

RHEOLOGICAL CHARACTERIZATION OF COFFEE-FLAVORED YOGURT WITH DIFFERENT TYPES OF THICKENER*

■ABSTRACT: Yogurt is a functional food that has great demand due to the consumer's search for a healthier diet. In order to expand the consumer market of this product, many flavors are available, satisfying the most varied preferences. Besides the taste attribute, consistency and viscosity of yogurt are some of the main factors involved in product quality and acceptance. Therefore, this work is a study of the influence of concentration of thickener in coffee-flavored yogurt. The thickener agent used was gelatin. The rheological behavior (flow and viscosity curves) of yogurts with and without addition of gelatin was compared with commercial yogurt, which contains another type of thickener (locust bean gum) in its formulation. The flow and viscosity curves were obtained from rotational rheometer Thermo Haake Mars, with a range of shear rate from 0.02 to 100 s⁻¹ (rising curve) and 100 to 0.02 s⁻¹ (descendent curve) at a total time of 20 minutes. Hysteresis was determined as the area between the curves and adjusted to the models of Bingham, Casson, Herschel-Bulkley and Ostwald-de-Waele. Were also carried out tests of thixotropy, by measuring the viscosity as a function of time at a constant rate of 100 s⁻¹ for 10 minutes. These curves were adjusted by the Weltman model. All samples showed pseudoplastic and thixotropic behavior. The Herschel-Bulkley model was the best fit to the three samples tested. The Weltman's model well described the thixotropy tests, except for the sample of commercial yogurt. The use of gelatin as a thickener showed protective character, reducing the structural break of the gel.

■KEYWORDS: Rheological models; pseudoplastic behavior; gel stability; viscosity.

INTRODUCTION

The search for a healthier diet is becoming increasingly prevalent throughout the world. People concerned about their health and well-being have ensured Thiago Rocha dos Santos MATHIAS** Itamar Cabral de CARVALHO JUNIOR*** Carlos Wanderlei Piler de CARVALHO**** Eliana Flávia Camporese SÉRVULO*****

a growing demand for healthy products. In this context, emerge the called functional foods, which besides their basic nutritional functions, provide many benefits to human health.^{3, 32} There are a wide variety of products with this appeal, and the dairy industry has an important role in this market.¹³

Yogurt can be noted between functional foods. It is a product obtained from the fermentation of milk by the symbiotic action of traditional lactic microorganisms, *Streptococcus thermophilus* and *Lactobacillus bulgaricus*.⁴ Rich in protein, folic acid, vitamins A, B and minerals, its regular consumption brings many health benefits, such as gastrointestinal regulation, stimulation of the immune system, etc.⁶

Coffee is a product consumed daily in the world by all social classes.²⁴ Brazil is the largest producer and second consumer market in the world.¹ The coffee has about 1 to 2.5% caffeine and other substances in greater quantity. The coffee beans (green coffee) feature a large variety of minerals, amino acids, lipids and sugars. Additionally, the coffee also has a vitamin B, niacin (vitamin B3 or vitamin PP), and chlorogenic acids, which, after roasting, form several compounds with pharmacological effects.^{2, 5, 11, 23}

Some effects of coffee on health are: reduction of cholesterol, aid against heart disease, antidepressant effects, reduced risk of Parkinson's disease, protection against type 2 diabetes, antioxidant, aiding in weight loss procedures and prevention of some types of cancer (colon and rectum). ^{7, 20, 21, 26, 30}

The consumption of yogurt has experienced significant increases and there are various types to achieve the broad consumer market. These products may vary according to the ingredients, composition, flavor, consistency, texture, caloric value, development process and nature of the process of post-hatching.²⁹ The final product quality has great importance in its acceptance, and is influenced mainly by its consistency and viscosity.

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The texture of the product and propensity to syneresis (serum separation) are the main characteristics that will define the quality of yogurt.¹⁹ Generally, to increase the viscosity, the practice used in industries is to increase the solids content by adding milk or whey powder.³⁷ However, thickeners can be added for this purpose, leaving a firmer texture, reducing syneresis and increasing the acceptability of the yogurt.

Various polymeric materials can be used as thickeners and its origins can be vegetable or microbial. Among the polysaccharides, are prominent: starch, pectin, carrageenan, alginates, xanthan gum, gellan gum and arabic gum. Between the proteinaceous materials, are the caseinate and gelatin.³⁹ According Walstra et al., ⁴⁰ and Tamime & Robinson, ³⁷ three of the most commonly used thickeners are starch, pectin and gelatin.

Many factors can affect the rheology of yogurt, as solids, protein and fat content, temperatures of heat treatment of milk and fermentation, homogenization pressure, etc.^{8, 28} So the aim of rheological studies is to verify the structural stability of the food ahead of possible processes, allowing the correct sizing of pumps, piping, heat exchangers, stirring and bottling operations, etc., without affecting the quality of the final product.²⁷ Besides these, other applications of rheological studies are the quality control, sensory analysis and shelf life testing.¹⁰

Rheological studies usually involve relationships between shear rate and shear stress. When the relationship is linear, the fluid is referred newtonian and its viscosity is constant, independent of the rate and shear stress applied. However, in many cases, this relationship is nonlinear and the fluid is classified as non-Newtonian. These fluids can be pseudoplastic fluids, which have lower viscosity with increasing shear rate applied.³¹

Besides the dependence of the rate, the pseudoplastic fluids can be thixotropic, also dependent on the time of shearing. In this case, even for constant shear rates, viscosity is reduced to a function of time. Flow curves of thixotropic fluids present distinction between the curves of increasing rate and decreasing rate, and this phenomenon is known as hysteresis. Models such as the Ostwald-de-Waele, Hershel-Bulkley and Casson, while not taking into consideration the dependence over time, have been well applied to describe the rheological behavior of these fluids.^{25, 27, 39}

Thus, this study aimed to assess the rheological behavior of coffee- flavored yogurt as a function of different concentrations of thickener (gelatin) and its comparison with commercial yogurt that use other thickeners in its formulation. It was verified the influence of thickeners on the viscosity as a function of time and shear rate, and adjusted the models of Bingham, Casson, Herschel-Bulkley and Ostwald-de-Waele to the flow curves and the Weltman's model to thixotropy test.

MATERIALS AND METHODS

The yogurt was developed and produced at Laboratory of Industrial Microbiology, at the Department

of Biochemical Engineering, School of Chemistry / Federal University of Rio de Janeiro.

The following materials were used to prepare the samples of the yogurt:18, 37 integral UHT milk (Elegê®, BRF Brasil Foods S.A, Chapecó, SC, Brazil) (1L), powered integral milk (Ninho/Nestlé®, São Paulo, SP, Brazil) (60g L⁻¹), refined sugar (União®, Copersucar, São Paulo, SP, Brazil) (100g L⁻¹) and commercial lyophilized starter cultures (1g L⁻¹) of trademark Rich® (Chr Hansen - Valinhos, SP) containing a superconcentrated of the two strains of lactic acid bacteria Lactobacillus bulgaricus and Streptococcus thermophilus. It was used instant coffee as flavoring agent (Nescafé/Nestlé®, São Paulo, SP, Brazil) (3g L⁻¹). The samples were prepared with two different concentrations of thickener (unflavored gelatin colorless - Dr. Oetker®), 0 and 6g L⁻¹, coded as follows: CYNT (coffee-flavoured yogurt without thickener) and CYT (coffee-flavored yogurt with thickener) respectively.

The yogurt manufacture was performed as indicated by Chandan et al.,⁶ Walstra et al., ⁴⁰ Tamime,³⁶ and Rasic & Kurmann: ²⁹ UHT milk was heated to about 43°C for addition of ingredients (milk powder, instant coffee, gelatin, sugar) and the mixture was homogenized. Then, it was inoculated and incubated at 43°C for fermentation. During the fermentations, at intervals of 30 minutes, the monitoring of the pH was made by direct measurement in a digital brand PHTEK (model PHS-3B). When the pH value reached 4.6, the process was interrupted, and soon after, the yogurt was cooled to 4°C, and stored at this temperature.

The commercial yogurt was purchased from the local market (Rio de Janeiro, Brazil) and coded as follows, with their thickener: CY (locust bean gum).

The yogurt samples were submitted to analytical determinations of pH values (by direct measurement on pHmeter) and water content (by drying at 105°C).¹⁷

Rheometry analysis was performed in triplicate at Thermo Haake MARS rotational rheometer (Karlsruhe, Germany), equipped with geometry plate / plate, with 35mm of diameter, available at Brazilian Agricultural Research Company (EMBRAPA/Rio de Janeiro, Brazil). The rheometer has a Haake Mars Controller Peltier type to temperature, whose value was adjusted to 8°C for all analyses.^{14, 33}

Before performing the analysis, all samples of yogurt were also subjected to agitation in mixer for 1 (one) minute, under rotation speed of 150rpm and then remained at rest under refrigeration (10°C) for 10 minutes.^{28, 33}

To obtain the flow and viscosity curves, analyzing the variation of tension and viscosity as a function of shear rate, respectively, the rate ranged from 0.02 to 100s⁻¹ (rising curve) and between 100 and 0.02s⁻¹ (descending curve) enabling the determination of the hysteresis as the area between the curves. The total analysis time was 20 min. and collected 80 points in total.

Thixotropy tests were performed by applying a constant shear rate of $100s^{-1}$ and the determination of

viscosity as a function of time of 10 min., collecting 40 points.

For all the analyses, it was used the software Haake RheoWin 3 to obtain the curves, adjusting the models and calculation of hysteresis. The results were fitted to the models of Bingham, Casson, Herschel-Bulkley and Ostwald-de-Waele.³³ The model of Weltman, time-dependent, was also tested.¹⁴

Statistical analysis of results by ANOVA was carried out using the software Microsoft Excel 2003®, to compare the different models adjusted.

Table 1 shows the rheological models adjusted.

RESULTS AND DISCUSSION

Independent of the concentration or type of thickener used, all samples of yogurt showed behavior of non-Newtonian and pseudoplastic fluid, with viscosity decreasing, with increasing shear rate applied.³¹ According to Horne¹⁶ and Lucey, ²² this can occur due to physical destruction of weak bonds between the molecules of the product and due to decreased energy of interaction between them. Several reviewed studies confirm the yogurt as a pseudoplastic fluid.^{12, 14, 28, 38, 40}

Figures 1 and 2 shows the flow and viscosity curves, respectively, for the different samples of yogurt.

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The sample of coffee-flavored yogurt with gelatin as a thickener showed the highest viscosity values (average viscosity about 381.1mPas) over the entire range of shear rates examined. Then, the highest values were obtained for the sample of coffee-flavored yogurt without gelatin (average viscosity about 307.3mPas) and finally the sample of commercial yogurt (average viscosity about 286.3mPas).

Supavititpatana et al., ³⁵ evaluating the effect of concentration of gelatin in corn milk yogurt, also observed that increasing the concentration promoted the formation of a more consistent product. The addition of thickeners was also assessed by Teles & Flores, ³⁸ who observed an increase in the viscosity of yogurt about 4 times due to increased concentration of gelatin from 4 to 8 g L⁻¹.

The results for analytical determinations are expressed in Table 2.

According to Tamime, ³⁶ low pH values can promote syneresis of yogurt, due to excessive repulsion charges, changing its rheological properties, as observed for the CY sample that showed lower pH and viscosity values. The water content was similar to the three samples tested, and in this case, it was of irrelevant influence on rheological behavior.

As observed in Figures 1 and 2, all samples showed thixotropic characteristics, due to differences of tension and viscosity between rising and descending curve. This

Model	Viscosity	Shear stress	
Bingham	$\eta = \eta_p + \tau_0 / \gamma$	$\tau = \tau_0 + \eta_p \gamma$	
Casson	$\eta = \sqrt[n]{(\tau_0 / \gamma)^n + (\eta_p)^n}$	$\tau^{0,5} = \tau_0^{0,5} + K(\gamma)^{\bullet,5}$	
Ostwald-de-Weale	$\eta = K \gamma^{n-1}$	$\tau = K \gamma^{n}$	
Hersch-Bulkley	$\eta = \tau_0 / \dot{\gamma} + K \dot{\gamma}^{n-1}$	$\tau = \tau_0 + K \dot{\gamma}^n$	
Weltman	-	$\tau = A - B.\log(t)$	
Where:			
$\eta = vis \cos ity(Pa.s)$			
K = consistency.index(P	$Pa.s^n$)		
n = behavior.index(dim)	ensionless)		
$\tau = shear.tension(Pa)$			
•			

 $\gamma = shear.rate(s^{-1})$

 $\eta_p = plasticvis \cos ity$

 $\tau_0 = yield.strength$

 $A = rheo \log ical. parameter. like. \tau_0$

 $B = rheo \log ical. parameter.to.breakage.rate$

t = time



FIGURE 1 – Flow curves of the yogurt samples.



FIGURE 2 – Viscosity curves of yogurt samples.

phenomenon is a result of breakage of the gel and can be quantified as the area between the flow curves. The larger the area between the curves, the higher the thixotropic effect.¹⁷ Values are given in Table 3.

As shown in Table 3, the sample of CYNT, without addition of thickener, presented higher thixotropic characteristic, represented by the largest calculated area of hysteresis. The remaining samples, CYT and CY showed slightly lower hysteresis values, in this sequence. This may indicate the protective effect of thicker agents on the gel of yogurt, reducing structural damage during processing.

Tables 4 and 5 shows the values of the regression coefficient r for models adjusted for the upward flow curves and viscosity of yogurt samples, respectively.

It is observed (Table 4) that none of the four models tested adequately described (see low values of r) the rheological behavior of the upward flow curves for the sample CYNT, which presented a very different profile from the others, as noted in Figure 1.

Performing analysis of variance (ANOVA) at 5% significance level, it was obtained P<0.05 for the models which fit the flow curves, indicating significant differences between them. As for the viscosity curves, P>0.05, showing that all models were well adjusted, with no significant difference. In virtually all cases, the best results were for the Herschel-Bulkley model (r>0,99), whose calculated parameters for each sample are shown in Table 6.

In contrast, the Bingham model presented the worst fit to the curves of flow and viscosity (is well noted for

upward curves), which confirms the nonlinear relationship between stress and shear rate, corroborating the classification of yogurt analyzed as non-Newtonian fluids.^{31, 34}

The Ostwald-de-Waele model, as well as the Herschel-Bulkley model, considers the nonlinear relationship between stress and shear rate. However, the first model does not take into account the yield stress (τ_0).³⁹ Because of the similarity between the models, except for a linear coefficient in the mathematical equation, both had fits with r values>0.99. However, the model of Ostwald-de Waele leads to a loss of an important information for the processing of yogurt (τ_0), which measures the minimum tension necessary for there to be flow.³⁴

It can be seen in Table 5 that all values of flow index (n) are smaller than 1, in agreement with literature data,

Table 2 – Analytical determinations of yogurt samples.			
Sample	pН	Water content (%)	
CY	4,11	78	
CYT	4,6	77,5	
CYNT	4,7	78.5	

Table 3 - Hysteresis of yogurt samples.

Sample	Hysteresis (Pa/s)
CYT	232.4
CYNT	355.5
CY	112

Table 4 - r values for the models adjusted to the flow curves.

Model –	r		
	CYT	CYNT	СҮ
Bingham	0.9341	0.7016	0.9263
Ostwald Weale	0.9935	0.8959	0.9962
Casson	0.9698	0.8006	0.9937
Herschel-Bulkley	0.9979	0.9256	0.9993

Table 5 - r values for the models adjusted to the viscosity curves.

Model -	r		
	СҮТ	CYNT	СҮ
Bingham	0.9777	0.9745	0.9898
Ostwald Weale	0.9988	0.9965	0.9997
Casson	0.9921	0.9873	0.9988
Herschel-Bulkley	0.9988	0.9990	0.9997

Table 6 - Parameters of Herschel-Bulkley model for the yogurt samples.

	Flow Curve (rising curve)		
-	τ_0 (Pa)	K (Pa.s ⁿ)	n
CYT	17.83	19.81	0.1322
CYNT	163.8	168.8	0.0104
CY	16.580	19.580	0.078

showing again the classification of fluids analyzed as non-Newtonian pseudoplastic.^{14, 38}

It should be noted that the results are consistent for CYT and CY samples, which were well fitted by the model (values of r² = 0.9979 and 0.9993, respectively). For these samples, the highest value of yield stress (τ_0), although quite close, was for the CYT sample, which showed higher viscosity values. Also consistent with these results, the highest consistency index (K) was obtained for the CYT sample, compared with CY sample. The results for CYNT sample are unreliable because the value of r² was the lowest, indicating poor model fit to the curve (r² = 0.9256).

Several works have studied the rheological behavior of different formulations of yogurt, obtaining as results for the yield stress (τ_0) values between 1.1 and 10.1 Pa, depending on the solids content, type of thickener, type of starter culture (exopolysaccharide-producing or not) and presence of prebiotics.^{14, 28, 33, 38} The results obtained here indicate that the gelatin acts as a thickener with a strong impact by restricting the flow of the final product.

The thixotropic characteristics are confirmed by the analysis of viscosity versus time at a constant shear rate. In accordance with the data presented in the literature, all yogurt samples showed a reduction of viscosity versus time.^{6, 9, 14, 25} The curves of viscosity over time are shown in Figure 3, which represent thixotropy tests for the three samples of yogurt.

Observing the curves it can be confirmed the highest values of viscosity for the CYT sample, followed by CYNT and CY samples in this sequence, confirming the data obtained in the viscosity curves. Also confirming the calculations of hysteresis, there are greater reductions in the viscosity over time for CYNT, CYT and CY samples, in that order.

Table 7 presents the results of thixotropy test adjusted to Weltman model.

The parameter A indicates the flow resistance, having a close correlation with the yield stress (τ_0), relating with the shear stress at the time 1s. It can be confirmed that Herschel-Bulkley model was not well adjusted to CYNT sample, because the τ_0 value does not match the value of the parameter obtained by the model of Weltman. As for the CY and CYT samples, the results are consistent with each other, although the Weltman model has not adequately described the thixotropy of the CY sample ($r^2 = 0.9226$).

The parameter B measures the speed of structural damage when the fluid is subjected to any shear rate. Negative values indicate a decrease in tension due to the increase of shear rate. It shows that the samples of coffee-flavored yogurt had the highest rates of structural breakdown. Thus, locust bean gum promotes a further decrease in the rate of breakdown of the gel.

Gonçalvez et al.¹⁴ evaluated the rheological behavior of yogurt with different concentrations of starch and gelatin, as thickeners, as well as free yogurt thickener.



FIGURE 3 - Curve of viscosity versus time of testing thixotropy of yogurt samples.

Table 7 - Parameters of Weltman model for the thixotropy test.

	Α	В	r ²
CYT	16.61	-5.24	0.9946
CYNT	15.13	-5.09	0.9870
CY	13.27	-1.06	0.9226

The yogurts with gelatin concentrations of 3 and 6 g L⁻¹ showed discrepancies between τ_0 and A values. However, it is noteworthy that in their study, the r values were low for the Weltman model for both samples (r <0.9).

Also according to Gonçalvez et al., ¹⁴ the values of parameters B calculated for the samples with gelatin were seven times higher than those obtained in this work. This difference can be explained by the poor fit of the model of Weltman and the results obtained by them (r<0.9).

CONCLUSIONS

All samples showed non-Newtonian pseudoplastic and thixotropic behavior. The three samples of yogurt with different concentrations and types of thickeners showed different ranges of viscosity as a function of shear rate. The CYT sample, which uses gelatin as a thickener, showed the highest viscosity values. The sample CYNT which did not contain a thickener in the formulation, showed the highest hysteresis value, indicating greater structural damage under processing. The Herschel-Bulkley model represents more accurately the flow curves of the three samples of yogurt. All values of flow index (n) were less than 1, confirming the pseudoplastic characteristic. Thickeners showed protective effect on the gel during the flow, with lower hysteresis values for samples CYT and CY. The Weltman model represented well the thixotropy tests, except for sample CY ($r^2 = 0.92$). Thus, we can conclude that the addition of gelatin as thickener agent promoted an increase of viscosity and consistency of the final product, besides promoting major protective effect on the gel - factors that have great importance on the product acceptability. When compared with commercial yogurt, the coffee-flavored yogurt with gelatin addition presented satisfactory rheological properties.

MATHIAS, T. R. S.; CARVALHO JUNIOR, I. C.; CARVALHO, C. W. P.; SÉRVULO, E. F. C. Caracterização reológica de iogurte sabor café com diferentes tipos de espessante. **Alim. Nutr.**, Araraquara, v. 22, n. 4, p. 521-529, out./dez. 2011.

■RESUMO: logurte é um alimento funcional que apresenta grande demanda devido à busca por uma vida e alimentação mais saudáveis. De forma a ampliar o mercado consumidor, diversos são os sabores existentes, satisfazendo as mais variadas preferências. Além do sabor, a consistência e viscosidade do iogurte são uns dos principais fatores envolvidos na qualidade e aceitação do produto. Este trabalho é um estudo da influência do tipo de espessante em iogurte sabor café. O espessante utilizado foi a gelatina. O comportamento reológico (curvas de fluxo e de viscosidade) dos iogurtes com e sem adição de gelatina foram comparados entre si e com iogurte comercial, o qual utiliza outro tipo de espessante (goma alfarroba) em suas formulações. As curvas de fluxo e de viscosidade foram obtidas em reômetro rotacional Thermo Haake Mars com variação de taxa de cisalhamento entre 0,02 e 100 s⁻¹ (curva ascendente) e 100 e 0,02 s⁻¹ (curva descendente) em um tempo total de 20 minutos. Foi determinada a histerese como a área entre as curvas e ajustados os modelos de Bingham, Casson, Herschel-Bulkley e Ostwald-de-Waele. Foram realizados também testes de tixotropia, pela medição da viscosidade em função do tempo à uma taxa constante de 100 s-1, por 10 minutos. Estas curvas foram ajustadas pelo modelo de Weltman. Todas as amostras apresentaram comportamento pseudoplástico e tixotrópico. O modelo de Herschel-Bulkley foi o que melhor se ajustou às três amostras testadas. O modelo de Weltman descreveu bem os testes de tixotropia, com exceção para a amostra de iogurte comercial. O uso da gelatina como espessante apresentou caráter protetor, reduzindo a quebra estrutural.

■PALAVRAS-CHAVE: Modelos reológicos; comportamento pseudoplástico; gelatina; estabilidade do gel; viscosidade.

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