating decreases as temperature increases. Theoretical average film thickness was estimated using a phenomenological mathematical model. A satisfactory agreement between experimental and theoretical data was obtained for all temperatures. As a consequence, the rheological properties of the analyzed commercial food glaze material depend on the temperature and influence the average film thickness obtained by dip coating. This information can be useful to control and predict the film thickness during an industrial food glazing process. It is important to mention that during dip coating, a drying process can occur after or at the same time as the withdrawing and draining steps. As a result, the rheological properties of the fluid are expected to change until the final stage of film deposition is reached. Those scenarios are the subject of ongoing studies in our research group.

NOMENCLATURE

A	Rheological parameter (τ_0, K or n)
A_0	Empirical constant of Eq. (2) (–)
A_T	Total area for coating (m^2)
a_i	Constant of Eq. (4) (kg/m^3)
E_A	Activation energy (J/mol)
${}_2F_1\ [a,b;c;z]$	Gauss hypergeometric function
g_x	Gravity acceleration (m/s^2)
h	Film thickness at the x position of the plate (m)
$\langle h \rangle$	Theoretical average film thickness (m)
$\langle h \rangle_{exp}$	Experimental average film thickness (m)
K	Consistency index (Pa·s$^{n/m}$)
n	First behavior index (–)
m	Second behavior index (–)
R	Universal gas constant (J/mol K)
$RMSE$	Root mean square error
S_{τ_0}	Ratio of the yield stress to the maximum stress [$\tau_0/(\rho g_x h)$]
T	Temperature (C)
T^*	Absolute temperature (K)
t	Time (s)
V_b	Total volume of air bubbles m^3)
V_g	Volume of glazing suspension (m^3)
w	Film weight (kg)
x	Position in the x-direction (m)
x_j	Weight fraction of the jth component of the food sample (–)

Greek symbols

$\dot{\gamma}$	Shear rate (1/s)
ε	Porosity (–)
η	Apparent viscosity (Pa·s)
ρ	Food density (kg/m^3)
ρ_j	Density of the jth component of the food sample (kg/m^3)
τ_0	Yield stress (Pa)

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CONFLICT OF INTEREST

The authors Bárbara E. Meza, Juan Manuel Peralta and Susana E. Zorrilla declare no competing financial interest.

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