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RHEOLOGICAL CHARACTERIZATION OF BODY LOTIONS: INFLUENCE OF TEMPERATURE AND WETTING AGENTS

Caracterização Reológica de Loções Corporais: Influência da Temperatura e de Agentes Umectantes

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Abstract

The skin protects the body from excessive dehydration, therefore, the use of body lotions is interesting to provide softness and elasticity for it. Humectants capture and retain water while maintaining hydration. Rheology has been recognized as an important parameter in the development of formulations in technical terms of preparation, filling and storage. Different formulations of commercial lotion with glycerol (10, 20 and 30%) and sorbitol (3, 5 and 10%) at temperatures of 10, 25 and 40ºC were evaluated on the Brookfield rheometer, model DVIII Ultra, CP25 coded rotor (spindle), sample volume of 2mL and variation shear rate from 0 to 400 s^{-1}. The rheological parameters of fluid behavior index (n), consistency index (K) and minimum shear stress (τ_0) were obtained from the shear stress versus shear rate curves, using the Rheocalc software; moreover, it was determined among mathematical models of Power Law, Herschel-Bulkley and Casson the one with the highest correlation to the data. The results of the rograms showed pseudoplastic behavior (n < 1), thixotropy and the most appropriate mathematical model to obtain values of the rheological parameters was the Herschel-Bulkley (highest correlation index, R^2 = 0.989). The best formulation / temperature was the composition with 20% glycerol at 25ºC, indicating a viable temperature for storage. The conclusions point out that the formulations have pseudoplasticity and thixotropy that are suitable for a cosmetic product and that the storage temperature must be carefully used by the consumer.

Keywords: Hydration. O/A emulsions. Rheology. Thixotropy. Pseudoplasticity.

Resumo

A pele protege o corpo da desidratação excessiva, portanto, o uso de loções corporais é interessante para proporcionar maciez e elasticidade para a mesma. Umectantes captam e retêm a água mantendo a hidratação. A Reologia tem sido reconhecida como parâmetro importante no desenvolvimento das formulações em termos técnicos de preparo, envase e armazenamento. Diferentes formulações da loção comercial com o glicerol (10, 20 e 30%) e sorbitol (3, 5 e 10%) em temperaturas de 10, 25 e 40ºC foram avaliadas no reômetro da marca *Brookfield*, modelo DVIII Ultra, rotor (*spindle*) codificado CP25, volume de amostra de 2mL e variação da taxa de cisalhamento de 0 a 400 s^{-1}. Os parâmetros reológicos de índice de comportamento do fluido (n), índice de consistência (K) e tensão mínima de cisalhamento (τ_0) foram obtidas a partir das curvas de tensão de cisalhamento *versus* taxa de cisalhamento, utilizando o *software Rheocalc*; além disso, foi

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determinado dentre modelos matemáticos de *Power Law*, *Herschel-Bulkley* e *Casson* o de maior correlação aos dados. Os resultados dos reogramas apresentaram comportamento pseudoplástico (n < 1), tixotropia e modelo matemático mais apropriado para obter valores dos parâmetros reológicos foi o de *Herschel-Bulkley* (maior índice de correlação, R^2 = 0,989). A melhor formulação/temperatura foi a composição com 20% de glicerol a 25ºC, indicando temperatura viável para estocagem. As conclusões apontam que as formulações possuem pseudoplasticidade e tixotropia que são adequadas para um produto cosmético e que a temperatura de estocagem deve ser cuidadosamente utilizada pelo consumidor.

Palavras-chave: Hidratação. Emulsões O/A. Reologia. Tixotropia. Pseudoplasticidade.

Introduction

The skin, the largest organ in the human body, acts not only as a physical barrier against external aggressions, but also protects the body against excessive dehydration due to the presence of a mixture of lipids (cholesterol and free fatty acids) and sweat called a hydrolipid mantle present in the epidermis (Rawling & Harding, 2004; Milan *et al*., 2007). The skin can become dehydrated for several reasons. In winter, this is because the air gets colder and drier thanks to the low humidity of the season. And because it is in constant contact with the environment, there is a great loss of water, recovering from dehydration. In summer, exposure to the sun, chlorine in the pool and sea water is much more frequent, increasing sweating and, consequently, dryness. This can occur on two levels: epidermal dehydration, which occurs in the most superficial layer of the skin, and dermal, responsible

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for sustaining the first. It is also common to occur in both, which causes a lot of discomfort in the most sensitive dermis (Bowsert *et al*., 1994; Borghetti & Knorst, 2006).

Therefore, body hydration aims to provide softness, elasticity, softness and improve the texture of body skin, essential for the maintenance of skin functions, including other factors such as delayed dryness, loss of collagen, flaccidity and premature aging. Some people don't care, but moisturizing your skin is just as important as doing oral hygiene every day. Sudden changes in temperature and excesses are the main aggressors of the skin, especially in winter, a time when the skin becomes drier, wrinkled, rough and opaque (Davis, 1997; Forter *et al*., 1997; Flynn *et al*., 2001; Roland et al., 2003; Pouns-Guiraud, 2007).

Water, the main component of cutaneous plasticity, has a transient hydration effect, so to retain it in the stratum corneum it is necessary to use humectants (glycerol and sorbitol, used in the present work), which due to the large number of hydroxyl groups present, they capture and retain water in the stratum corneum and hydration occurs (Verdier-Sevrain & Bonté, 2007; Fredrick et al., 2010; Gorcea & Laura, 2010).

To be considered suitable for use, an emulsion must meet certain criteria, such as having long-term stability and adequate consistency for application and release of assets. So decisive characteristics can be determined and adjusted through the rheological analysis that consists of the study of the properties of the flow and deformation of the matter (Diavao, 2009; Coutinho *et al*., 2018). The flow properties of an emulsion are physical characteristics that determine the balance of the system, as they provide information on the physical stability and aesthetic aspect of the product. The correlation between rheological assessment and these physical and commercially accepted aspects is of great importance in

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predicting performance and developing new products (Schueller & Romanowski, 1998; Trados, 2004).

The rheological behavior of an emulsion depends on the interaction between the components of the system. The basic parameters that determine it are; rheology of the continuous phase, maturity of the particles of the dispersed phase (size, concentration and deformability) and the interactions between them (Almeida & Bahia, 2003; Wassan & Nikolov, 2007).

In systems emulsified by non-ionic surfactants and fatty alcohols where there is formation of lamellar bilayers in the gel phase, rheology is influenced by the displacement of water between the interlayer space and the dispersing phase (Ribeiro *et al*., 2004). According to Eccleston (1990) and Ribeiro *et al*. (2004) the capacity to store water between the lamellae and, consequently, the rheological properties of these emulsions are determined by the size of the lipophilic chain and the number of ethoxylations of the surfactant.

Through rheological characterization, it is possible to determine whether a sample has Newtonian or non-Newtonian flow. Newtonian fluids obey Newton's law which establishes that the flow velocity is directly proportional to the applied stress, therefore, the viscosity is independent of the shear rate. This behavior is generally observed in solutions of molecules with low molecular weight and diluted solutions (Barnes, 1994).

Emulsified systems generally have non-Newtonian flow. In these, the viscosity depends on the shear rate, that is, different viscosities can be obtained according to the variation of the speed and the applied force as a function of time. This behavior is due to deviations from Newton's law. Three different types of non-Newtonian behavior are known

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depending on the type of deviation: plastic flow, pseudo-plastic flow and dilating flow, with or without thixotropy (Barnes, 1994; Corrêa *et al*., 2005).

For cosmetic products, the pseudoplastic flow with thixitropy is of interest. Materials that exhibit this behavior show a decrease in yield strength as the shear rate increases. Viscosity decreases with increasing deformation speed. This decrease in viscosity is reversible when the shear rate is decreased or stopped. It is interesting for cosmetic products, as they become less viscous at the time of application, facilitating the use of the formulation (Morris & Ross, 2002; Gao, 2003).

This work aimed to present the importance of temperature and the wetting agents in body lotion formulations using Rheology as a parameter for physical-chemical quality control. First, the three commercial lotions most sold and used by the final consumer (market evaluation) were characterized by Rheology at different temperatures (10, 25 and 40ºC). Then, formulations with different wetting agents (glycerol and sorbitol) were characterized by Rheology at the different temperatures using the commercial lotion with the lowest thixotropy value.

Material and Methods

Commercial Lotions

Three commercial lotions (CL-1, CL-2 and CL-3) were chosen by a market study (with higher sales and consumption) at the first semester of the year 2020.

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Formulations

The commercial lotion with the lowest thixotropy value was used to develop formulations in different proportions for glycerol and sorbitol, both purchased in the drugstore. The six formulations were characterized by rheology at three different temperatures.

Rheology

All commercial lotions and formulations were evaluated in the Brookfield rheometer, DVIII Ultra model, CP25 coded rotor (spindle), sample volume of 2mL and variation shear rate from 0 to 400 s^{-1}. The rheological parameters of fluid behavior index (*n*), consistency index (*K*) and minimum shear stress (τ_0) were obtained from the shear stress *versus* shear rate curves, using the 'Rheocalc' software; moreover, it was determined among mathematical models of Power Law, Herschel-Bulkley and Casson the one with the highest correlation to the data.

Results and Discussion

Figure 1 shows the viscosity *versus* shear rate curves for the three commercial lotions at 25ºC, indicating the pseudoplastic behavior (decreased viscosity with increased shear rate) and the thixotropic phenomenon, the ascending curves (AC) are superior to the descending ones (DC), suitable for cosmetic products (Almeida & Bahia, 2003; Côrrea *et al*., 2005).

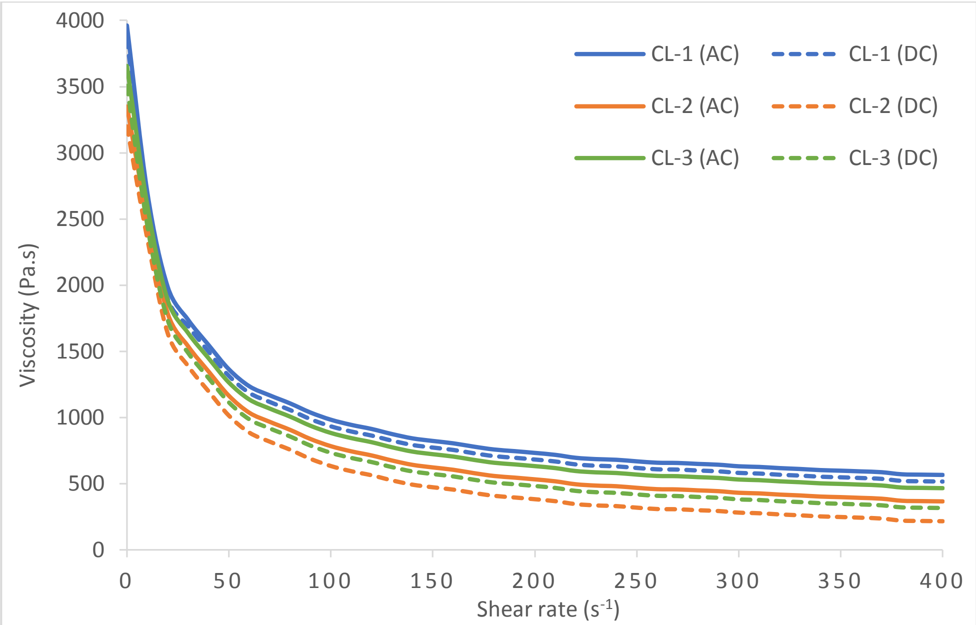

Figure 1: Curves of the relationship between viscosity and shear rate in commercial lotions at 25ºC.

Table 1 shows the rheological parameters calculated on 'Rheocalc' software in relation to the three mathematical rheological models (Power Law, Herschel-Bulkley and Casson). Pseudoplastic behavior was confirmed due to the values less than 1 for fluid behavior index ($n < 1$). The Commercial Lotion 1 was chosen for the following formulations since it had the lowest thixotropy value (smallest hysteresis area between ascending and descendig curves).

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Table 1: Rheological Parameters calculated by the 'Rheocalc' Software.

Samples (25°C)	**Power Law** $\tau = K \cdot \gamma^n$	**Herschel-Bulkley** $\tau = \tau_0 + K \cdot \gamma^n$	**Casson** $\tau^{0,5} = K_0 + K \cdot \gamma^{0,5}$ $(K_0^2 = \tau_0)$	**Thixotropy**
CL-1	K = 36,5 Pa.s n = 0,34 R^2 = 0,985	τ_0 = 87,7 Pa K = 8,8 Pa.s, n = 0,51 R^2 = 0,987	τ_0 = 115,6 Pa R^2 = 0,976	13,7 Pa/s
CL-2	K = 34,5 Pa.s n = 0,30 R^2 = 0,984	τ_0 = 96,3 Pa K = 7,8 Pa.s, n = 0,55 R^2 = 0,988	τ_0 = 188,4 Pa R^2 = 0,975	18,2 Pa/s
Cl-3	K = 35,8 Pa.s n = 0,41 R^2 = 0,980	τ_0 = 121,3 Pa K = 7,4 Pa.s, n = 0,57 R^2 = 0,988	τ_0 = 139,0 Pa R^2 = 0,973	25,3 Pa/s

Figures 2, 3 and 4 shows the relationship between viscosity and shear rate for the formulations with glycerol at differents temperatures (10, 25 and 40ºC, respectively). The results of the reograms showed pseudoplastic behavior (n < 1), thixotropy (the ascending curves are superior to the descending ones).

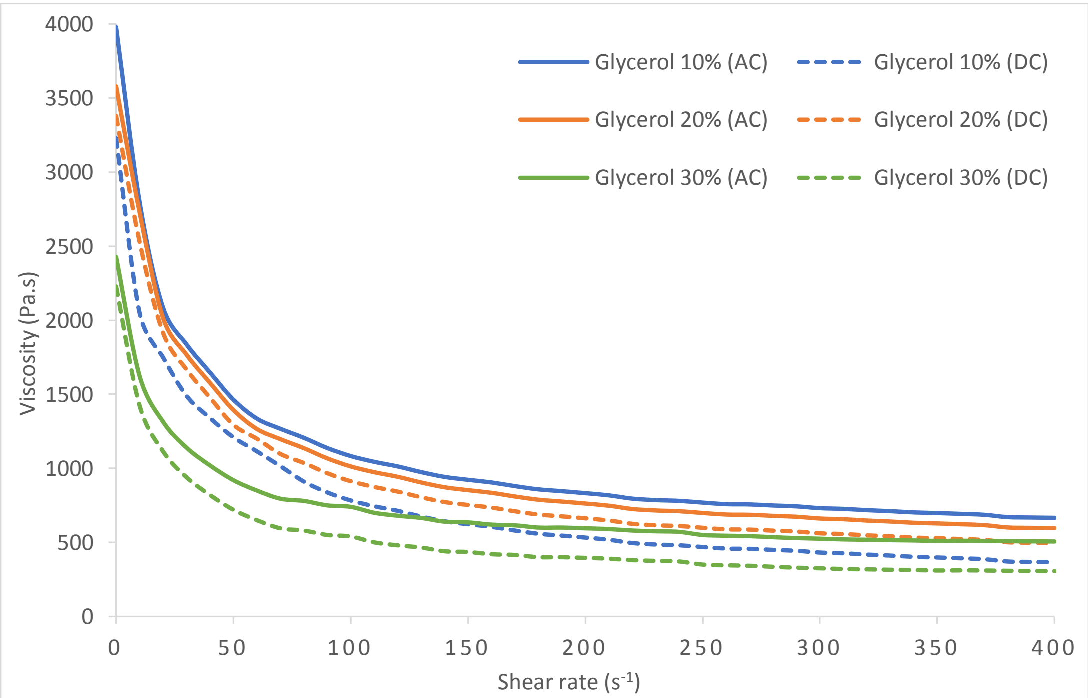

Figure 2: Curves of the relationship between viscosity and shear rate in formulation with glycerol at 10ºC.

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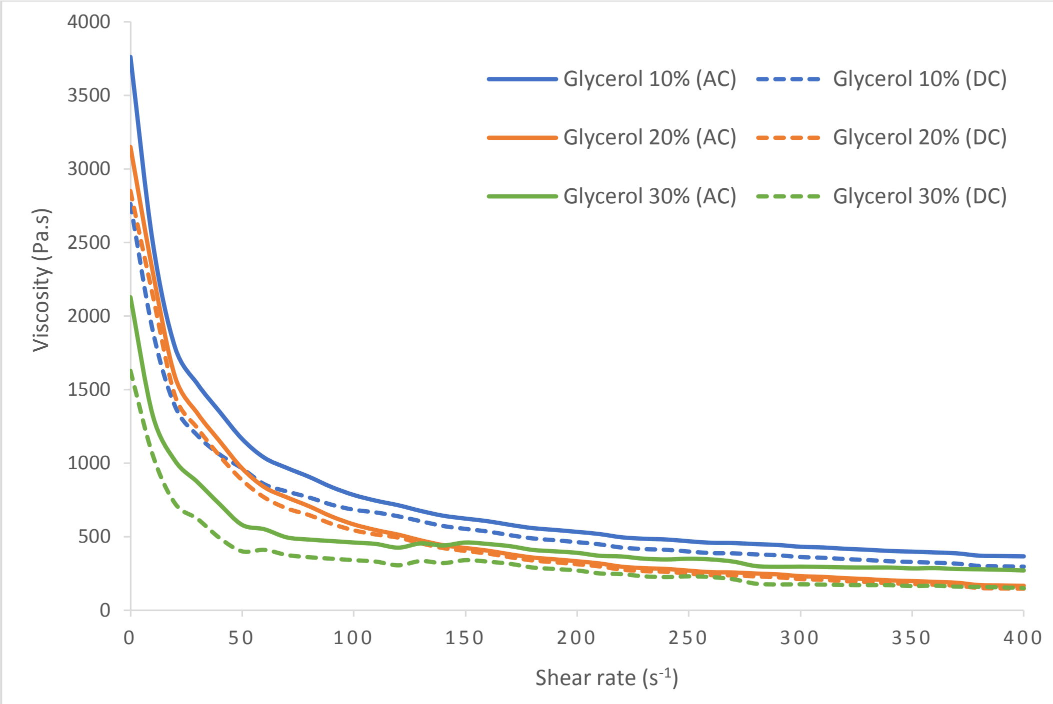

Figure 3: Curves of the relationship between viscosity and shear rate in formulation with glycerol at 25ºC.

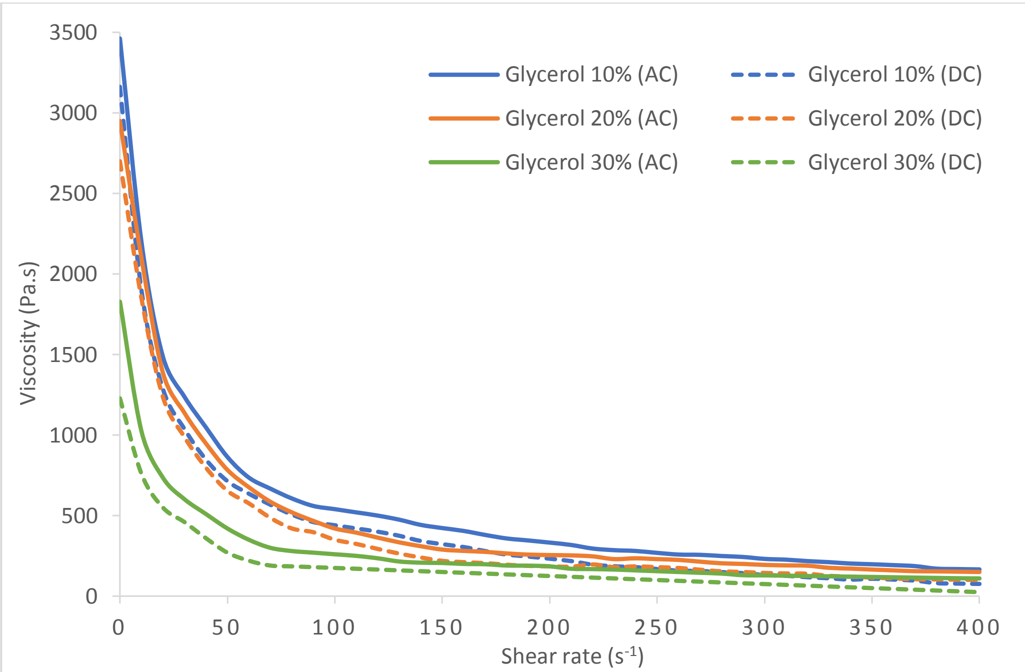

Figure 4: Curves of the relationship between viscosity and shear rate in formulation with glycerol at 40ºC.

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The most appropriate mathematical model for obtaining values of the rheological parameters was the Herschel-Bulkley (highest correlation index, R^2 = 0.989) in Table 2. Based on the choice of the smallest hysteresis area (area formed by the ascending and descending curves), according to Table 2 the combination of formulation/temperature was the 20% glycerol at 25ºC.

Table 2: Rheological Parameters calculated by the 'Rheocalc' Software for Formulations with Glycerol

Samples	**Power Law** $\tau = K \cdot \gamma^{n}$	**Herschel-Bulkley** $\tau = \tau_0 + K \cdot \gamma^{n}$	**Casson** $\tau^{0,5} = K_0^{0,5} + K \cdot \gamma$ ($K_0^2 = \tau_0$)	**Thixotropy**
Glycerol 10%/10ºC	K = 142,4 Pa.s n = 0,45 R^2 = 0,979	τ_0 = 78,2 Pa K = 95,4 Pa.s, n = 0,50 R^2 = 0,988	τ_0 = 94,2 Pa R^2 = 0,971	64,3 Pa/s
Glycerol 10%/25ºC	K = 128,5 Pa.s n = 0,41 R^2 = 0,979	τ_0 = 65,3 Pa K = 90,7 Pa.s, n = 0,49 R^2 = 0,988	τ_0 = 90,3 Pa R^2 = 0,972	40,4 Pa/s
Glycerol 10%/40ºC	K = 114,1 Pa.s n = 0,44 R^2 = 0,978	τ_0 = 60,6 Pa K = 89,7 Pa.s, n = 0,48 R^2 = 0,988	τ_0 = 88,6 Pa R^2 = 0,971	47,2 Pa/s
Glycerol 20%/10ºC	K = 77,9 Pa.s n = 0,37 R^2 = 0,980	τ_0 = 59,4 Pa K = 60,7 Pa.s, n = 0,52 R^2 = 0,989	τ_0 = 85,4 Pa R^2 = 0,976	22,5 Pa/s
Glycerol 20%/25ºC	K = 68,2 Pa.s n = 0,38 R^2 = 0,980	τ_0 = 55,6 Pa K = 56,2 Pa.s, n = 0,53 R^2 = 0,989	τ_0 = 80,6 Pa R^2 = 0,975	15,2 Pa/s
Glycerol 20%/40ºC	K = 54,5 Pa.s n = 0,42 R^2 = 0,980	τ_0 = 51,8 Pa K = 50,3 Pa.s, n = 0,51 R^2 = 0,989	τ_0 = 77,3 Pa R^2 = 0,975	30,4 Pa/s
Glycerol 30%/10ºC	K = 22,6 Pa.s n = 0,37 R^2 = 0,978	τ_0 = 45,2 Pa K = 34,7 Pa.s, n = 0,42 R^2 = 0,988	τ_0 = 64,1 Pa R^2 = 0,973	69,3 Pa/s
Glycerol 30%/25ºC	K = 20,8 Pa.s n = 0,31 R^2 = 0,977	τ_0 = 40,2 Pa K = 30,1 Pa.s, n = 0,45 R^2 = 0,987	τ_0 = 60,5 Pa R^2 = 0,971	45,4 Pa/s
Glycerol 30%/40ºC	K = 18,4 Pa.s n = 0,35 R^2 = 0,977	τ_0 = 37,4 Pa K = 25,9 Pa.s, n = 0,41 R^2 = 0,987	τ_0 = 57,3 Pa R^2 = 0,972	48,9 Pa/s

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Figures 5, 6 and 7 shows the relationship between viscosity and shear rate for the formulations with sorbitol at differents temperatures (10, 25 and 40ºC, respectively). The results of the reograms showed pseudoplastic behavior (*n* <1) and thixotropy (the ascending curves are superior to the descending ones) too.

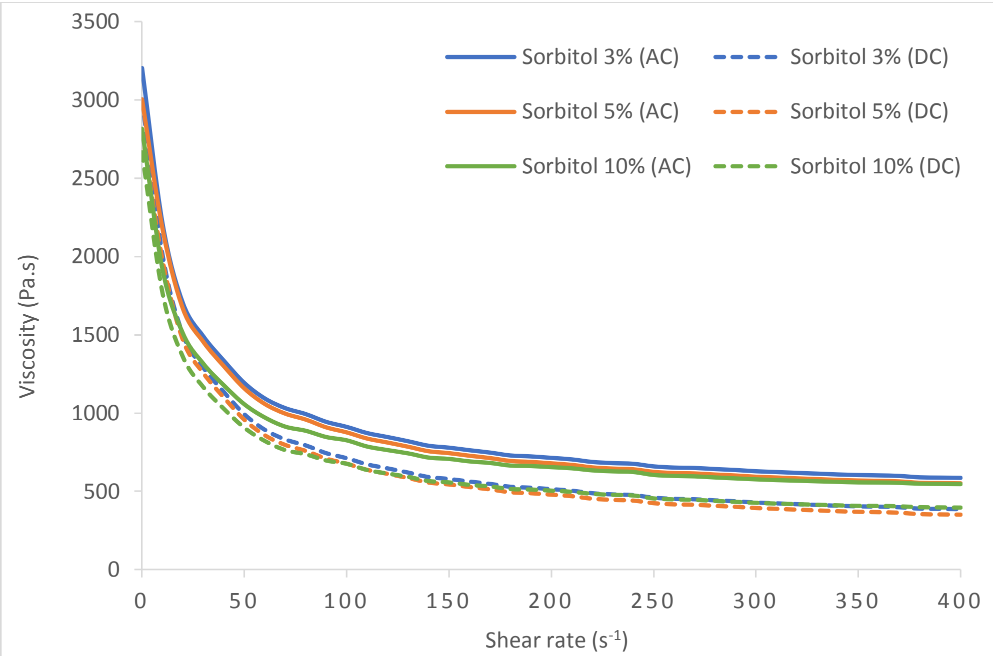

Figure 5: Curves of the relationship between viscosity and shear rate in formulation with sorbitol at 10ºC.

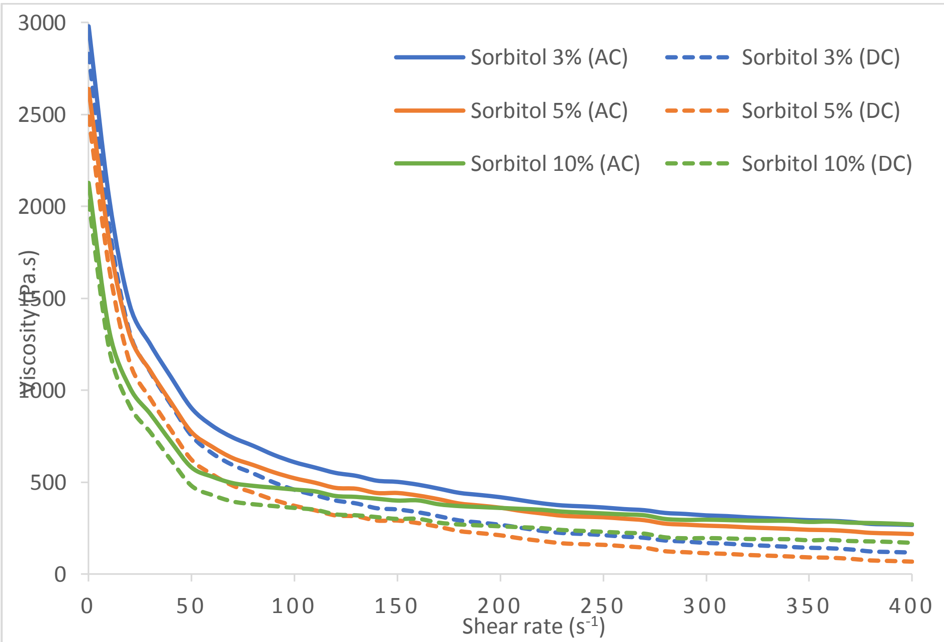

Figure 6: Curves of the relationship between viscosity and shear rate in formulation with sorbitol at 25ºC.

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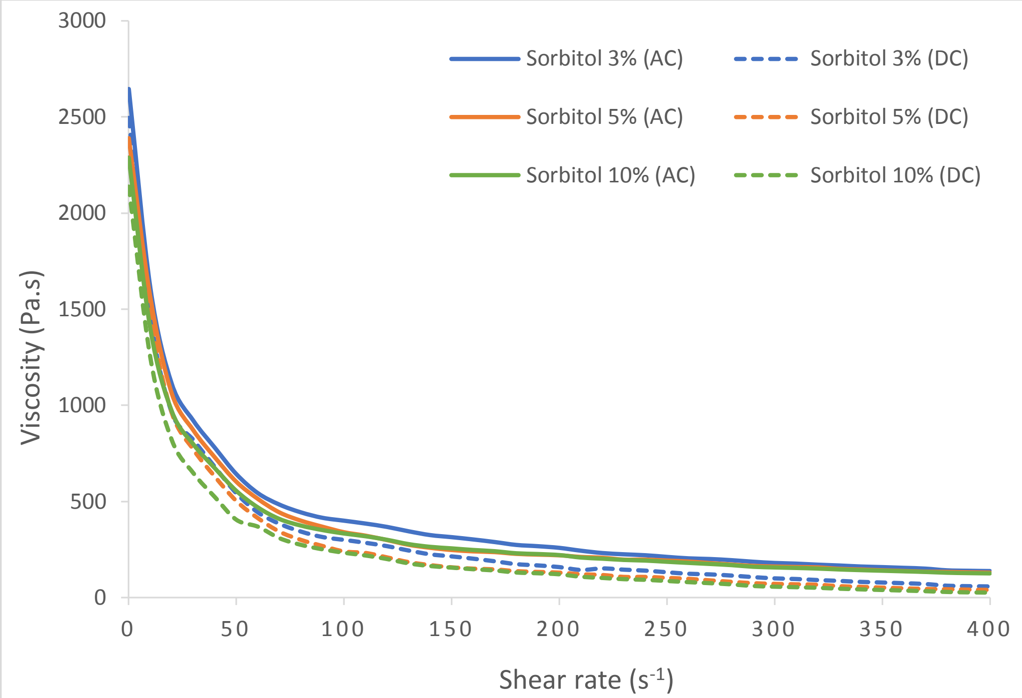

Figure 7: Curves of the relationship between viscosity and shear rate in formulation with sorbitol at 40ºC.

The most appropriate mathematical model for obtaining values of the rheological parameters was the Herschel-Bulkley (highest correlation index, R^2 = 0.979) in Table 3. Based on the choice of the smallest hysteresis area (area formed by the ascending and descending curves), according to Table 3 the combination of formulation/temperature was the 5% sorbitol at 40ºC.

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Table 3: Rheological Parameters calculated by the 'Rheocalc' Software for Formulations with Sorbitol

Samples	Power Law $\tau = K . \gamma^n$	Herschel-Bulkley $\tau = \tau_0 + K . \gamma^n$	Casson $\tau^{0,5} = K_0 + K . \gamma^{0,5}$ $(K_0^2 = \tau_0)$	Thixotropy
Sorbitol 3%/10ºC	K = 14,4 Pa.s n = 0,45 R^2 = 0,969	τ_0 = 28,2 Pa K = 45,7 Pa.s, n = 0,58 R^2 = 0,974	τ_0 = 83,1 Pa R^2 = 0,959	83,4 Pa/s
Sorbitol 3%/25ºC	K = 12,8 Pa.s n = 0,44 R^2 = 0,969	τ_0 = 25,3 Pa K = 40,3 Pa.s, n = 0,57 R^2 = 0,975	τ_0 = 80,3 Pa R^2 = 0,960	55,7 Pa/s
Sorbitol 3%/40ºC	K = 11,1 Pa.s n = 0,47 R^2 = 0,967	τ_0 = 20,6 Pa K = 39,2 Pa.s, n = 0,56 R^2 = 0,976	τ_0 = 78,5 Pa R^2 = 0,961	67,6 Pa/s
Sorbitol 5%/10ºC	K = 37,9 Pa.s n = 0,47 R^2 = 0,974	τ_0 = 30,3 Pa K = 52,7 Pa.s, n = 0,55 R^2 = 0,979	τ_0 = 72,3 Pa R^2 = 0,966	47,4 Pa/s
Sorbitol 5%/25ºC	K = 40,2 Pa.s n = 0,48 R^2 = 0,969	τ_0 = 45,3 Pa K = 52,4 Pa.s, n = 0,53 R^2 = 0,977	τ_0 = 80,9 Pa R^2 = 0,962	45,1 Pa/s
Sorbitol 5%/40ºC	K = 44,4 Pa.s n = 0,47 R^2 = 0,970	τ_0 = 41,8 Pa K = 50,3 Pa.s, n = 0,54 R^2 = 0,978	τ_0 = 85,6 Pa R^2 = 0,964	40,3 Pa/s
Sorbitol 10%/10ºC	K = 56,8 Pa.s n = 0,48 R^2 = 0,970	τ_0 = 64,5 Pa K = 55,6 Pa.s, n = 0,57 R^2 = 0,977	τ_0 = 74,1 Pa R^2 = 0,963	59,0 Pa/s
Sorbitol 10%/25ºC	K = 50,4 Pa.s n = 0,40 R^2 = 0,967	τ_0 = 61,7 Pa K = 59,7 Pa.s, n = 0,50 R^2 = 0,977	τ_0 = 70,8 Pa R^2 = 0,961	55,2 Pa/s
Sorbitol 10%/40ºC	K = 48,6 Pa.s n = 0,45 R^2 = 0,967	τ_0 = 57,8 Pa K = 55,4 Pa.s, n = 0,54 R^2 = 0,975	τ_0 = 68,4 Pa R^2 = 0,960	58,4 Pa/s

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All the formulations studied, as they present pseudoplasticity and thixotropy, are suitable for a cosmetic product, since by decreasing their resistance to shear, it will cause adequate spreading and good coverage on the skin (Almeida & Bahia, 2003; Milan *et al*., 2007).

According to Tables 2 and 3, regardless of the temperature and the wetting agent used, the most appropriate mathematical model to obtain values of the rheological parameters was considered the Herschel-Bulkley, having the highest correlation index, R^2 = 0.989 in the formulation with 20% glycerol.

Conclusion

The conclusions are that the storage temperature, indicated on the labels, must be carefully used by the consumer; and that the formulations studied, as they present pseudoplasticity and thixotropy, are suitable for a cosmetic product, since by decreasing their resistance to shear, it will cause adequate spreading and good coverage on the skin. The most interesting formulation in this study was that containing 20% glycerol at a temperature of 25ºC (smallest hysteresis area and the highest value of R^2, at the same time).

Conflicts of interest

The authors declare no conflicts of interest.

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