https://doi.org/10.17221/57/2022-CJFS

Use of rheological plastic models to describe the flow behaviour of unconventional chocolate masses

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Citation: Kouřilová V., Dufková R., Hřivna L., Kumbár V. (2022): Use of rheological plastic models to describe the flow behaviour of unconventional chocolate masses. Czech J. Food Sci., 40: 305–312.

Abstract: The chocolate mass behaves like a typical non-Newtonian plastic liquid defined by the yield stress and the plastic shear stress. The rotary rheometer with a cone-plate spindle system was chosen to determine the flow properties of chocolate masses. The effect of shear stress on shear deformation rates was measured at a temperature of 40 °C in an ascending mode from 1 s^{-1} to 500 s^{-1} for chocolate samples [white chocolate (WC), ruby chocolate (RC), and caramelised Amber chocolate (AC)]. Plastic models, according to Casson, Bingham and Herschel-Bulkley, were used for the mathematical description of this dependence. The Herschel-Bulkley model was evaluated as the most suitable mathematical model for describing the flow behaviour of unconventional chocolate masses. The Herschel-Bulkley model was chosen based on a high value of the coefficient of determination R^2 and a low value of the sum of the square error estimate (SSE). The non-Newtonian plastic behaviour was confirmed, and the yield stress was determined for all types of tested chocolate masses.

Keywords: white chocolate; ruby chocolate; Amber chocolate; rheology; plastic behaviour; yield stress

Chocolate is very popular all over the world especially due to its easy melting in the mouth and typical sensory and textural properties (Hinneh et al. 2019). White chocolate (WC) is a delicacy made from cocoa butter, milk or dairy products, sweeteners, or other ingredients (Afoakwa 2010; Glicerina et al. 2016). WC does not contain cocoa mass (Coady 2000), only cocoa butter in an amount of at least 20%. Another legislative requirement is a milk component content of at least 14% with a milk fat content of at least 3.5%. The fat is mixed with sweeteners and then with milk powder or other dairy products [Directive (EC) No 36/2000 of the European Parliament and of the Council of 23 June 2000 relating to cocoa and chocolate products intended for human consumption]. This chocolate has a consistency very similar to milk chocolate and goes well with some fruits. Poor quality WC is greasy and can leave a greasy mouthfeel (Coady 2000). WC contains fewer aromatic compounds in contrast to dark and milk chocolate. Closely related to this fact is its lower popularity worldwide as well as the processors' efforts to improve and enhance the aroma achieved by caramelising WC (Aydin et al. 2021). During caramelisation, new organoleptic attributes are created (appearance, texture, smell, taste, colour) (Kroh 1994). The rheological and organoleptic properties of caramel chocolates are associated with caramelisation conditions, especially temperature and time (Aydin et al. 2021).

Supported by the Mendel University in Brno, Czech Republic, the Internal Grant Agency of the Faculty of AgriSciences (Project No. AF-IGA-2020-TP006).

https://doi.org/10.17221/57/2022-CJFS

A new type of chocolate has been known on the market, called ruby chocolate (RC), since 2017. The Belgian-Swiss company called Barry Callebaut came to the world market with this new type of chocolate. RC is characterised by its pink to reddish colour, not caused by genetic modification of the cacao tree or the addition of dyes (Šeremet et al. 2019; Burke et al. 2021), but it is a natural colour of cocoa beans and therefore does not need to colour the final product. It follows that the composition of RC is not fundamentally different from classic chocolate consisting of cocoa solids, cocoa butter, and sugar. The colour of the cocoa solids depends on how the cocoa beans are processed. In the case of the standard procedure, the fermentation of cocoa beans takes place. The technological process is different for cocoa beans intended for the production of RC. The fermentation process is eliminated to achieve a violet-pink or red-pinkish colour. The characteristic taste of RC is less bitter, intensely sweet and sour with a fruity undertone of berry fruit without the need for further flavouring (Sulaiman and Yang 2015; Šeremet et al. 2019; Montoya et al. 2021). In the literature, RC is often referred to as the fourth type of chocolate, but it has not been officially accepted by a Standard for Chocolate and Chocolate Products (Montoya et al. 2021). Even European legislation currently addresses only the issue of already known and researched three types of chocolate (dark, milk, and white). RC has, therefore, still not been incorporated into the legislative requirements, but it should be noted that according to the information on the packaging, it can be called chocolate (it contains at least 47% cocoa solids and at least 26% milk solids).

Rheological, sensory, and textural properties are crucial properties for chocolate, affecting the final quality of products. Determining the rheological (flow) properties of chocolate is important to producing high-quality products with a well-defined texture (Servais et al. 2007; Vásquez et al. 2019). In terms of rheological properties, chocolate exhibits non-Newtonian behaviour behaving like a plastic liquid with yield stress limit and plastic viscosity. The viscosity is not directly proportional to the shear rate for non-Newtonian fluids. If the viscosity did not change with the shear rate and if the shearing of the liquid was stopped immediately after the stress fell to zero, then the flow curve (shear stress *vs.* shear rate) would cross zero. However, the viscosity is not constant and is dependent on shear thinning or thickening, temperature, time (thixotropy, rheopexy) and pressure for most samples, including chocolate (da Silva Lannes and Medeiros 2008). Thanks to the use of various mathematical models (for measured shear stress data), the flow behaviour of chocolate can be adequately described (Vásquez et al. 2019). From the point of view of the production process, knowledge of the flow properties of chocolate is important, especially due to the prediction of the behaviour of the chocolate mass in the processing process (de Jesus Silva et al. 2019), ultimately reflected in its quality.

The aim of this study was to determine whether non-traditional types of chocolate show the same flow properties as traditional types of chocolate masses (dark and milk).

MATERIAL AND METHODS

Material. Three types of chocolate masses were selected, namely white (WC), caramelised (Amber) white chocolate (AC) and red respectively ruby chocolate (RC) to determine the flow properties. The WC and AC masses were obtained from the manufacturer Belcolade and the RC mass from Barry Callebaut. Detailed specifications of individual chocolate masses are presented in Table 1.

Methods. RST rotary rheometer (Brookfield, US) with a cone-plate spindle arrangement (RCT-50-2) with a sample temperature duplicator was used to measure the flow properties of the chocolate samples. The flow properties of chocolate are possible to measure with the help of this rheometer. The measured data were evaluated by specialised software called Rheo 3000 (Brookfield, US), thanks to which it is possible to program measuring cycles, insert mathematical models and statistically evaluate the obtained data. The following mathematical models were chosen for modelling the flow curves, such as Casson, Herschel-Bulkley, and Bingham.

A 1 g portion of the sample was dissolved on a rheometer plate at 36 °C followed by pressing and tightening the cone to the plate. Subsequently, the temperature was raised to 40 °C, and the shear stress was measured at the shear strain rate at this temperature. The shear strain rate was gradually increased from 1 s^{-1} to 500 s^{-1} over 1 min.

Statistical analysis. The experimental data were processed by MATLAB R2018b (MathWorks, US) and Statistica 12 (StatSoft, US). A statistically significant difference between the measured values of the shear stress and dynamic viscosity at the selected temperatures of the individual chocolate masses was used for the determination. The degree of accuracy and suitability of the plastic flow models were evaluated using the coefficient of determination R^2 and the sum of the

https://doi.org/10.17221/57/2022-CJFS

Table 1. Characteristics of used chocolates

Type	Cocoa solids content at least (%)	Composition	energy value (kJ)	energy value (kcal)	fats (of which saturated fatty acids) (g)	carbohydrates (of which sugars) (g)	proteins (g)	salt (g)
WC	28.0	sugar, cocoa butter (28%), whole milk powder emulsifier: soy lecithin (E322) aroma: natural vanilla	2 360.5	565.7	35.1 (21.3)	56.1 (56.1)	6.4	0.234
RC	47.3	sugar, cocoa butter (29.5%), milk powder, cocoa mass emulsifier: soy lecithin (E322) acidity regulator: citric acid aroma: natural vanilla	2 356.0	563.0	35.9 (21.5)	49.6 (48.5)	9.3	0.270
AC	30.0	sugar, cocoa butter (30%), skimmed milk powder, whey, milk fat, butter emulsifier: sunflower lecithin (E322) aroma: natural vanilla	2 353.3	564.2	35.8 (21.8)	53.3 (53.3)	7.2	0.496

Nutritional information per 100 g

WC – white chocolate; RC – ruby chocolate; AC – Amber chocolate

square error estimate (SSE) as in Kumbár et al. (2015) and Trost et al. (2021).

RESULTS AND DISCUSSION

Mathematical models, according to Casson, Herschel-Bulkley and Bingham (Afoakwa 2010), are suitable for describing the flowing behaviour of chocolate. Table 2 presents the results of mathematical modelling according to these flow models.

The Casson model is recognised in the chocolate industry as a standard for measuring and comparing yield stresses and plastic viscosities depending on the fat content, chocolate mass structure and the amount and type of emulsifier (Singh and Heldman 2014). The Casson model is the International Office of Cocoa, Chocolate and Sugar Confectionery (IOCCC) method and a recognised official method for plotting the flow properties of chocolate using flow curves (Servais et al. 2007). The Equation 1 for the Casson model is as follows:

$$\sqrt{\tau} = \sqrt{\tau_0} + \sqrt{\eta_C}\sqrt{\dot{\gamma}} \quad (1)$$

where: τ – shear stress; τ_0 – value of the stress in the yield stress; η_C – value of the Casson viscosity; $\dot{\gamma}$ – shear strain rate (Cahyani et al. 2019).

Some studies indicate that the Casson model, despite IOCCC recommendations, fails to accurately describe the flow behaviour of chocolate at lower shear rates (Afoakwa et al. 2009; Taylor et al. 2009). Another study questions the use of the Casson model because the authors found inconsistencies across laboratories testing the same chocolate sample. The result of the study was the creation of a new IOCCC 2000 method that recommends comparing viscosity at specific shear rates. The Herschel-Bulkley model (Aeschlimann and Beckett 2000) became a popular model for comparison with Casson. The obtained data were also described using the Herschel-Bulkley model (Equation 2):

$$\tau = \tau_0 + K\dot{\gamma}^n \quad (2)$$

where: K – consistency index; n – flow index (de Jesus Silva et al. 2019).

If $\tau_0 < \tau$, the sample behaves like a Herschel-Bulkley fluid. Otherwise, the sample behaves as a solid (Kumbár et al. 2021). Shear thinning (pseudoplasticity) is typical for non-Newtonian fluids, with the viscosity decreasing with increasing shear rate (Bair and McCabe 2007). This occurs when $n < 1$. If $n > 1$, the fluid solidifies with an increasing shear rate. A Newtonian

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Table 2. Coefficients of the flow models (T = 40 °C; 1–500 s^{-1})

Type	Casson				Herschel-Bulkley					Bingham			
	τ_0 (Pa)	η_C (Pa·s)	SSE	R^2	τ_0 (Pa)	K (Pa·s^n)	n	SSE	R^2	τ_0 (Pa)	η_B (Pa·s)	SSE	R^2
WC	3.937	1.072	1 266	0.9991	13.880	1.776	0.9433	450	0.9997	18.43	1.264	1 541	0.9990
RC	1.848	1.002	784	0.9993	9.797	1.323	0.9726	190	0.9998	11.68	1.123	368	0.9997
AC	9.161	1.249	11 170	0.9949	23.370	2.567	0.9166	8 010	0.9964	31.89	1.556	11 630	0.9947

WC – white chocolate; RC – ruby chocolate; AC – Amber chocolate; T – temperature; τ_0 – yield stress; η_C – Casson viscosity; SSE – square error estimate; R^2 – coefficient of determination; K – consistency index; n – flow index; η_B – Bingham viscosity

fluid is in a situation in which $n = 1$ and $\tau_0 = 0$ (Kumbár et al. 2021). The last used flow model was the Bingham model. Equation 3 for the Bingham model is:

$$\tau = \tau_0 + \eta_B\dot{\gamma} \quad (3)$$

where: η_B – Bingham viscosity (Zzaman et al. 2014).

Bingham (ideal) plastic fluid is characterised by the presence of yield stress representing the necessary stress to start the flow. Below the value of the yield stress, the sample shows solid properties and does not form a flat surface under the influence of gravity (da Silva Lannes and Medeiros 2008; Bergemann et al. 2018). Bergemann et al. (2018) concluded in their research that the Bingham model is the most suitable for modelling chocolate flow curves.

Chocolate is characterised by yield stress (τ_0), respectively, the yield stress tension that needs to be developed to initiate the flow of chocolate (Servais et al. 2007; da Silva Lannes and Medeiros 2008; Fernandes et al. 2013; Glicerina et al. 2016; Bergemann et al. 2018). The yield stresses were marked by all samples. The highest values were recorded for AC while the lowest values were measured for RC. The content of cocoa butter usually has a significant effect on the flow behaviour of chocolate, especially on the yield stress. In general, a lower cocoa butter content results in higher yield stresses (Afoakwa 2010; de Graef et al. 2011; Vásquez et al. 2019). This was confirmed in WC and RC (Tables 1, 2). AC deviated from this rule by its behaviour. Caramelisation of sugars probably manifested itself here.

Thanks to the coefficient of determination (R^2), the most suitable mathematical model for the description of chocolate flow curves can be determined. Lower values of the coefficient of determination (R^2) mean a greater distance of the measured data from the given model. The best model would be a model with a coefficient of determination of 1 (Lannes et al. 2004). Briggs and Wang (2004) state that the Herschel-Bulkley model is better than the Casson model although the Casson model has been the most widely used in the chocolate industry. Herschel-Bulkley provides a better fit

Table 3. Comparison of viscosity at selected values of shear strain rate (T = 40 °C)

$\dot{\gamma}$ (s^{-1})	Type	η (Pa·s)
1	WC	11.142
	RC	7.642
	AC	32.889
2	WC	8.889
	RC	5.986
	AC	16.566
5	WC	5.024
	RC	3.618
	AC	8.165
10	WC	3.050
	RC	2.245
	AC	4.299
20	WC	2.051
	RC	1.691
	AC	2.797
50	WC	1.599
	RC	1.339
	AC	2.081
100	WC	1.526
	RC	1.268
	AC	1.906
200	WC	1.407
	RC	1.206
	AC	1.827
500	WC	1.273
	RC	1.129
	AC	1.483

WC – white chocolate; RC – ruby chocolate; AC – Amber chocolate; T – temperature; $\dot{\gamma}$ – shear strain rate; η – dynamic viscosity

https://doi.org/10.17221/57/2022-CJFS

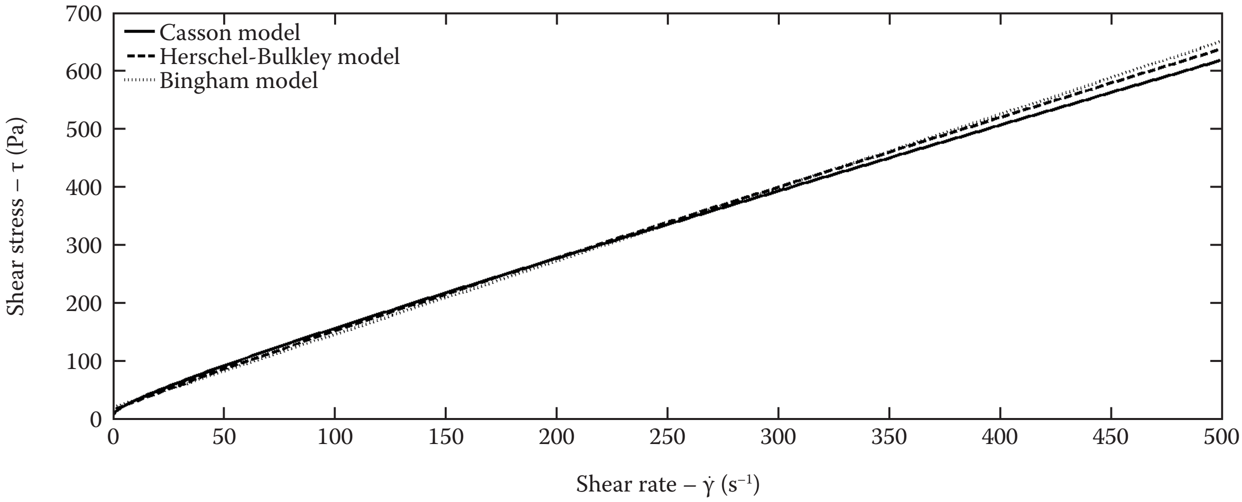

Figure 1. Comparison of the flow curve models of white chocolate (WC) at the temperature 40 °C

of the flow curve confirmed in a study by Kumbár et al. (2021) and in this study. According to the measured data with subsequent modelling, the model according to Herschel Bulkley (R^2 = 0.9964 – 0.9998), the model according to Bingham (R^2 = 0.9947 – 0.9997) and Casson (R^2 = 0.9949 – 0.9993) are almost as suitable. This applies to the interval of shear deformation rates 1–500 s^{-1}. This is confirmed by the low values of SSE.

Table 3 shows a comparison of viscosity at selected values of shear strain rate.

As the shear rate increases, the viscosity decreases so this is a plastic behaviour. This fact is until the viscosity becomes independent of the shear rate at high speeds (Servais et al. 2007; da Silva Lannes and Medeiros 2008; Afoakwa 2010; Aydin et al. 2021).

Figures 1–3 present a comparison of flow curves of individual types of chocolates at a temperature of 40 °C. Figure 4 shows the flow curves of three types of chocolates (WC, AC, RC) according to the Casson model.

WC and RC show almost identical flow curves but with a difference in lower viscosity for RC. AC showed large differences in flow curves according to individual mathematical models. AC (unlike WC and RC) showed significant yield stress.

The flow behaviour of chocolate depends on the measurement temperature. A temperature of 30 °C is crucial for consistency. Below 30 °C, chocolate exhibits a soft solid behaviour while at temperatures above 30 °C, chocolate exhibits visco-plastic liquid behaviour (Ardakani et al. 2014). If powdered milk is present in the recipe composition of chocolate, its flow behaviour is affected. Low values of shear stress may indicate the presence of milk powder in a given sample (Lucisano et al. 2006; Afoakwa 2010; Glicerina et al. 2016).

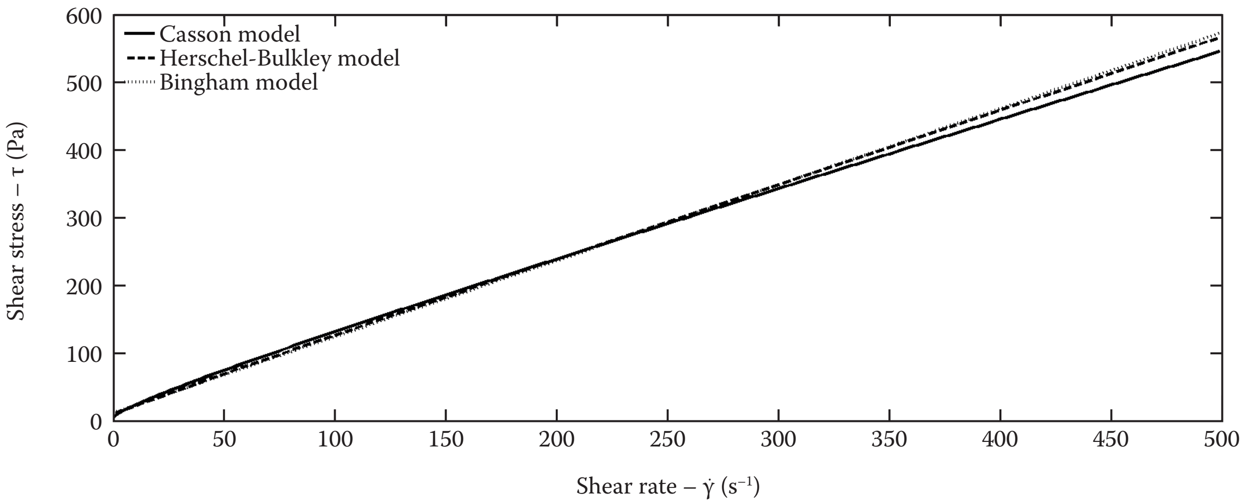

Figure 2. Comparison of the flow curve models of ruby chocolate (RC) at the temperature 40 °C

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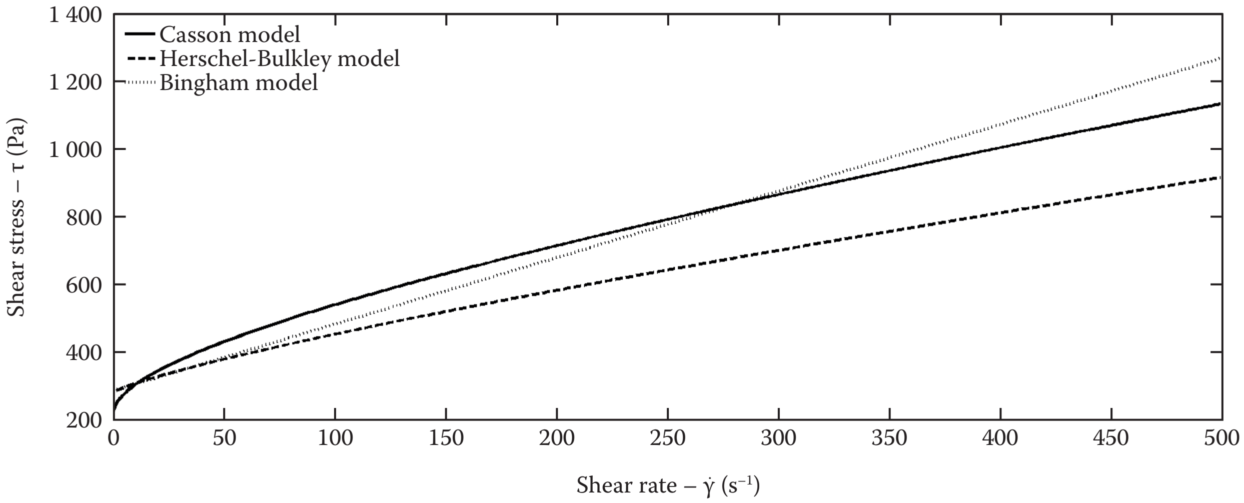

Figure 3. Comparison of the flow curve models of Amber chocolate (AC) at the temperature 40 °C

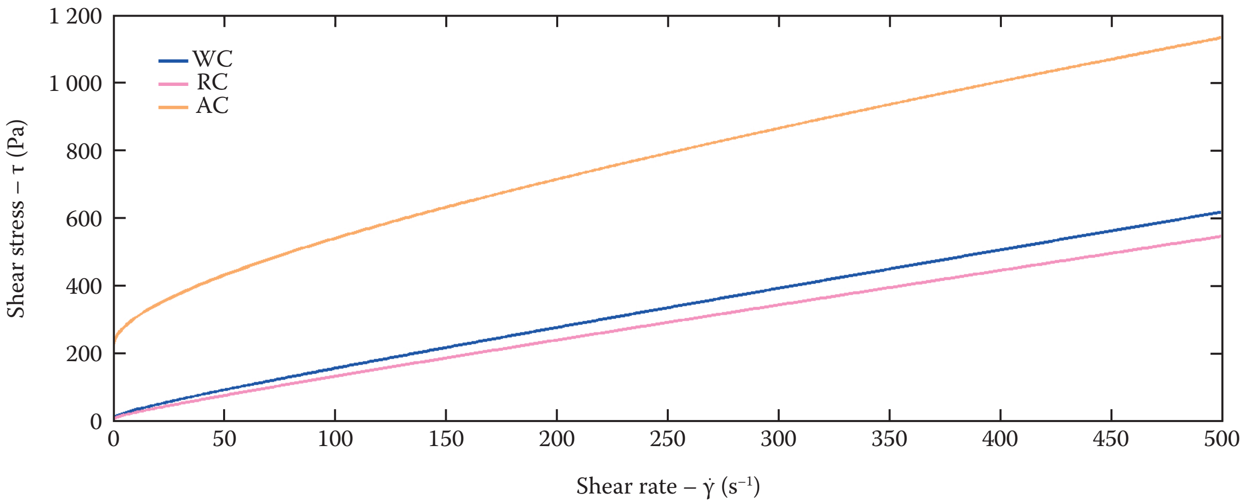

Figure 4. Flow curves (using Casson model) of white chocolate (WC), ruby chocolate (RC) and Amber chocolate (AC) at the temperature of 40 °C

CONCLUSION

The change of shear stress depending on the rate of shear deformation was monitored in three samples of chocolate masses at a temperature of 40 °C. All selected rheological plastic models (Casson, Bingham, Herschel-Bulkley) appeared to be suitable for describing the flow behaviour of non-traditional chocolate materials (WC, RC, AC). The Herschel-Bulkley model can be evaluated as the best model for describing the flow properties of chocolate masses based on the obtained coefficients as the values of the coefficient of determination (R^2) ranged from 0.9964 to 0.9998 and low values of the SSE were achieved at the same time.

It was confirmed that even non-traditional types of chocolate such as WC, RC and AC show similar flow behaviour (plastic) as traditional chocolate masses (dark and milk).

The technology of chocolate mass processing requires knowledge of their flow properties, especially in the process of transporting chocolate mass by pipeline in the food company but also outside it as well as in the process of mixing, shaping chocolate products and storage. Knowledge of this issue can also be positively reflected in the search and solution of technological problems in the production process and to improve and respond appropriately to storage conditions. Current trends require both technical knowledge of fluid flow modelling and knowledge of technological require-

https://doi.org/10.17221/57/2022-CJFS

ments for flowing equipment in the food industry such as 3D food printing (including printing of chocolate products) in this case for the tempered chocolate mass.

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Received: April 1, 2022
Accepted: June 6, 2022
Published online: August 17, 2022